

**United States Air Force
611th Civil Engineer Squadron
Elmendorf AFB, Alaska**

**Cape Lisburne Long Range Radar Station,
Alaska**

**QUALITY PROGRAM PLAN
ADDENDUM FOR INTERIM REMEDIAL ACTIONS
FINAL**

DECEMBER 1996

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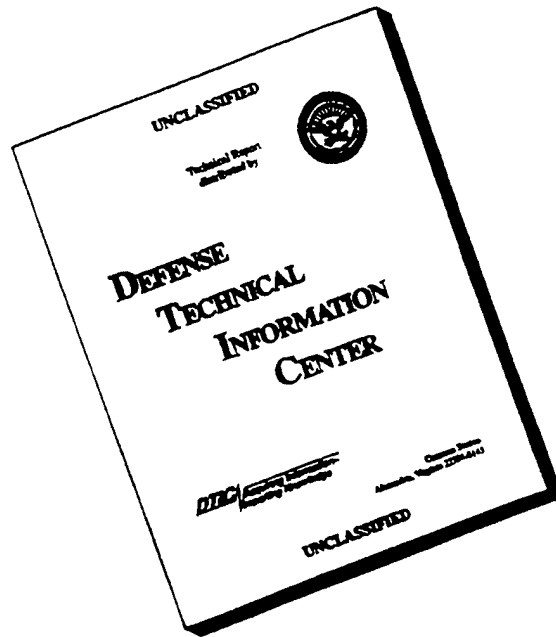
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TABLE OF CONTENTS

1.0 INTRODUCTION.....	1
1.1 Scope of Work.....	1
1.2 Project Organization	13
1.3 Site History	13
1.3.1 Historical Investigations	13
1.3.1.1 1993 Remedial Investigation.....	13
1.3.1.2 1994 Interim Remedial Actions	13
1.4 Geology and Hydrology	26
1.4.1 Geology	26
1.4.2 Hydrology.....	26
2.0 CONSTRUCTION QUALITY PLAN.....	27
2.1 Mobilization	27
2.2 Contaminated Soil Removal at Sites SS03, SS08, and SS09.....	27
2.2.1 SS03 - White Alice Site Soil Excavation	27
2.2.2 SS08 - Upper Camp Transformer Building Soil Excavation.....	35
2.2.3 SS09 - Lower Camp Transformer Building Soil Excavation.....	35
2.3 Survey and Removal of Soil and Drums at Site LF01	36
2.3.1 Geophysical Survey of Site LF01	36
2.3.2 Site LF01 Excavation	36
2.3.3 Drum Inventory and Logging	38
2.3.4 Field Screening and Transfer of Drum Contents	38
2.3.5 Drum Cleaning and Placement at Site Landfill	43
2.4 Soil Stockpiling	44
2.5 Stockpiled Soil Sampling	45
2.6 Soil Sampling for Confirmation of Cleanup Levels in Excavations	45

2.7 Sampling of Bulk Drum Contents	45
2.8 Containerized Waste Manifesting and Transportation	45
2.9 Backfill and Site Restoration.....	46
3.0 HEALTH AND SAFETY PLAN	47
3.1 Chemical Hazards.....	47
3.1.1 Petroleum Hydrocarbons	47
3.1.2 Polychlorinated Biphenyls.....	50
3.1.3 Ethylene Glycol	50
3.1.4 General.....	51
3.2 Physical Hazards	51
3.2.1 Heavy Equipment.....	51
3.2.2 Excavations	51
3.3 Temperature Stress	51
3.3.1 Cold Stress	51
3.3.2 Heat Stress	52
3.4 Biological Hazards	52
3.4.1 Bears.....	52
3.5 Drum Handling	53
3.6 Hazardous Material Bulking.....	53
3.7 Personal Protective Equipment.....	54
3.8 Emergency Facilities.....	54
3.9 Site Control.....	55
3.10 Decontamination	55
3.10.1 Personnel Decontamination.....	55
3.10.2 Equipment Decontamination	55
4.0 SAMPLING AND ANALYSIS PLAN	57

4.1 Quality Assurance Program Plan (QAPP)	57
4.1.1 Data Categories	57
4.1.2 Precision, Accuracy, Representativeness, Completeness, and Comparability	58
4.1.2.1 Precision	58
4.1.2.2 Accuracy	59
4.1.2.3 Representativeness	60
4.1.2.4 Completeness	60
4.1.2.5 Comparability	60
4.1.3 Method Detection Limits, Practical Quantitation Limits, and Instrument Calibration Requirements	61
4.1.3.1 Method Detection Limits	61
4.1.3.2 Practical Quantitation Limits	61
4.1.3.3 Instrument Calibration	61
4.1.4 Elements Of Quality Control	61
4.1.4.1 Laboratory Control Sample	62
4.1.4.2 Matrix Spike/Matrix Spike Duplicate	62
4.1.4.3 Surrogates	63
4.1.4.4 Internal Standards	63
4.1.4.5 Retention Time Windows	63
4.1.4.6 Interference Check Sample	64
4.1.4.7 Method Blank	64
4.1.4.8 Equipment Blank	64
4.1.4.9 Trip Blank	65
4.1.4.10 Field Duplicates	65
4.1.5 Quality Control Procedures	66
4.1.5.1 Holding Time Compliance	66
4.1.5.2 Confirmation	66
4.1.5.3 Standard Materials	66
4.1.5.4 Supplies and Consumables	67
4.1.5.5 Sample Dilutions	67

4.1.6 Sampling Procedures.....	67
4.1.6.1 Field Sampling	67
4.1.6.2 Sample Handling and Custody.....	67
4.1.7 Screening Analytical Methods	70
4.1.8 Analytical Methods and Procedures.....	71
4.1.8.1 Method SW6010A-Trace Elements (Metals)	72
4.1.8.2 Method AK101 - Gasoline Range Organics	75
4.1.8.3 Method AK102 - Diesel Range Organics.....	78
4.1.8.4 Method AK103 - Residual Range Organics	81
4.1.8.5 Method SW8015M - Glycols.....	84
4.1.8.6 Method SW8081 - Polychlorinated Biphenyls (PCBs)	87
4.1.8.7 Method SW8260A - Volatile Organics	90
4.1.8.8 Method SW8270B-Semivolatile Organics	97
4.1.9 Data Review, Validation, and Reporting	104
4.1.10 Corrective Actions	109
4.1.10.1 Response to Corrective Actions.....	109
4.1.10.2 Re-establishment of Control.....	109
4.1.11 Quality Assurance Reports	110
4.2 Field Sampling Plan	110
4.2.1 Final Characterization Sampling.....	110
4.2.2 Soil Sampling for Confirmation of Cleanup Levels in Excavations	111
4.2.3 Sampling of Bulk Drum Contents	111
4.2.4 Sample Labels.....	114
4.2.5 Sample Packing	114
4.2.6 Field Logbook.....	115
4.2.7 Chain of Custody.....	116
4.2.8 Soil Sampling Analysis.....	116
4.2.9 Liquid Waste Analysis	116
4.2.10 Quality Control.....	117

LIST OF TABLES

Table 3-1. Summary Of Contaminants.....	48
Table 3-2. Toxicity Characteristics	49
Table 4-1. Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times	68
Table 4-2. PQLs for Method SW6010A.....	72
Table 4-3. QC Acceptance Criteria for Method SW6010A	72
Table 4-4. Summary of Calibration and QC Procedures for Method SW6010A..	73
Table 4-5. PQLs for Method AK101	75
Table 4-6. QC Acceptance Criteria for Method AK101	75
Table 4-7. Summary of Calibration and QC Procedures for Method AK101	76
Table 4-8. PQLs for Method AK102	78
Table 4-9. QC Acceptance Criteria for Method AK102	78
Table 4-10. Summary of Calibration and QC Procedures for Method AK102	79
Table 4-11. PQLs for Method AK103	81
Table 4-12. QC Acceptance Criteria for Method AK103	81
Table 4-13. Summary of Calibration and QC Procedures for Method AK103	82
Table 4-14. PQLs for Method 8015M.....	84
Table 4-15. QC Acceptance Criteria for Method 8015M	84
Table 4-16. Summary of Calibration and QC Procedures for Method 8015M....	85
Table 4-17. PQLs for Method SW8081	87
Table 4-18. QC Acceptance Criteria for Method SW8081	87
Table 4-19. Summary of Calibration and QC Procedures for Method SW8081 ..	88
Table 4-20. PQLs for Method SW8260A.....	91
Table 4-21. QC Acceptance Criteria for Method SW8260A.....	93
Table 4-22. Summary of Calibration and QC Procedures for Method SW8260A95	
Table 4-23. PQLs for Method SW8270B.....	98
Table 4-24. QC Acceptance Criteria for Method SW8270B	100
Table 4-25. Summary of Calibration and QC Procedures for Method SW8270B102	
Table 4-26. Data Qualifiers	105
Table 4-27. General Flagging Conventions.....	106
Table 4-28. Flagging Conventions Specific to Organic Methods	107
Table 4-29. Flagging Conventions Specific to Inorganic Methods	108
Table 4-30. Sample Summary.....	112

LIST OF FIGURES

Figure 1-1 Cape Lisburne LRRS Site Plan.....	3
Figure 1-2 White Alice Site (SS03).....	5
Figure 1-3 Upper Camp Transformer Building (SS08)	7
Figure 1-4 Lower Camp Transformer Building (SS09)	9
Figure 1-5 Landfill and Waste Accumulation Area (LF01).....	11
Figure 1-6 Organizational Chart.....	15
Figure 1-7 White Alice Site (SS03) Previous Sample Locations	17
Figure 1-8 Upper Camp Transformer Bldg. (SS08) Sample Locations	19
Figure 1-9 Lower Camp Transformer Bldg. (SS09) Sample Locations	21
Figure 1-10 Landfill and Waste Accumulation Area (LF01) Sample Locations ...	23
Figure 2-1 SS03 Anticipated Excavation Area	29
Figure 2-2 SS08 Anticipated Excavation Area	31
Figure 2-3 SS09 Anticipated Excavation Area	33
Figure 2-4 Field Characterization Flow Chart.....	41

LIST OF APPENDICES

APPENDIX A:	References
APPENDIX B:	Memo to ADEC from the Air Force
APPENDIX C:	Ohmicron Immunoassay Description and Procedures
APPENDIX D:	Analytical Methods AK101, AK102, and AK103

LIST OF ACRONYMS AND ABBREVIATIONS

AALA	American Association of Laboratory Accreditation
AA	Atomic Absorption Spectroscopy
AAC	Alaskan Air Command
ADEC	Alaska Department of Environmental Conservation
AFCEE	Air Force Center for Environmental Excellence
Air Force	United States Air Force
BFB	Bromoflourobenzene
BHC	Benzene Hexachloride
BTEX	Benzene, Toluene, Ethylbenzene, and Total Xylenes
CAS	Chemical Abstracts Service
CCC	Calibration Check Compound
CB	Calibration Blank
CVC	Calibration Verification Check
CERCLA	Comprehensive Environmental Restoration Compensation and Liability Act
CL	Confidence Limit
COC	Chain of Custody
CQC	Construction Quality Control
DDD	Dichlorodiphenyldichloroethane
DDE	Dichlorodiphenyldichloroethylene
DDT	Dichlorodiphenyltrichloroethane
DFTPP	Decafluorotriphenylphosphine
DOD	Department of Defense
DOPM	Delivery Order Project Manager
DQO	Data Quality Objectives
DRO	Diesel Range Organics
DRPH	Diesel Range Petroleum Hydrocarbons
EPA	U.S. Environmental Protection Agency
FS	Feasibility Study

FSP	Field Sampling Plan
GC/MS	Gas Chromatography/Mass Spectrometry
GF	Graphite Furnace
GRO	Gasoline Range Organics
GRPH	Gasoline Range Petroleum Hydrocarbons
HDPE	High Density Polyethylene
HPLC	High Performance Liquid Chromatography
ICPES	Inductively Coupled Argon Plasma Atomic Emission Spectroscopy
ICS	Interference Check Samples
ICV	Initial Calibration Verification
IDW	Investigation Derived Waste
IRA	Interim Remedial Action
IRP	Installation Restoration Program
IS	Internal Standard
LOD	Limit of Detection
LCL	Lower Confidence Limit
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LRR	Long Range Radar
MDL	Method Detection Limit
MSA	Method of Standard Additions
MSL	Mean Sea Level
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NCR	Non Conformance Report
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
PCBs	Polychlorinated Biphenyls
PE	Performance Evaluation
PQL	Practical Quantitation Limit
QAO	Quality Assurance Officer

QAPjP	Quality Assurance Project Plan
QAPP	Quality Assurance Program Plan
QA/QC	Quality Assurance/Quality Control
QPP	Quality Program Plan
RAGS	Risk Assessment Guidance for Superfund
RBSLs	Risk-Based Screening Levels
RCRA	Resource Conservation and Recovery Act
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
REL	Recommended Exposure Limits
RF	Response Factor
RI/FS	Remedial Investigation/Feasibility Study
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Regional Program Manager
RRO	Residual Range Hydrocarbons
RRT	Relative Retention Time
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SHSO	Site Health and Safety Officer
SPCC	System Performance Check Compounds
SVOC	Semi-Volatile Organic Compound
TDS	Total Dissolved Solids
TIC	Tentatively Identified Compound
TOC	Total Organic Carbon
TPH	Total Petroleum Hydrocarbons
UCL	Upper Confidence Limit
USAF	United States Air Force
VOC	Volatile Organic Compound

WAA	Waste Accumulation Area
WACS	White Alice Communications System

LIST OF MEASUREMENTS

cm	centimeters
cy	cubic yards
mg/kg	milligrams per kilogram
µg/kg	micrograms per kilogram
mg/L	milligrams per liter
µg/L	micrograms per liter
mg/m ³	milligrams per cubic meter
ppm	parts per million
ppb	parts per billion
°F	degrees Fahrenheit
°C	degrees Celsius

1.0 INTRODUCTION

The Air Force is conducting interim remedial actions at Cape Lisburne Long Range Radar Station (LRRS) as part of the Installation Restoration Program (IRP). These actions are based on the results of the Remedial Investigation/Feasibility Study (RI/FS) conducted from 1993 to 1995. The remedial actions are to be conducted at Sites LF01 (Landfill and Waste Accumulation Area #2/Dump #1), SS03 (White Alice Site), SS08 (Upper Camp Transformer Building) and SS09 (Lower Camp Transformer Building). Figure 1-1 presents the Cape Lisburne LRRS site plan.

This QPP is site-specific to work at the Cape Lisburne LRRS, and is an addendum to the Program Quality Program Plan (QPP) dated June 1995. This QPP Addendum includes the following elements:

- Construction Quality Plan (includes Waste Plan)
- Health and Safety Plan
- Sampling and Analysis Plan, including:
 - Quality Assurance Project Plan
 - Field Sampling Plan

1.1 Scope of Work

Figures 1-2, 1-3, 1-4, and 1-5 show the four sites included in this project: White Alice Site (SS03), Upper Camp Transformer Building (SS08), Lower Camp Transformer Building (SS09), and the Landfill and Waste Accumulation Area (LF01).

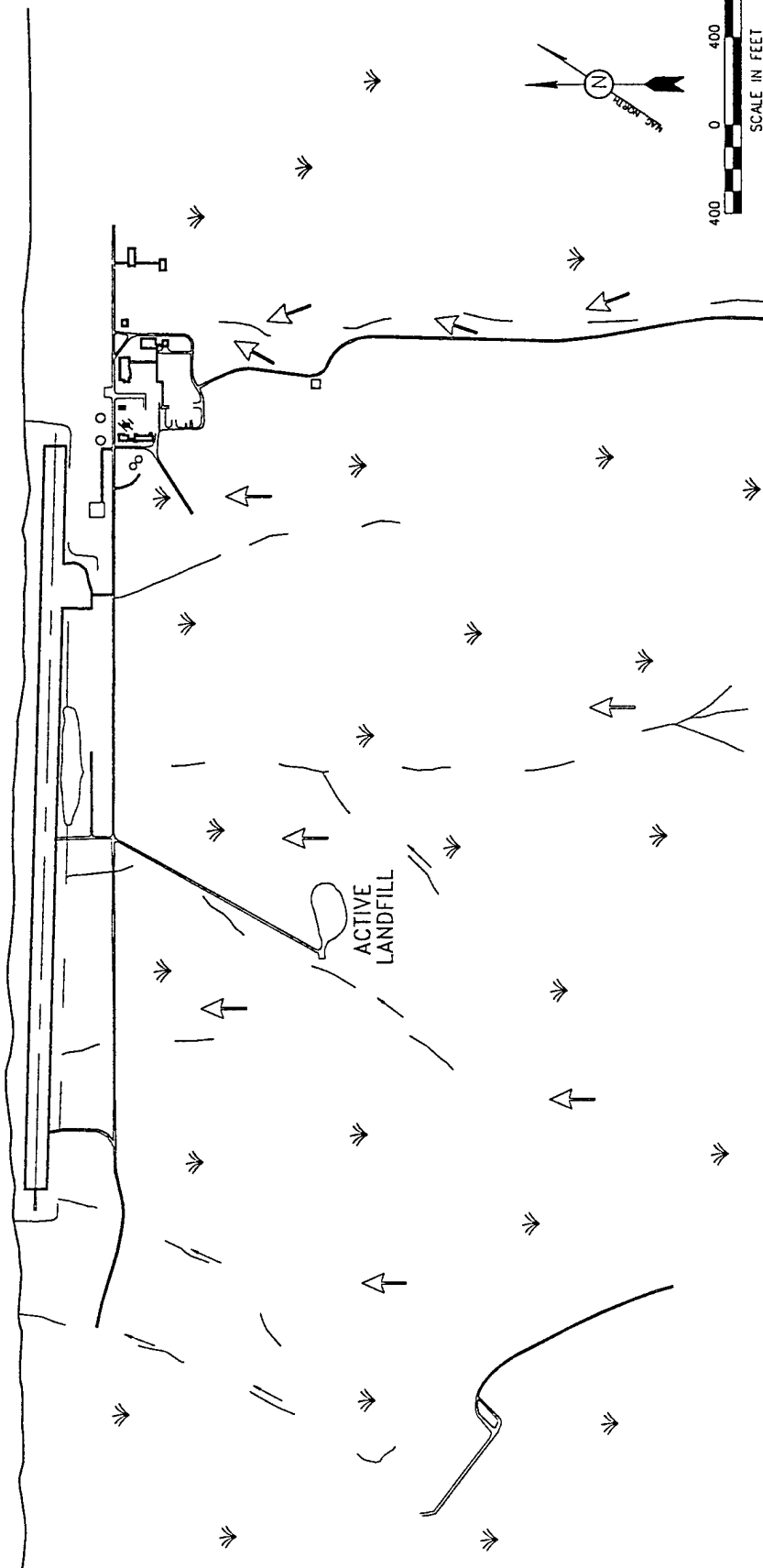
The objective of this project is to conduct remedial actions at the Cape Lisburne LRRS site to enable future environmental compliance status. This will be done by performing the following tasks:

- Conduct a geophysical survey in an attempt to locate buried drums
- Excavate, remove, and dispose of drums, residual liquids, sludges, and other waste material
- Containerize waste, label, manifest, and transport
- Excavate contaminated soil; areas of contamination have been identified during previous investigations
- Characterize excavated soil
- Collect and analyze confirmation samples of excavated areas
- Backfill and revegetate excavated areas
- Perform stockpile maintenance, both existing and those created during execution of this project

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DRAWING No. 9608--001

Arctic Ocean



ALASKA REMOTE
RADAR INSTALLATIONS

USAF 611th CES

FIGURE NO. 1-1

CAPE LISBURNE
SITE PLAN

LEGEND

- BUILDINGS, STRUCTURES
- ROADS
- TUNDRA
- SURFACE WATER
- RIVER, STREAM, OR CREEK
- SURFACE DRAINAGE

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ROAD

FRESH GRAVEL PILE

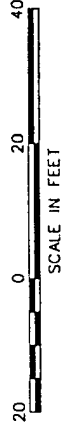
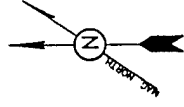
DOUBLE DOORS

DOOR AND STAIRS

DOUBLE DOORS

ROAD

DOORS AND STAIRS



LEGEND

- BUILDINGS, STRUCTURES
- ROADS

SURFACE DRAINAGE

CAPE LISBURN
RADAR INSTALLATION

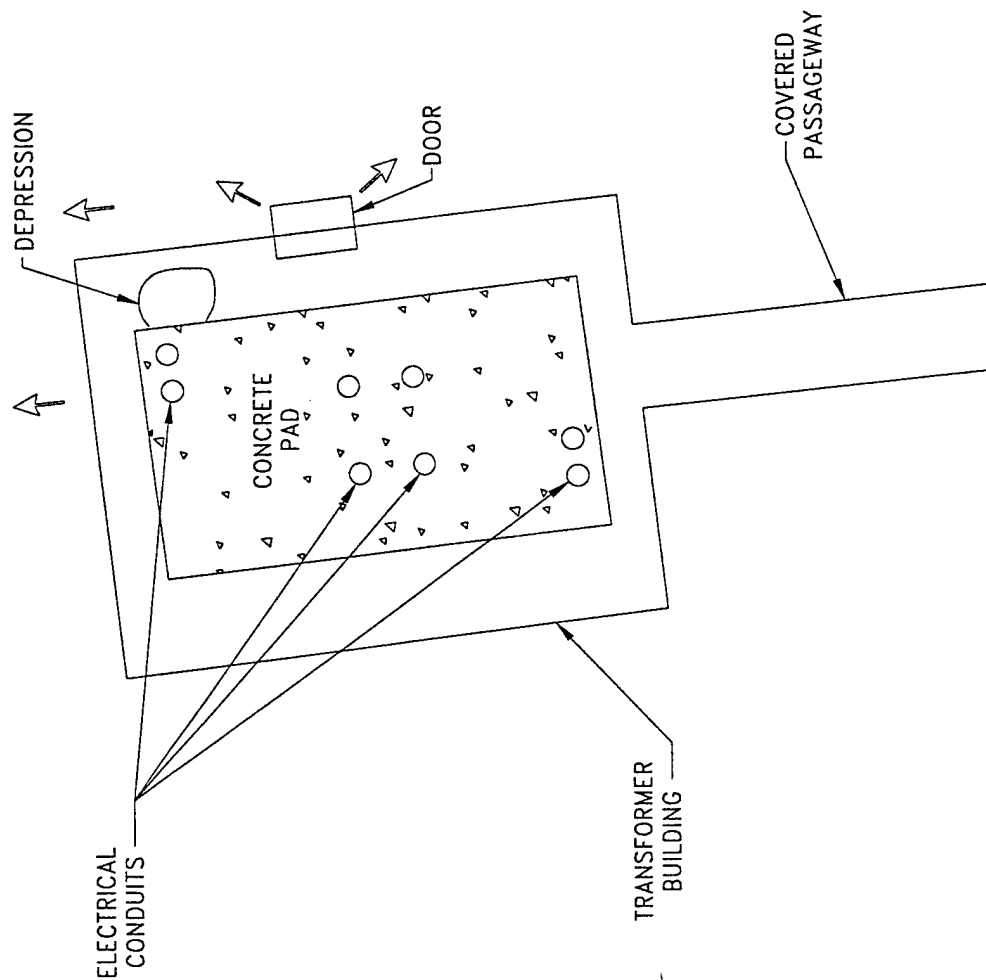
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FIGURE NO. 1-2

WHITE ALICE SITE (SS03)
SITE PLAN

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CAPE LISBURNE
RADAR INSTALLATION

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FIGURE NO. 1-3
UPPER CAMP TRANSFORMER
BUILDING (SS08)
SITE PLAN

LEGEND

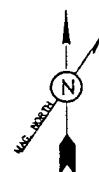
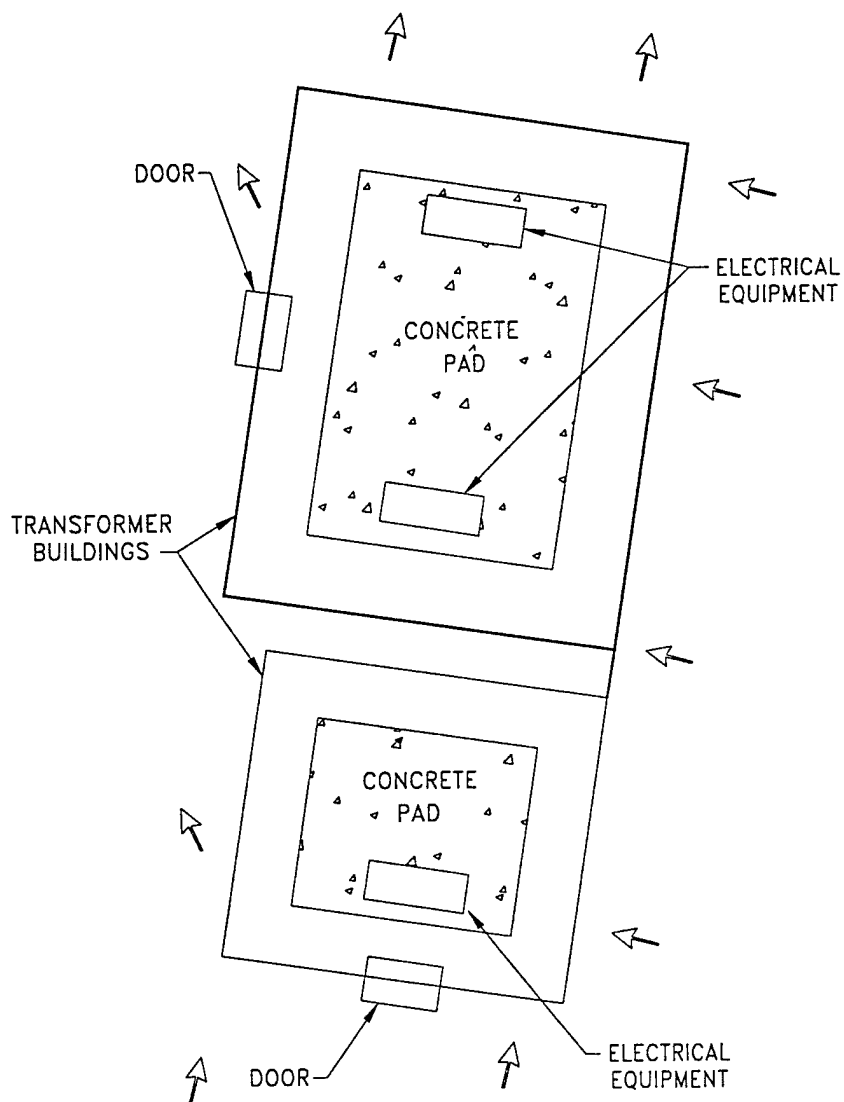
BUILDINGS, STRUCTURES



SURFACE DRAINAGE



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5 0 5 10
SCALE IN FEET

ALASKA REMOTE
RADAR INSTALLATIONS

USAF 611th CES

FIGURE NO. 1-4
LOWER CAMP TRANSFORMER
BLDG (SS09) PREVIOUS
SITE PLAN

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Arctic Ocean

BENCHMARK

BEACH

BEACH

Lake

GRAVEL COVER

CONTAINMENT CELL

GRAVEL COVERED AREA #2

ROAD

MAIN STATION
1/2 MILE

BEACON FACILITY

CAPE LISBURN
RADAR INSTALLATION

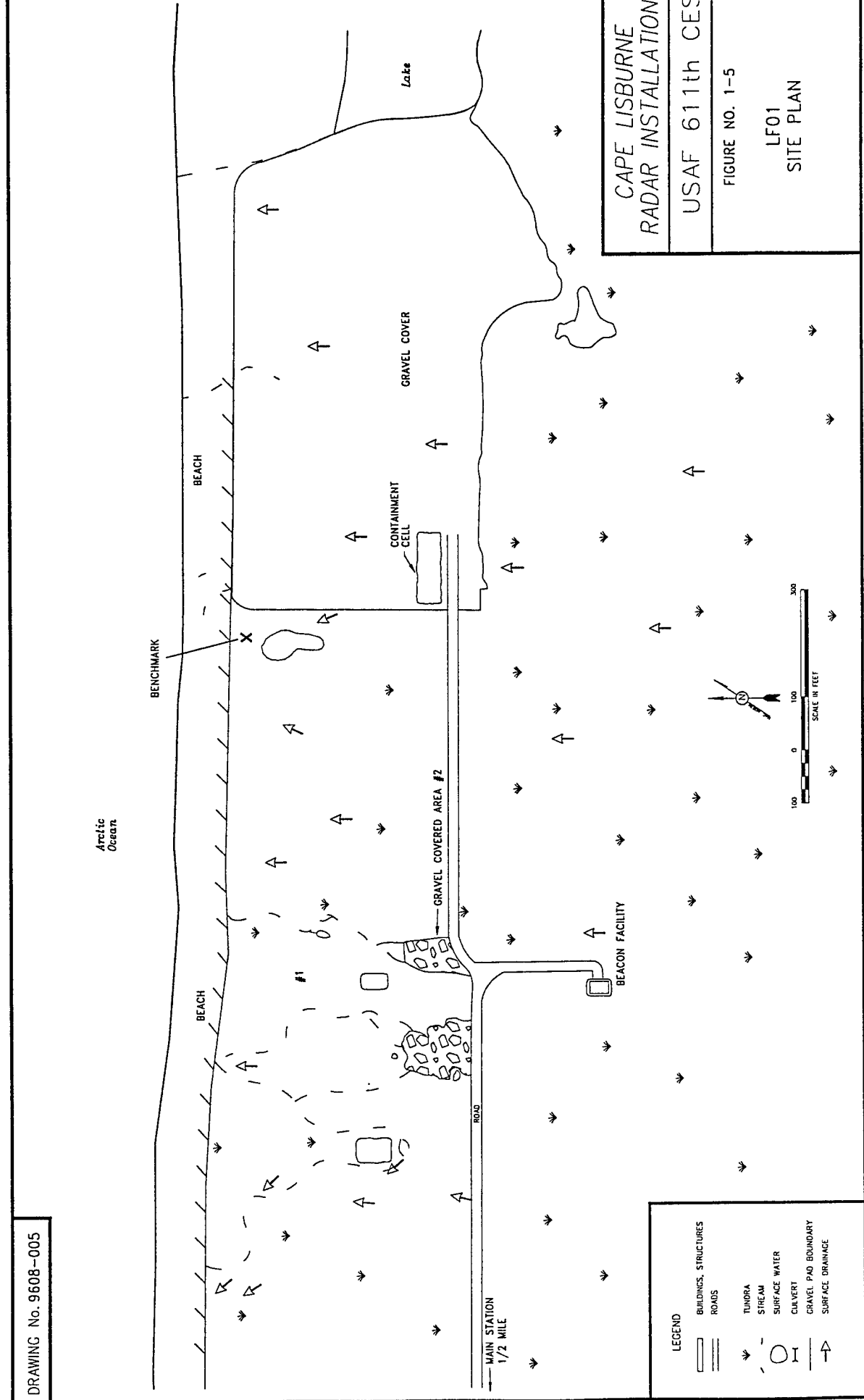
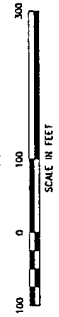
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FIGURE NO. 1-5

LF01
SITE PLAN

LEGEND

- BUILDINGS, STRUCTURES
- ROADS
- TUNDRA
- STREAM
- SURFACE WATER
- CULVERT
- GRAVEL PAD BOUNDARY
- SURFACE DRAINAGE



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1.2 Project Organization

The organizational chart for this project is provided as Figure 1-6. The organizational chart hierarchy reflects the supervisory oversight and lines of communication.

1.3 Site History

The Cape Lisburne installation is located on the shore of the Chukchi Sea and within the Alaska Maritime National Wildlife Refuge. It is located approximately 810 miles northwest of Anchorage and 570 miles northwest of Fairbanks. Point Hope is the nearest community, approximately 25 miles to the southwest. In general, the installation is only accessible by air or by sea.

The Cape Lisburne LRRS facility is one of the 31 original White Alice Communications Systems (WACS) built to establish an air defense system in Alaska. It was constructed in 1952 and 1953. The WACS began operation in August 1957. The WACS was deactivated in 1979 and replaced with a satellite earth terminal. Currently, four contract personnel at the station operate and maintain a Long Range Radar satellite earth terminal system located at the Upper Camp. Buildings at the Cape Lisburne installation are of traditional construction for wood frame, sheet-metal buildings. Support facilities in the Lower Camp include living quarters, a garage, a warehouse, inactive structures, and a 5,009 foot-long runway.

A variety of past activities at the station may have resulted in environmental contamination. These activities include the operation of communications and power generation equipment, and equipment maintenance.

1.3.1 Historical Investigations

The following sections summarize previous site activities leading up to the remedial actions to be conducted during this project.

1.3.1.1 1993 Remedial Investigation

The Air Force performed a remedial investigation/feasibility study (RI/FS) at Cape Lisburne LRRS between May 1993 and June 1995. Figures 1-7 through 1-10 summarize the analytical test results of the RI/FS. These results provide the basis of the current remedial actions.

1.3.1.2 1994 Interim Remedial Actions

Previous interim remedial actions (IRAs) have been conducted at site LF01. An IRA was also conducted at a site not included in this project referred to as the Spill/Leak #3 (ST07) site. Previous IRAs have not been conducted at site SS03, site SS08, and site SS09. The following is taken directly from *Remedial Investigation and Feasibility Study, Cape Lisburne Radar Installation, Alaska*, dated February 1996.

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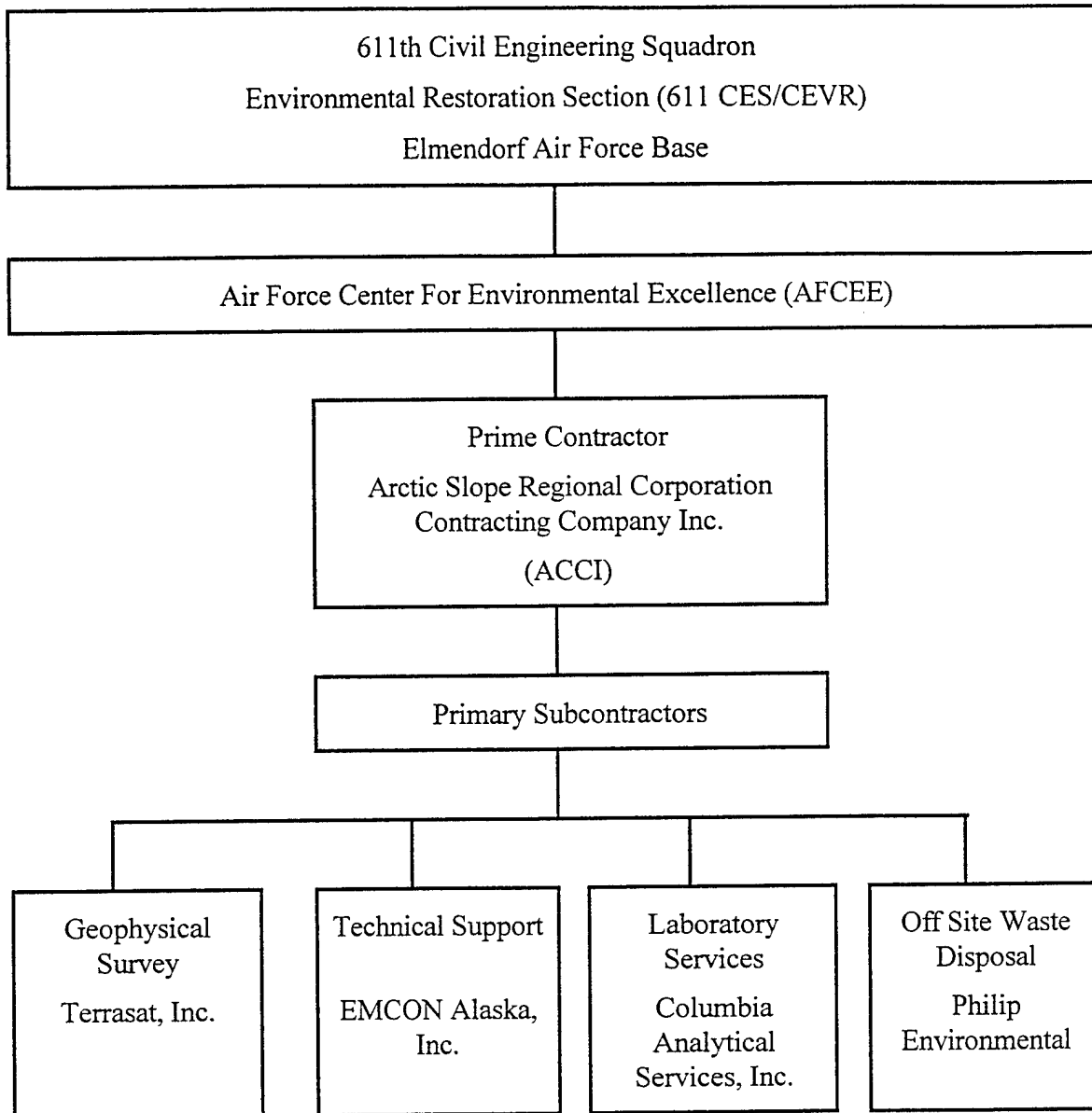


Figure 1-6 Organizational Chart

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The map shows a complex of buildings and roads. Key features include:

- Roads:** Labeled "ROAD" in two locations.
- Buildings and Structures:**
 - Top left building with "DOUBLE DOORS" and "DOOR AND STAIRS".
 - Top right building with "DOORS AND STAIRS".
 - Bottom left building with "DOUBLE DOORS".
 - Bottom right building with "DOUBLE DOORS".
- Environmental Data Points:**
 - 2006:** 14.0 ppm SVOC, 0.004 ppm VOC, 114.1 ppm PCBs.
 - 2005:** 1.8 ppm PCBs.
 - 2004:** 1.75 ppm PCBs.
 - 2003:** 5.5 ppm PCBs.
 - 2002:** 0.8 ppm PCBs.
 - 2001:** ND.
 - 2000:** 1 ppm PCBs.
 - 1999:** 1 ppm PCBs.
 - 1998:** 1 ppm PCBs.
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 - 1815:** 1 ppm PCBs.
 - 1814:** 1 ppm PCBs.
 - 1813:** 1 ppm PCBs.
 - 1812:** 1 ppm PCB

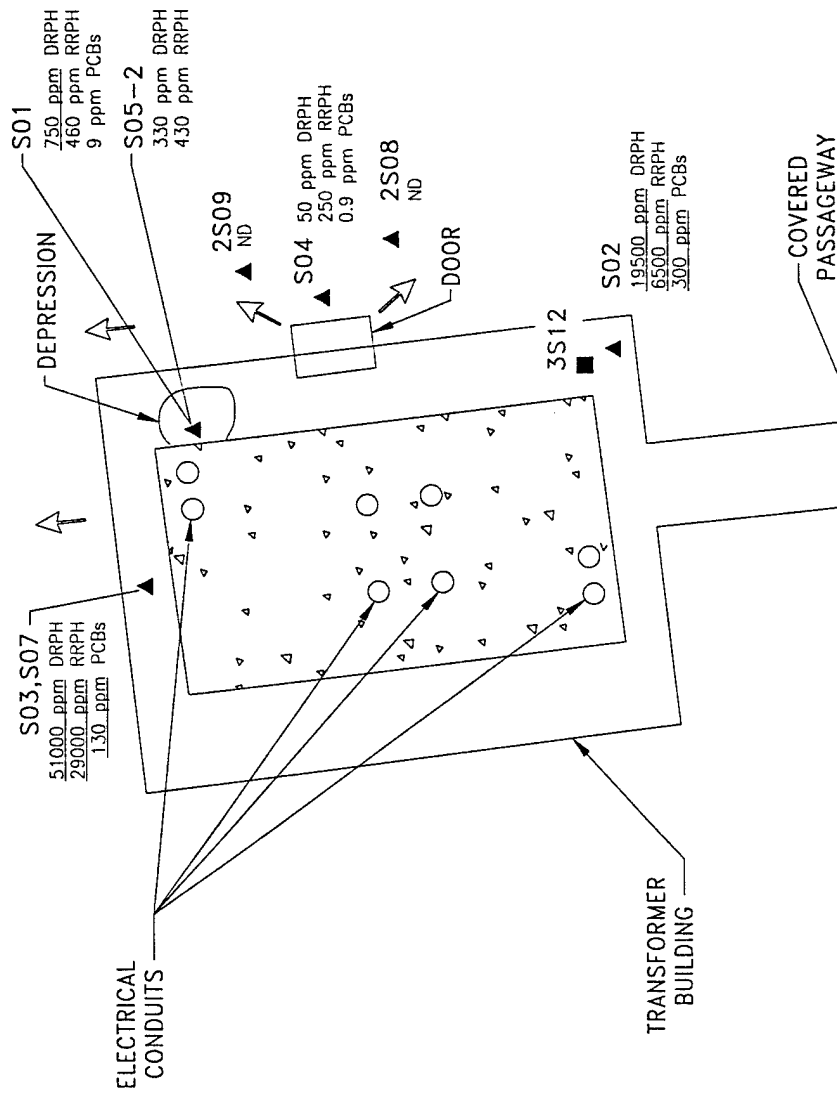
USAF 611th CES

FIGURE NO. 1-7
WHITE ALICE SITE (SS03)
PREVIOUS SAMPLE LOCATIONS
WITH ANALYTICAL RESULTS

Symbol	Feature
	BUILDINGS, STRUCTURES
	ROADS
	94 SOIL SAMPLE
	SOIL SAMPLE
	WOODWARD-CLYDE SAMPLE
	SURFACE DRAINAGE
	ANALYTE CONCENTRATIONS

CONCENTRATIONS ARE ABOVE ACTION LEVELS	NO CONTAMINATION DETECTED	DIESEL RANGE PETROLEUM HYDROCARBONS	POLYCHLORINATED BIPHENYLS
0.0000	ND	DIRPHI	PCBS

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ALASKA REMOTE
RADAR INSTALLATIONS

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FIGURE NO. 1-8
UPPER CAMP TRANSFORMER
BLDG (SS08) PREVIOUS
SAMPLE LOCATIONS WITH
ANALYTICAL RESULTS

LEGEND

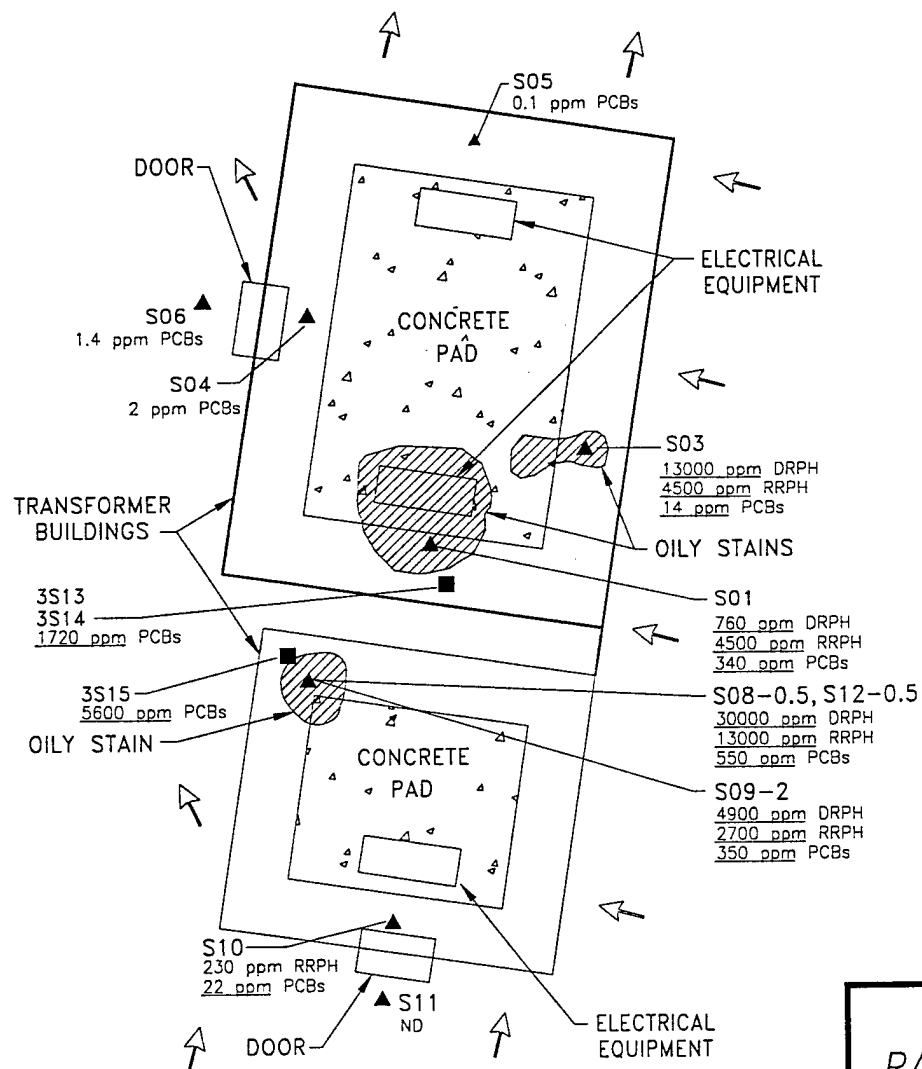
- ▬ BUILDINGS, STRUCTURES
- 94 SOIL SAMPLE
- ▲ SOIL SAMPLE
- SURFACE DRAINAGE
- 0.9 ppm ANALYTE CONCENTRATIONS

CONCENTRATIONS ARE ABOVE
ACTION LEVELS

ND NO CONTAMINATION DETECTED
DRPH DIESEL RANGE PETROLEUM
HYDROCARBONS
RRPB RESIDUAL RANGE PETROLEUM
HYDROCARBONS
PCBs POLYCHLORINATED BIPHENYLS

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▲ S07
ND



CAPE LISBURN RADAR INSTALLATION

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FIGURE NO. 1-9
LOWER CAMP TRANSFORMER
BUILDINGS (SS09)
SAMPLE LOCATIONS
AND
ANALYTICAL RESULTS

LEGEND

- ▭ BUILDINGS, STRUCTURES
- 94 SOIL SAMPLE
- ▲ SOIL SAMPLE
- SURFACE DRAINAGE

1500 ppm ANALYTE CONCENTRATION

0000 CONCENTRATIONS ARE ABOVE ACTION LEVELS

ND NO CONTAMINATION DETECTED

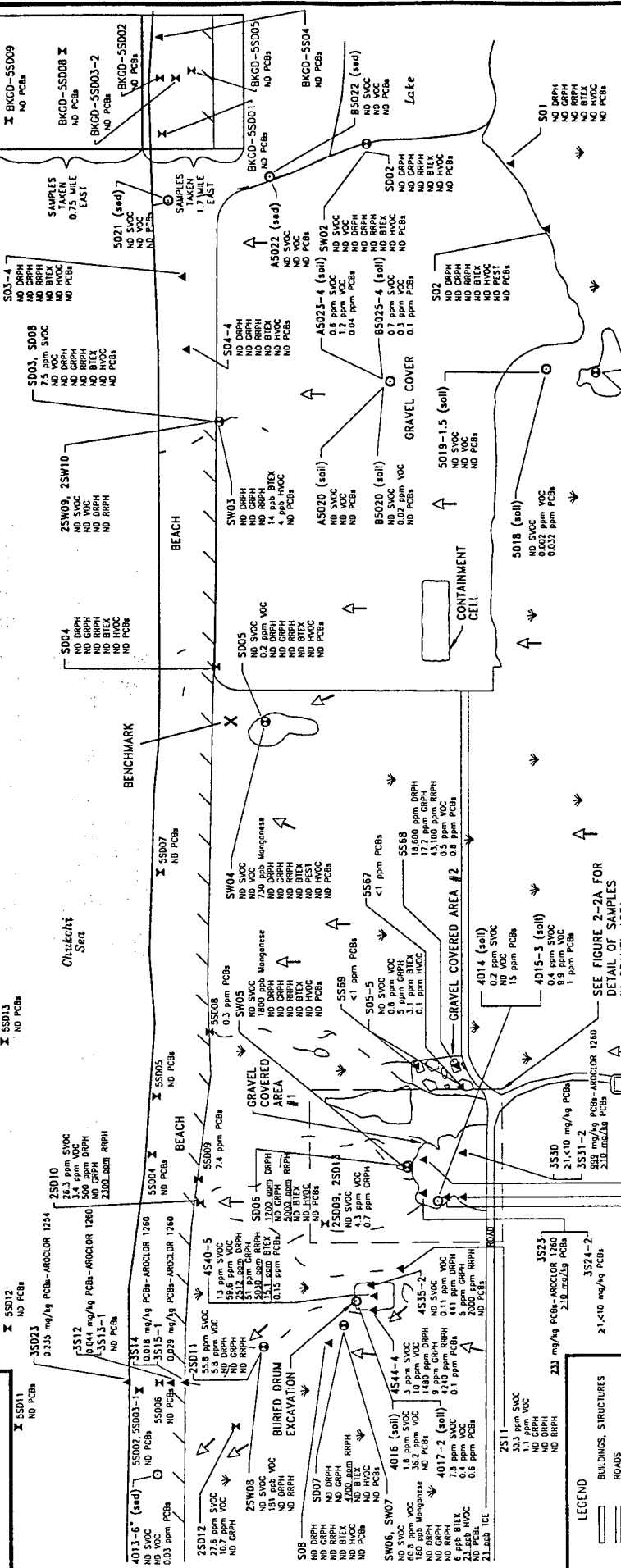
DRPH DIESEL RANGE PETROLEUM HYDROCARBONS

RRPH RESIDUAL RANGE PETROLEUM HYDROCARBONS

PCBs POLYCHLORINATED BIPHENYLS

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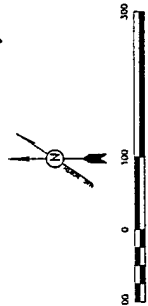
DRAWING No. 9608-010



CAPE LISBURN
RADAR INSTALLATION

USAF 611th CES

FIGURE NO. 1-10
LANDFILL AND WASTE
ACCUMULATION AREA
(LF01) SAMPLE
LOCATIONS AND
ANALYTICAL RESULTS



LEGEND

- BUILDINGS, STRUCTURES
- ROADS
- SOIL SAMPLE
- SEDIMENT WATER SAMPLE
- SEDIMENT AND WATER SAMPLES
- 1982 WOODWARD CLOT SAMPLE
- TUNDRA
- STREAM
- SURFACE WATER
- CULVERT
- GRAVEL PAD BOUNDARY
- SURFACE DRAINAGE
- CITE DATA
- FAB DATA
- 1982 WOODWARD CLOT DATA
- 9/94 FIELD SCREENING SAMPLE DATA

0000	CONCENTRATIONS ARE ABOVE ACTION LEVELS
NO	NO CONTAMINATION DETECTED
SVOC	TOTAL SEMI-VOLATILE ORGANIC COMPOUNDS
VOC	TOTAL VOLATILE ORGANIC COMPOUNDS
DRPH	DIESEL RANGE PETROLEUM HYDROCARBONS
GRPH	GASOLINE RANGE PETROLEUM HYDROCARBONS
RRPH	RESIDUAL RANGE PETROLEUM HYDROCARBONS

BITX	TOTAL BITX COMPOUNDS
HWOC	TOTAL HALOGENATED VOLATILE ORGANIC COMPOUNDS
PEST	PESTICIDES
PCBs	POLYCHLORINATED BI-PHENYLS
TCE	TRICHLOROETHENE

SEE FIGURE 2-2A FOR
DETAIL OF SAMPLES
IN GRAVEL AREA

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The RI/FS determined that IRAs were necessary at two Cape Lisburne sites to prevent migration of site contaminants. The IRAs included excavation and containment of a buried drum area at the site LF01, and installation of an interceptor trench and oil/water separator system at the site ST07.

The IRAs conducted at the Landfill Waste Accumulation Area (LF01) and Spill/Leak #3 (ST07) are strictly interim actions. Using information gained from the IRP investigations conducted during the RI/FS, the Air Force will determine remedial actions that will be required at contaminated sites at the Cape Lisburne installation. Preliminary evaluation of RI data has determined that some sites at the installation may require remediation.

An IRA was initiated in September 1994 at the Landfill and Waste Accumulation Area. A sludge pile/contaminated soils source area that was causing contamination to move downgradient in the natural tundra drainage and towards the Chukchi Sea was removed. Observations made during the 1993 RI activities conducted at the Cape Lisburne installation indicated that the sludge pile area was approximately 200 square feet and extended to a depth of as much as one foot.

During hand excavation in September 1994 at the sludge pile, excavated areas filled with water and a thin layer of floating free oily product. As excavation continued, six semi-crushed drums were exposed. After six cubic yards of contaminated soils had been removed and containerized, a geophysical survey was conducted using a metal detector. The survey indicated the presence of between 20 and 30 buried metal objects, presumably drums, in the immediate area. It became apparent that the source of black surface sludge was liquids that had leaked from the buried drums. Henceforth, the sludge pile area is referred to as the Buried Drum Area.

A continuation of the IRA was scheduled to remove the buried drums prior to the spring thaw, after which surface and active layer water would be likely to fill any excavation. The IRA was initiated in May 1995.

A geophysical survey conducted in September 1994 indicated that buried drums were located beneath an area of approximately 25 feet by 35 feet. In May 1995, excavation of buried drums was initiated using a ripper on a D8 bulldozer to loosen the frozen soil and drums. As drums were exposed, any liquids present in the partially crushed and/or ripped drums were transferred into new drums using long-handled scoops. The loosened soils and drums were pushed up into piles and transferred to a containment cell using a large front-end loader. A metal detector was used during excavation to assist in locating buried drums and determining the direction and depth of further excavation.

Approximately 100 drums were excavated over a period of five days. It was estimated that 50 of these drums were full of liquids and the rest were empty, full of ice, or contained ice and product. Approximately 450 gallons of liquid were recovered during the excavation process. The majority of liquids appeared to be used motor oils; however, the contents of drums varied from heavy lubricating greases to almost clear liquids that may have contained solvents. Liquids that could not be recovered and poured into drums were scooped up with soil using the heavy equipment and transported to the containment

cell. The completed excavation measured approximately 21 feet by 66 feet, with a maximum depth of approximately 6 feet.

Prior to backfilling the excavation with a mixture of gravel and sand from the installation quarry, samples were collected from the bottom and side walls of the excavation, and 80 pounds of calcium peroxide were applied to assist in bioremediation of any residual contaminated soils. During IRA activities in late June 1995, the backfilled area at the site was fertilized and seeded. All excavated soils and debris were placed in a lined containment cell constructed on a gravel-covered area (closed landfill) located approximately 800 feet east of the Buried Drum Area.

1.4 Geology and Hydrology

1.4.1 Geology

The following discussion of site geology is taken directly from *Final Remedial Investigation and Feasibility Study, Cape Lisburne Radar Installation, Alaska*, dated February 1996.

Bedrock found at the Cape Lisburne installation is comprised of sandstone, chert, shale, and conglomerates of the Shublik formation. The bedrock found two miles east of the cape, which consists of granite, schist, limestone, and gneiss, may contribute to the eroded fluvial and alluvial material found at the installation. The surficial Quaternary deposits are composed of coarse and fine-grained deposits associated with moderate to steep sloped mountains and hills. Bedrock exposures are mostly restricted to upper slopes, crestlines, and eroded areas.

The surface deposits of the Lower Camp area are up to 50 feet thick and are dominated by highly permeable talus and alluvial fan deposits consisting of clay, silt, sand, gravel, cobbles, and large boulders. A moderately well-sorted alluvium has been deposited in the channel of Selin Creek. The Upper Camp geology consists of a thin, gravelly layer overlying bedrock; this is typical of the steeper slopes.

The occurrence of permafrost is relatively continuous in the Cape Lisburne area and may reach maximum depths of 600 to 800 feet below grade in zones near large bodies of water. Farther inland, maximum permafrost depths may reach 1,330 feet. Local excavations have encountered permafrost at depths ranging from one to seven feet below grade (AF, 1996d).

1.4.2 Hydrology

Installation drainage is via a suprapermafrost overland flow to diversion channels terminating at the Chukchi Sea. Some runoff from the site is directed to Selin Creek, which also discharges to the Chukchi Sea. Water resources for the site are taken from the shallow water gallery underlying Selin Creek. The U.S. Army Corps of Engineers has indicated that some minor flooding has occurred in the area near the runway. Definitive information regarding the presence of groundwater at the site is not available.

2.0 CONSTRUCTION QUALITY PLAN

The following sections describe the planned site activities for the remedial actions at LF01, SS03, SS08, and SS09. Exact activities and methods may differ from these described due to unforeseen site specific conditions, or changes in the Air Force requirements.

2.1 Mobilization

Equipment and personnel will be mobilized to Cape Lisburne LRRS from Anchorage. All personnel equipment, materials, and supplies will be transported by air. Upon arrival, personnel will unload, unpack, and stage equipment and supplies. Once all equipment and personnel are assembled at the site, work will begin.

Once mobilized to the site and prior to commencing remediation activities, temporary facilities (shelters, treatment and/or decontamination areas) will be constructed, and initial surveying and site inspection will be performed.

2.2 Contaminated Soil Removal at Sites SS03, SS08, and SS09

The areas designated as SS03 (White Alice Site), SS08 (Upper Camp Transformer Building), and SS09 (Lower Camp Transformer Building) will be excavated to remove polychlorinated biphenyls (PCBs) and petroleum hydrocarbon contaminated soil. The areas anticipated for cleanup will be identified and marked at the start of site activities. These planned excavation areas are indicated on Figures 2-1 through 2-3.

Soil removal activities will be conducted to remove soil which exceeds the following criteria:

- PCBs ≥ 1 mg/kg
- TPH (DRO/RRO, C₁₀ - C₃₆) $\geq 10,000$ mg/kg

Upon mobilization, each site will be examined, identifying and marking estimated excavation boundaries. Also at this time, issues directly related to access, demolition, sampling, and excavation will be identified. Any potential planning or mitigation related to these issues will be resolved prior to beginning work.

The buildings, previously used for housing electrical transformers for the upper and lower camp, may be fully or partially removed to allow for the excavation of contaminated soil within the building perimeters and in their close proximity. Both buildings are stick frame construction with corrugated sheet metal exterior panels.

2.2.1 SS03 - White Alice Site Soil Excavation

The White Alice Site is estimated to have up to 650 cubic yards of PCB contaminated soil, consisting largely of gravels. The White Alice Site building is elevated by piers. The soil contamination in the vicinity of this building is indicated as being chiefly around the perimeter of the building with some areas extending under the structure.

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The map shows several buildings and areas with associated environmental data:

- Top Left Building:**
 - 3S21: 6.5 ppm PCBs
 - 2S09-2.5: 380 ppm DRPH
 - 2S10-1.5: ND
 - 2S11-2.5: ND
 - Feature: FRESH GRAVEL PILE
- Top Right Building:**
 - 3S22-1
 - S06: 2 ppm PCBs
 - Feature: DOORS AND STAIRS
- Bottom Left Building:**
 - 2006: 14.0 ppm SVOC, 0.004 ppm VOC, 114.1 ppm PCBs
 - S05: 1 ppm PCBs
 - 2S08: ND
 - 3S18-2
 - 3S17-2
 - 2005: 1.8 ppm PCBs
 - 3S15-1
 - 3S24-1
 - Feature: DOOR AND STAIRS
- Bottom Center Building:**
 - 3S19, 3S23
 - 3S20-1
 - Feature: DOOR AND STAIRS
- Bottom Right Building:**
 - 3S16-1
 - 3S14
 - 3S13-2
 - 2S07-2, 2S13-2: 1.8 ppm PCBs
 - S01: ND
 - Feature: DOUBLE DOORS
 - Feature: DIRT
- Other Labels:**
 - ROAD (top left)
 - ROAD (top right)
 - DOUBLE DOORS (multiple locations)

Legend:

- 2006: 14.0 ppm SVOC, 0.004 ppm VOC, 114.1 ppm PCBs
- S05: 1 ppm PCBs
- 2S08: ND
- 3S18-2
- 3S17-2
- 2005: 1.8 ppm PCBs
- 3S15-1
- 3S24-1
- 3S16-1
- 3S14
- 3S13-2
- 2S07-2, 2S13-2: 1.8 ppm PCBs
- S01: ND
- 3S19, 3S23
- 3S20-1
- 3S21: 6.5 ppm PCBs
- 2S09-2.5: 380 ppm DRPH
- 2S10-1.5: ND
- 2S11-2.5: ND
- S06: 2 ppm PCBs

Scale and Orientation:

- Scale: 0 to 10 FEET
- North Arrow: N

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FIGURE NO. 2-1
WHITE ALICE SITE (SS03)
ANTICIPATED EXCAVATION
AREA

ANTICIPATED
EXCAVATION
AREA

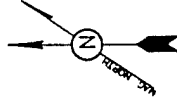
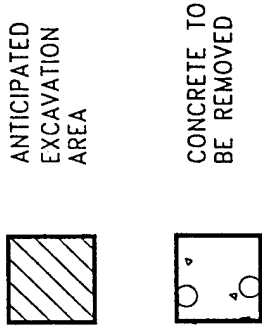
	BUILDINGS, STRUCTURES
	ROADS
	94 SOIL SAMPLE
	SOIL SAMPLE
	WOODWARD-CLYDE SAMPLE
	SURFACE DRAINAGE
0.3 ppm	ANALYTE CONCENTRATION

CONCENTRATIONS ARE ABOVE ACTION LEVELS	NO CONTAMINATION DETECTED	DIESEL RANGE PETROLEUM HYDROCARBONS	POLYCHLORINATED BIPHENYLS
0000	ND	DRPH	PCBs

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DRAWING No. 9608-022

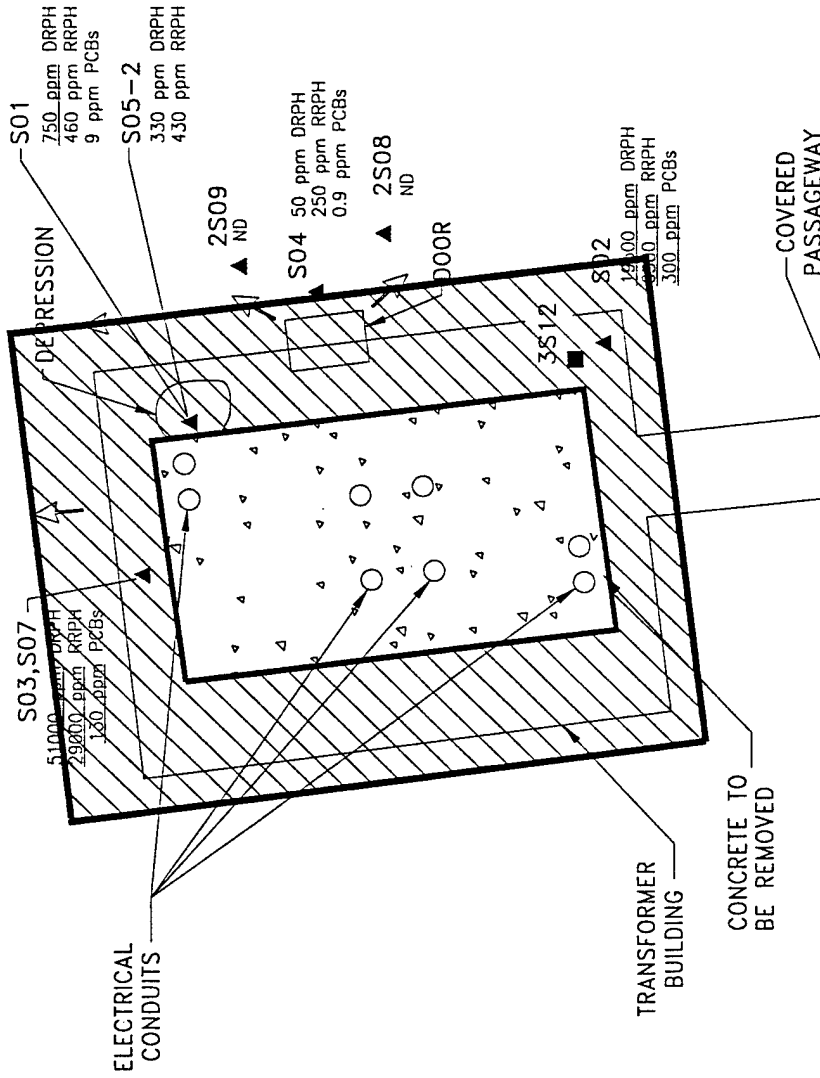
LEGEND



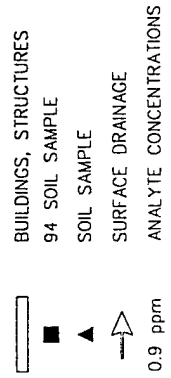
ALASKA REMOTE
RADAR INSTALLATIONS

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FIGURE NO. 2-2
UPPER CAMP TRANSFORMER
BLDG (SS08) PREVIOUS
ANTICIPATED EXCAVATION
AREA



LEGEND



CONCENTRATIONS ARE ABOVE ACTION LEVELS

ND NO CONTAMINATION DETECTED

DRPH DIESEL RANGE PETROLEUM HYDROCARBONS

RRPB RESIDUAL RANGE PETROLEUM HYDROCARBONS

PCBs POLYCHLORINATED BIPHENYLS

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▲ S07
ND

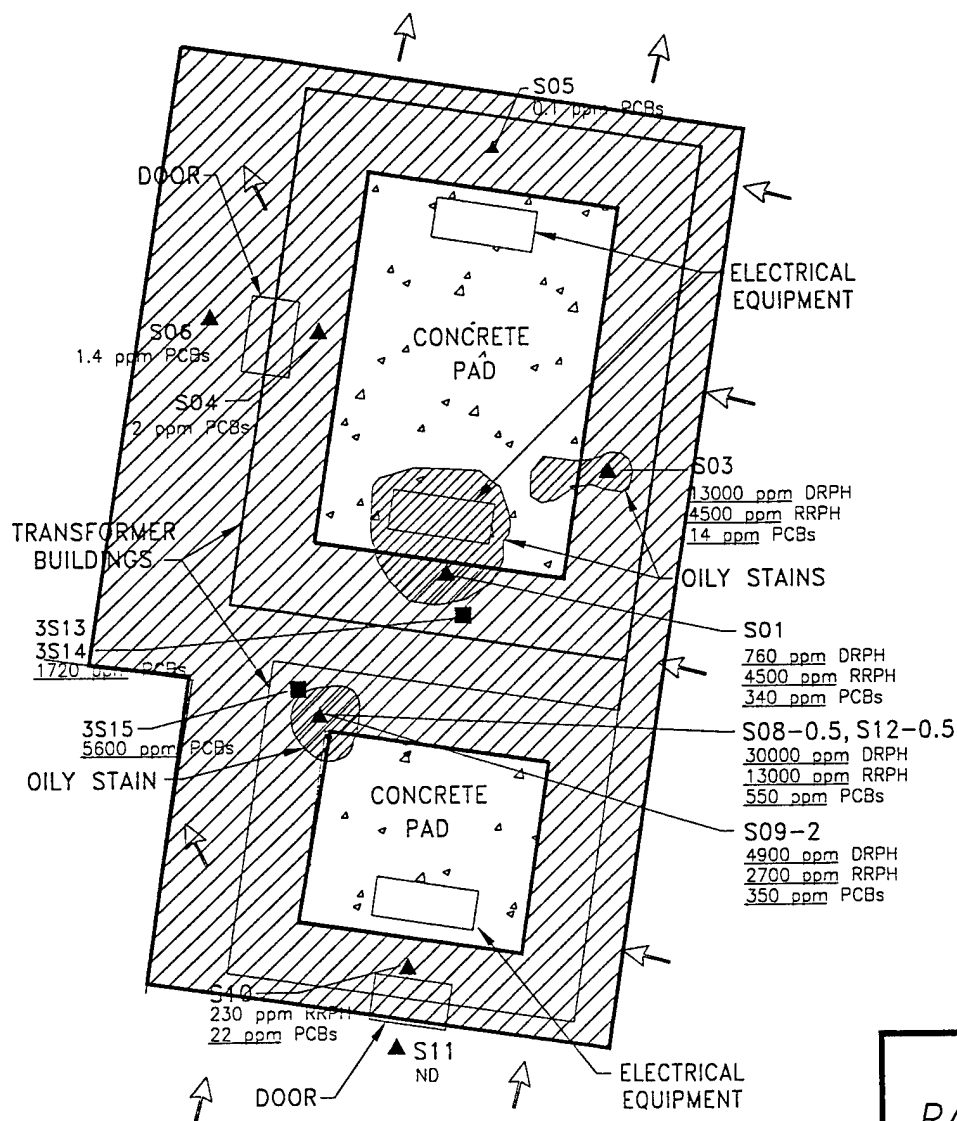
LEGEND



ANTICIPATED
EXCAVATION
AREA



CONCRETE TO
BE REMOVED



LEGEND



BUILDINGS, STRUCTURES



94 SOIL SAMPLE



SOIL SAMPLE



SURFACE DRAINAGE

1500 ppm ANALYTE CONCENTRATION

0000 CONCENTRATIONS ARE ABOVE ACTION LEVELS

ND NO CONTAMINATION DETECTED

DRPH DIESEL RANGE PETROLEUM HYDROCARBONS

RRPH RESIDUAL RANGE PETROLEUM HYDROCARBONS

PCBs POLYCHLORINATED BIPHENYLS

CAPE LISBURN
RADAR INSTALLATION

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FIGURE NO. 2-3
LOWER CAMP TRANSFORMER
BUILDINGS (SS09)
ANTICIPATED
EXCAVATION AREA

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Figure 2-1 shows the anticipated excavation area. After the stairs (and other building parts, as necessary) are removed, a backhoe will be used to excavate the soil around the perimeter of the building and in those areas under the building where the backhoe can reach without damaging the structure. In areas where the backhoe cannot reach, soil will be removed by hand digging.

All excavation will be guided using Ohmicron test kits for PCB detection. Soil will be excavated incrementally until the immunoassay test results indicate the soil contains less than 1 mg/kg of PCBs. After excavation is complete, confirmation samples will be collected in accordance with Section 4.2.

Contaminated soil and concrete will be transported to the area adjacent to the LF01 containment cell and placed in a stockpile dedicated for PCB contaminated materials stockpiling. The stockpile will be clearly labeled, indicating contents, origin, nature of contaminants, size, and date the stockpile was created.

2.2.2 SS08 - Upper Camp Transformer Building Soil Excavation

The Upper Camp Transformer Building is estimated to have 10 cubic yards of gravelly soil and 1.5 cubic yards of concrete contaminated with PCBs. The soil consist largely of gravels. All of the contamination is indicated to be inside the building perimeter.

If demolition of the building is necessary to complete contaminated soil removal, building components as well as concrete foundation slabs will be wipe sampled prior to demolition to identify any contaminated components. Areas with visible contamination will be physically scraped and scrubbed with a degreaser. A ten centimeter square sampling area will be selected from the scrubbed areas. This area will be wiped horizontally and vertically with a hexane soaked swab and placed into a sample container. Random samples will also be collected from areas without visible staining. Results will be reported in micrograms per 100 cm².

Wood and metal debris determined to be free of contamination will be loaded into the dump truck for transport to the Cape Lisburne landfill. Special care will be exercised to avoid disturbing underlying soil. Wood and metals will be separated prior to placement in the landfill if material segregation is a local landfill facility permit requirement. Figure 2-2 shows the anticipated area of excavation; field conditions and findings will determine the final dimensions of the excavation. Contaminated soil will be field screened and excavated as previously described for site SS03. After excavation is complete, confirmation samples will be collected in accordance with Section 4.2.

Any building material shown to be contaminated will be reduced to size sufficient for placement in drums. All drums will be handled according to the methods described in Section 3.4.

2.2.3 SS09 - Lower Camp Transformer Building Soil Excavation

The Lower Camp Transformer Building is estimated to have 40 cubic yards of gravelly soil and 5 cubic yards of concrete contaminated with PCBs. Portions of the building may be removed from this site prior to excavation to provide equipment access.

Contaminated soil will be field screened and excavated as previously described for site SS03. Figure 2-3 shows the anticipated area of excavation; field conditions and findings will dictate the final dimensions of the excavation. After excavation is complete confirmation samples will be collected in accordance with Section 4.2.

2.3 Survey and Removal of Soil and Drums at Site LF01

Site LF01 is the old waste accumulation area. This area is estimated to contain 2,000 cubic yards of contaminated soil. Previous investigations indicate contaminants in the planned excavation area include PCBs and TPH. Figure 1-9 shows these results. It is anticipated that up to 400 drums are buried in this excavation area. Subject drums are expected to contain up to 5,000 gallons of recoverable liquid product, ice, and water. LF01 soil is expected to consist largely of gravel materials. It is also expected that some of the excavated subsurface materials will be frozen.

A geophysical survey will be used to locate areas of magnetic anomalies which could indicate buried drums. These areas will be excavated to confirm the geophysical survey and removal drums. Contaminated soil encountered during excavation activities will be removed and stockpiled. Soil contamination will be identified using field test kits and laboratory analysis. Contaminated soil excavation of each buried drum location will continue to remove soil which exceeds the following criteria:

- PCBs $\geq 1\text{mg/kg}$
- TPH (DRO/RRO, $C_{10} - C_{36}$) $\geq 10,000\text{ mg/kg}$

2.3.1 Geophysical Survey of Site LF01

Initially, a geophysical survey will be performed at LF01 to identify areas with a high probability of buried drums. There are three known buried drum areas as shown in Figure 1-9; labeled "Buried Drum Excavation," "Gravel Covered Area #1," and "Gravel Area #2." The geophysical survey cannot always differentiate buried drums from other buried metallic debris. Therefore, areas suspected to be subsurface drum locations will be delineated with surveyors stakes and ground paint. These areas will be evaluated through careful, incremental excavation.

The geophysical survey will be conducted by use of a cesium vapor magnetometer with a sensitivity of 0.1 gamma over a range of 20,000 to 100,000 gammas. When the sensor passes near a ferrous object, the intensity of the magnetic field changes. The shape of the change, or anomaly, depends on the mass of metal object, its orientation, and its distance from the instrument sensor. A typical ferrous drum anomaly shows magnetometer amplitude variation of up to tens of gammas or more. During drum detection efforts, measurements will be taken on lines spaced of 25 feet apart. Magnetometer measurements will be recorded in one-foot increments along each line.

2.3.2 Site LF01 Excavation

After the geophysical survey is completed and excavation boundaries are marked, a surface sampling survey will be conducted using Ohmicron test kits for PCBs and

laboratory testing for TPH. All areas appearing to be stained with hydrocarbons will be sampled. This survey will be conducted to detect any surface soil which requires removal and stockpiling.

A sample for PCB screening will be collected from each area delineated during the geophysical survey. The PCB screening will be field conducted using Ohmicron test kits described in Section 4.1.7. If a hydrocarbon stained area does not contain PCB concentrations above 1 mg/kg, as indicated by field screening, soil samples will be collected for laboratory analysis for TPH (C₁₀ - C₃₆). These results will be obtained with a rapid laboratory turnaround time. Samples collected each day will be transported by the daily Cape Smythe Air flight to Anchorage. Once in Anchorage, a laboratory courier will pick the samples up and deliver them to the laboratory. Results will be obtained within 48 hours.

If surface contamination is detected in soil samples, the affected area will be delineated with ground paint and surveyors stakes.

Excavation within the LF01 area will be accomplished with a hydraulic excavator and a rubber tire backhoe. Hand labor will be readily available for spotting and hand excavating around buried metal objects, and to facilitate the identification and protection of buried drums. When a buried object is identified, hand excavation will be used until it can be determined whether or not the object is a drum.

Once a drum has been identified, hand excavation will be used to determine the condition of the drum. A sounding method (tap test) will be used to determine the level of contents in drums. Drums with good integrity will be lifted with either the excavator or the backhoe and moved to the drum processing area for sampling of the contents and transfer of the drum contents to a new container. If the drum integrity is questionable (i.e. terminal corrosion, open holes etc.) subject drum contents will be sampled and transferred without moving the drum. Drums that contain product will only be moved from the excavation area if they are intact and structurally sound. The procedure and rationale for sampling drum contents and bulking of drum contents into new containers is discussed in Section 2.3.4 of this document.

During the excavation process, the final disposition of excavated soil will be guided by the results of Ohmicron testing for PCBs and by laboratory analysis for TPH. PCB field tests will be completed at a rate of one per 100 square feet of excavation for every 18 inches excavated. TPH samples will be collected for laboratory analysis at a rate of one per 50 cubic yards of excavated soil. PCB contaminated soil regardless of TPH content, will be segregated into a stockpile. TPH contaminated soil, which does not contain PCBs, will be placed in a separate stockpile. Clean soil will be separately stockpiled to be used for backfill material.

When field screening data indicates that the clean up objectives have been achieved, samples will be collected in accordance with Section 4.2.

The excavation will be backfilled with clean excavation material and material developed from an on-site borrow source, as directed by the Air Force. Disturbed areas will be revegetated using a seed mixture of 60% Bering Hair Grass and 40% Arctared Fescue

applied at 40 lb. per acre. The revegetated areas will be fertilized with nitrogen at 80 lb. per acre, phosphorus at 150 lb. per acre, and potassium at 60 lb. per acre. The re-vegetation effort will take place at the beginning of the 1997 field season (approximately June 1997).

A decontamination pad will be installed on the LF01 site to prevent contaminated materials from spreading off the site. All equipment and personnel leaving the site will use these facilities unless there is an emergency which requires immediate medical attention.

PPE and decontamination procedures outlined in Section 3 of this document will be used for activities at site LF01. Final decontamination of construction equipment used at the LF01 area will be accomplished by pressure washing using Orange Sol detergent. Sampling equipment will be cleaned with Alconox, a laboratory-grade cleaner.

2.3.3 Drum Inventory and Logging

The field team will inventory drums by locating, numbering, visually inspecting, and logging drum characteristics. The purpose of the inventory is to identify and document drum locations and characteristics. In addition, the inventory will allow the field team to identify drums requiring waste characterization and to plan efficient bulking and staging.

Drums will be clearly numbered using paint markers, spray paint, or other permanent device. Drum locations will be mapped with reference to permanent site characteristics. A drum characterization log entry will be made for each drum as it is assigned an identification number. Drum characterization will be maintained by the site superintendent and will include the following:

- Date
- Unique drum number
- Drum size
- Drum type
- General condition (leak status)
- Drum color/markings (if any)
- Description of drum contents
- Amount of material in drum, if applicable
- Waste stream characterization indication
- Location of drum in LF01 grid
- Results of field screen test

2.3.4 Field Screening and Transfer of Drum Contents

During the course of the excavation, if a drum is encountered, the soil surrounding the drum will be removed to the maximum extent possible in order to determine the integrity

of the drum. If the drum appears to have good integrity, it will be lifted using a mechanical drum lifter attached to either the excavator or the backhoe and the drum will be moved to the designated drum staging area. If the drum has questionable integrity it will be punctured pneumatically, in place. Using a glass drum thief, a sample will be extracted. The sample will be placed in a plastic specimen cup with a cover. The balance of the drum contents will be decanted into a new drum. When the damaged drum is empty (or as empty as practical) it will be moved to the designated drum staging area. If the drum is damaged to a point that any handling of the drum would increase the chance a product release, the drum will be overpacked into an 85 gallon salvage drum. The overpacked drum will be transferred to the drum staging area. Subject drum will be sampled from within the overpack drum. Once emptied, the overpacked drum will be removed from the salvage drum, cleaned and placed with other clean/empty drums. The salvage drum will be cleaned and prepared for reuse.

An emergency spill kit (consisting of vermiculite, sorbent boom, sorbent pads and open top drums) will be available at all times during the excavation in the LF01 area.

All drums excavated at site LF01 containing liquids will be sampled. The sample will be characterized in the field to determine waste stream compatibility as described in Section 4.1.7. Related materials will be combined into new containers.

If drums to be removed at Cape Lisburne during this project were truly considered as "drums containing unknown materials," the drums would be subject to strict drum handling guided by the National Institute of Safety and Health (NIOSH) and the Department of Transportation (DOT). However, due to site specific information provided by the Air Force, qualifying assumptions have been made to facilitate drum handling and characterization activities. These assumptions are based on information collected during recent RI/FS investigation activities (USAF, 1995 Interim Remedial Action Report) at LF01 as well as at drum removal projects performed at similar sites (e.g. Tin City LRRS). These assumptions are:

- The likely unknowns are limited to: petroleum products (such as motor oil and diesel); ethylene glycol; chlorinated solvents (perchloroethylene or tetrachloroethylene); PCBs.
- The primary objective of characterization activities is to identify compatible waste streams in order to consolidate wastes for shipment and/or disposal.

Once a drum sample is obtained, it will be taken into the personnel shelter adjacent to the LF01 excavation area. The sample will be "logged in" to document the date, time, and location of the removed "parent" drum. All drums will be clearly labeled with a containerization date, origin, bulking category (as determined by the waste stream characterization process), and approximate volume of contents. Potential hazardous waste will be clearly marked as such, including the words "CERCLA Derived Waste."

The narrative that follows describes the process by which field characterization will be conducted. The principle objective of field characterization is to reduce the number of drums that must ultimately be removed from Cape Lisburne. This will be done by

identifying and combining the contents of the same (or similar) waste streams. The basic process is outlined below. An accompanying flow chart is provided as Figure 2-4.

This process is an adaptation of information provided in "A System of Field Identification and Classification of Commonly Spilled Materials" (DHS HazCat Report, January 1986):

- Using an eye dropper, between 3 and 5 ml of the sample will be added to 10 ml of deionized water — observe whether the sample dissolves, floats, or sinks in the water. Also, observe whether any bubbling or warming of the container occurs, which would indicate that the sample is water reactive.
- If the sample is observed to be water reactive, the material will be placed in a new container and no other material will be mixed in the same container.
- If not observed to be water reactive, cap the sample container and shake vigorously, then observe again -- does layering occur, does the water turn "milky", or does the sample dissolve? NOTE: the mixture may turn milky before it is agitated. This may indicate a pesticide and extreme caution must be taken to avoid any contact or inhalation of the material. A pesticide will usually give a clean white color; motor oil or other hydrocarbons tend to give a brownish color (like coffee with milk in it).
- If the sample sinks, it is likely a chlorinated solvent or PCB.
- If the sample floats, it is most likely a petroleum product. However, pure PCB oil may also float and further testing will be done to determine the presence or absence of chlorine.
- If the sample dissolves, it is likely ethylene glycol. Shaking the mixture will help confirm those observations -- if the water becomes milky (with agitation), it is a petroleum product, specifically oil. If it appears to dissolve, then separates into layers again, it is probably a fuel or a chlorinated solvent.
- If the sample is basically clear, place 10 - 20 ml in a vial and add crystal of iodine.
- If the solution turns red, it is likely perchloroethylene.
- If the solution turns purple, it is likely TCE or carbon tetrachloride, or freon. If the solution turns orange, it is likely freon (the color seen may be due to the iodine, and not the reaction of iodine with the solvent if too much iodine is added). To further identify the material, do flammability testing.
- Place a small amount of the sample in a closed cup flashpoint tester. Cycle the tester and record the results. Flammable is to be considered a flashpoint of up to 141°F. Combustible is to be considered a flashpoint of between 141° and 200°F. Non-combustible is to be considered a flashpoint of above 200°F.

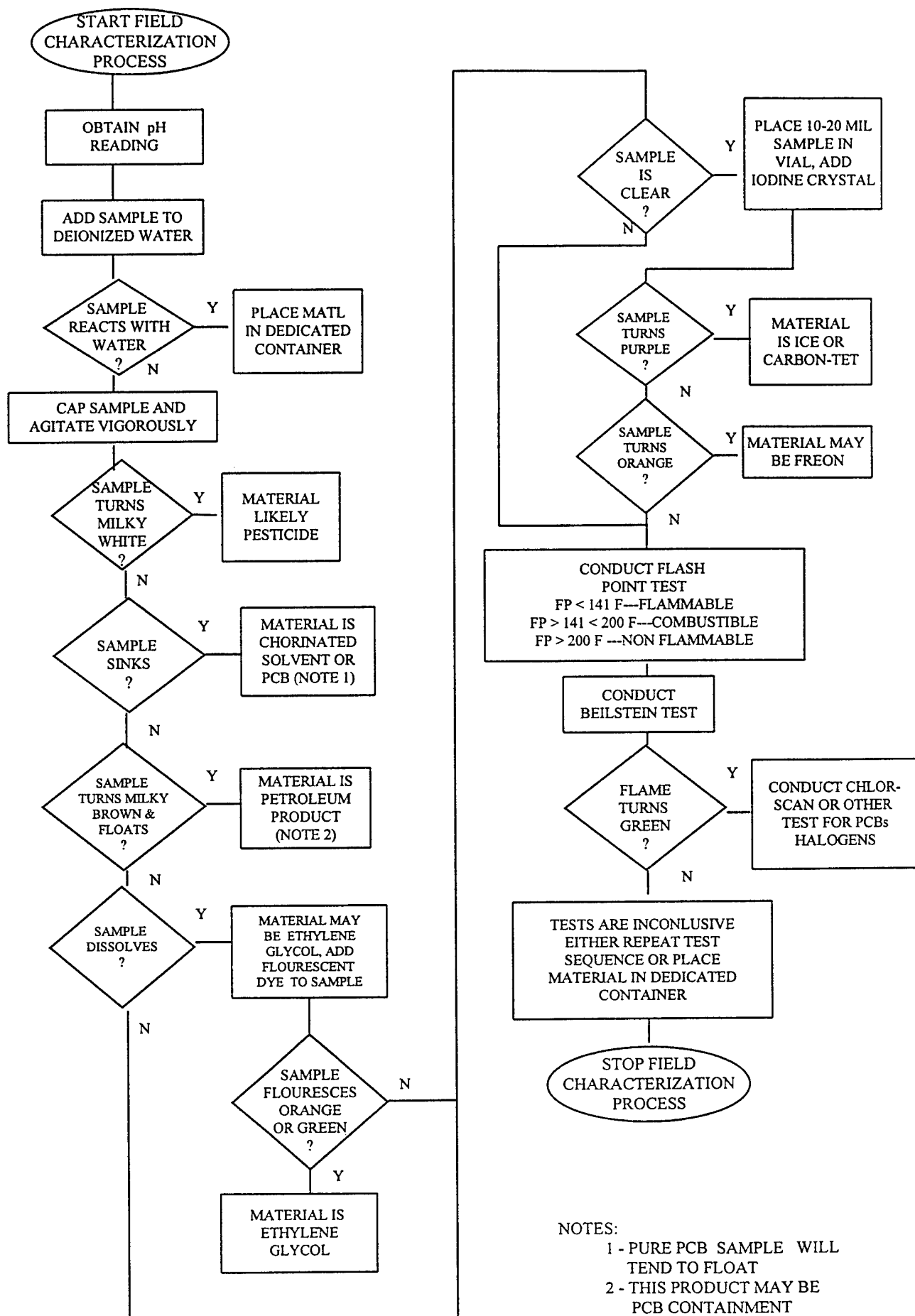


Figure 2-4 Field Characterization of Drum Contents

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- All samples will be tested by dipping a copper wire in the sample and placing the dipped portion of the copper wire in a flame. If a green flame is observed the sample will be tested with the Dexsil Products' Clor-N-Oil® test kits to test for the presence of PCBs, and Hydroclor-Q® to test for the presence of halogenated hydrocarbons.
- Observe the sample color. If the sample fluoresces yellow, orange, or green the sample is most probably ethylene glycol.
- All samples will be returned to the container from which they were extracted.

All drum contents will be transferred to new drums based on the results of field characterization. Only those drum contents with the same characteristics will be placed together. If distinct patterns (in labeling, or other drum characteristics) are observed during the drum discovery process, it will be considered during waste stream characterization. All new drums will be clearly marked with labeling guidance provided in Section 4 of this document.

Water encountered in drums suitable for filtering and discharge will be processed through an activated carbon filtration system. A prefilter system will be set up so that water flows through a gross particulate filter, followed by a 4 or 5 micron filter, then into the carbon canister. Ice will be allowed to liquefy prior to entering the treatment unit. After filtration, the water will be stored in drums pending laboratory analysis. If analytical results indicate subject water is within the following parameters, it will be discharged on the ground at the excavation site.

- BTEX: 10 ppm
- benzene: 5 ppb
- PCBs: 0.5 ppb
- no visible sheen

The total BTEX and benzene levels are based on discharge requirements agreed upon by ADEC and the Air Force for a similar project at Kotzebue. A copy of a memo from the Air Force describing the process of filtering contaminated water and the acceptable discharge levels is included in Appendix B. The Alaska Water Quality Standards, 18 AAC 70, do not provide PCB discharge standards. Therefore, the PCB discharge level is the Maximum Contaminant Level for a public water system per the Alaska Drinking Water Standards, 18 AAC 80. If the analytical results indicate the water is outside the parameters shown above, the water will be shipped to a private treatment, storage, and disposal facility (TSDF).

2.3.5 Drum Cleaning and Placement at Site Landfill

The contents of drums removed from LF01 area will be emptied as stated in the previous section. Each drum will be de-headed using a pneumatic drum de-header. The de-header will cut the drum just below the top chime of the drum. If the drum is too disfigured to

be cut with the pneumatic de-header, the drum will be de-headed with a hand held pneumatic chisel.

After the drum is de-headed, it will be swabbed clean using sorbent pads that are attached to a broom. A drum will be considered clean when there is no free product remaining in the drum. The used sorbent pads will be placed in red 10 mil plastic bags. Sorbent pads will be separated by the same process used to bulk drum contents. Sorbent pads containing compatible materials will be placed in the same containers. Other disposable materials of like waste streams will also be placed in the compatible containers. Bagged sorbent pads will be subsequently placed in open top drums and sealed to prepare for shipment to a private TSDF.

The de-headed and cleaned drums will then be replaced in LF01 excavations. If possible, these drums will be buried in a single location, and their relative position will be documented. All sorbent pads, PPE, and other material will be collected and packaged for transport to the TSDF.

2.4 Soil Stockpiling

As stated in previous sections of this document, soil exceeding 1 mg/kg of PCBs and 10,000 mg/kg of TPH will be placed in stockpiles. One stockpile will be designated for PCB contaminated material and/or material contaminated with PCBs and TPH; the other stockpile will be material with TPH contamination only. As there is the potential that more than one TPH stockpile will need to be constructed, adequate liner and covering material will be on hand for all necessary stockpiles.

All stockpiles will be identical in construction. A 30 mil puncture resistant HDPE bottom liner will be placed upon a surface which has been leveled and from which all sharp objects have been removed to prevent puncture of the liner. A berm of native material, at least 2 feet high, will be placed around the perimeter of the stockpile. The bottom liner will extend continuously over the top of the berm on all sides of the stockpile. If required, additional sheets will be joined by heat welding them together. Repairs to existing stockpile liners will be done in the same fashion.

Soil will be placed on the bottom liner within the berms. The installed soil will be placed up to approximately 5.5 feet in height, at an angle of less than 45 degrees from the bottom of the inside surface of the berm to the crest of the placed soil. The equipment placing the soil will not drive on the bottom liner. Placement will be accomplished by keeping the bottom liner rolled up within 6 feet of the leading edge of the stockpiled soil so the equipment can reach over the rolled liner and place the soil. As the materials are placed, the bottom liner will be unrolled to expand the capacity of the stockpile.

Once the material is placed in the lined, bermed area, a top cover will be placed over the stockpile. The cover will be 20 mil puncture resistant HDPE. Before the cover is placed, the stockpile will be visually inspected and any sharp particles that could potentially puncture the cover will be removed. The cover will continue uninterrupted over the entire stockpile and berms. The bottom edge of the cover will be secured by placing

additional native soil at the crest of the berm. The cover will also be secured using burlap sand bags. At least one sand bag for each 25 square feet of top cover will be placed.

2.5 Stockpiled Soil Sampling

After stockpiling contaminated soil is completed and before placing the covers, the stockpiles will be sampled in accordance with parameters shown in Table 4-27. The samples will be collected and analyzed in accordance with the procedures presented in Section 4.2.

2.6 Soil Sampling for Confirmation of Cleanup Levels in Excavations

All of the excavation sites (LF01, SS03, SS08, and SS09) will be sampled prior to backfill placement to confirm field screening results and to ensure action levels have been met. The samples will be collected and analyzed in accordance with the procedures presented in Section 4.2.

2.7 Sampling of Bulk Drum Contents

To establish shipping and disposal requirements; a discrete sample will be collected from each drum of bulked liquid from excavated drums. Each sample will be analyzed as shown in Table 4-27. Samples will be collected and analyzed in accordance with the procedures presented in Section 4.2.3. Analytical data will be provided to Philip Environmental to profile and manifest the waste for disposal.

2.8 Containerized Waste Manifesting and Transportation

Waste material generated as a result of this project and previous projects will be manifested (when appropriate), transported, and disposed of. Transportation of drums will occur as soon as the drummed contents have been characterized and manifested. In general, the following materials will be included in the activity:

- The 25 drums of liquids remaining on site from previous work at this location
- All liquids transferred from excavated drums, except water treated and discharged on site
- All contaminated sorbent pads, PPE, sampling instruments and other solids not eligible for placement in either the site landfill, the soil stockpiles, or the site incinerator

Philip Environmental has been contracted to prepare the manifests and labels, and transport and dispose of the waste. The manifests will be prepared as soon as the analytical data is available for that purpose. All manifests will be signed by the authorized government representative. A copy of the manifests will be provided to the project manager.

2.9 Backfill and Site Restoration

All of the excavations will be backfilled using either existing or developed borrow material. Backfill material will be comprised of gravel and/or a sand and gravel mixture. The Air Force will indicate the areas to be developed for backfill.

All excavations will be filled with borrow material to match existing grades and lines. In the case of the SS03, SS08, and SS09 sites, the gravel material will be placed to the surface. In the case of the LF01 site, the uncontaminated surface soil, initially separated during excavation, will be returned to the surface of the excavation. It is expected that these materials are more conducive to re-vegetation. If this is not the case or there is a shortage of materials conducive for re-vegetation, the Air Force will indicate where suitable materials may be developed for this purpose.

This area will be revegetated using a seed mixture of 60 percent Bering Hair Grass and 40 percent Arctared Fescue applied at 40 lb. per acre. The revegetated areas will be fertilized with nitrogen at 80 lb. per acre, phosphorus at 150 lb. per acre and potassium at 60 lb. per acre. The revegetation effort will take place at the beginning of the 1997 field season (approximately June of 1997).

Soil stockpiles will be checked and the covers will be repaired as necessary during the 1997 site visit. If additional sandbags are needed, they will be filled and placed. During the 1997 site visit, the excavations at sites SS03, SS08, and SS09 will be checked for depressions. If depressions are noted, additional material will be placed to conform to existing grades and/or other natural site features. No equipment will be mobilized to support this activity. Equipment anticipated to already be on-site will be used for this activity.

3.0 HEALTH AND SAFETY PLAN

The work at Cape Lisburne LRRS requires the demolition of two buildings, excavation of contaminated soils, excavation of full or partially full drums, transfer of drum contents, construction of soil stockpiles and backfilling of excavations. The expected drum contents include: used oils with metals, used oils with PCB, used oils with solvents, antifreeze with water and antifreeze with oils. Expected soil contaminants include PCBs and petroleum hydrocarbons. Each site will be evaluated and constantly monitored for potential chemical and physical hazards. Site control, personal protective measure, levels of protection, safety indoctrination, safety meetings and other health and safety issues, as well as standard work practices, are discussed in the Program Health and Safety Plan (AF 1995e).

3.1 Chemical Hazards

Summaries of chemical contaminants reported during the remedial investigation of various remedial actions and RI/FSs at the sites are provided on Table 3-1. Chemicals other than those discussed in this section may be present at the site. If other contaminants are identified during the course of remedial field activities, additional hazard assessments will be made: changes in monitoring requirements will be instituted; PPE will be reevaluated and changed as needed; additional safety measures will be incorporated; personnel will be informed; and action levels will be implemented, as appropriate. Table 3-2 presents a summary of toxicity, time frame of exposure, routes of entry, target organs, and symptoms of exposure.

3.1.1 Petroleum Hydrocarbons

Petroleum hydrocarbons have been detected in soils at concentrations of up to 43,100 mg/kg. Petroleum hydrocarbons consist of a complex mixture of aromatic paraffinic, olefinic, and naphthenic hydrocarbons. The analytical method used to detect petroleum hydrocarbons was not specific and components could range from gasoline to heavy oil. However, toluene, ethylbenzene and xylenes were detected at low levels during previous field activities, so it is believed that the petroleum hydrocarbons present are associated with diesel fuel.

Characteristics: Flammable, mildly toxic.

Petroleum hydrocarbons are mildly toxic by ingestion and inhalation and are severe eye and skin irritants. Symptoms of exposure are similar to those of other refined petroleum products and include depression of the central nervous system and intoxication. Prolonged dermal contact can produce dermatitis. Aspiration of the liquid or inhalation of high concentrations in air can cause severe pulmonary injury.

Table 3-1. Summary Of Contaminants

Contaminant	CAS Number	Media	Maximum soil Concentration	Recommended Exposure Limits ^a
Diesel Range Petroleum	77650-28-3	Soil	51,000 mg/kg	as low as feasible ^b
Residual Range Petroleum	NA	Soil	43,100 mg/kg	NA
Gasoline Range Petroleum	8006-61-9	Soil	96 mg/kg	as low as feasible
Toluene	108-88-3	Soil	10 mg/kg	100 ppm
Ethylbenzene	100-41-4	Soil	8 mg/kg	100 ppm
Xylenes	1330-20-7	Soil	43 mg/kg	100 ppm
PCB	1336-36-3	Soil	<4 mg/kg	0.001 mg/m ³
Ethylene Glycol	107-21-1	Drums	unknown	none published

From NIOSH Pocket Guide to Chemical Hazards, June 1994.

- a. Recommended exposure limits (RELs) are time-weighted average concentrations for up to a 10-hour work day during a 40-hour work week.
- b. Guidance for diesel and aviation gas will follow that of gasoline.

Table 3-2. Toxicity Characteristics

Substance	Hazard Parameter	PEL ^a	Action Level	Primary Route Of Entry	Target Organs	Symptoms Of Exposure
Ethylene Glycol	Toxic	None published	None published	Absorption, inhalation, ingesting	Eyes, Gastrointestinal Tract (GI)	Irritation, dark urine, dermatitis
TPH	Toxic	None published	None published	Inhalation, ingestion, absorption	Eyes, Skin, Central Nervous System	Skin/Eyes irritation, Intoxication
PCBs	Toxic, suspected carcinogen	0.5 mg/m ³	0.30 mg/m ³	Inhalation, ingestion, absorption, skin/eye contact	Skin, Eyes, Liver	Irritation of skin and eyes,

From NIOSH Pocket Guide to Chemical Hazards, June 1994.

a. The permissible exposure limits (PELs) are time-weighted average concentrations that must not be exceeded during any 8-hour work shift of a 40-hour work week.

Hazard Assessment: Apparent hazard is low.

Exposure to petroleum hydrocarbons in large uncontained quantities or concentrations in the air is not expected. It is not anticipated that free product or saturated soils will be encountered during excavation activities. Heavier petroleum hydrocarbons are not extremely volatile, so inhalation of vapors is not anticipated to represent a significant risk and the temperatures encountered will further reduce the likelihood of exposure of gases and vapors of the petroleum hydrocarbons. Ingestion or inhalation of contaminated particulate, however, does present a health risk.

3.1.2 Polychlorinated Biphenyls

The type of PCBs is suspected to be Aroclor 1260, a dielectric fluid typical of PCB-containing transformer oils.

Characteristics: Regulated to be toxic and alleged to be carcinogenic.

PCBs are a series of isomers that vary in physical form from a mobile, oily liquid to a white crystalline powder and hard resins. They may affect the skin and liver. Symptoms of exposure include nausea, vomiting, loss of weight, jaundice, edema, and abdominal pain. The permissible exposure limit for PCBs is 0.5 mg/m³.

Hazard Assessment: Apparent hazard is low to moderate.

Personnel may be exposed to PCBs during construction activities at LF01, Upper Camp Transformer Building (SS08), the Lower Camp Transformer Building (SS09), and the White Alice Site (SS03). Section 3.7 describes PPE requirements.

3.1.3 Ethylene Glycol

Ethylene glycol, used as antifreeze, is expected to be found in the buried drums at LF01. Concentrations in the drums are not known at this time.

Characteristics: Toxic by ingestion.

Ethylene glycol is a colorless, sweet-tasting, hygroscopic liquid. It is highly toxic. It may affect the central nervous system, skin, eyes, and the respiratory system. Symptoms of exposure include nausea, vomiting, central nervous system depression, skin sensitization, and abdominal pain. The lethal dose for humans is reported to be 100 ml. The recommended exposure limit for ethylene glycol is 50 ppm per NIOSH Pocket Guide to Chemical Hazards, June 1994 Edition.

Hazard Assessment: Apparent hazard is low to moderate.

Personnel may be exposed to ethylene glycol during drum handling and bulking activities at LF01. Due to the toxic nature of this substance, caution should be taken to limit unnecessary dermal contact and ingestion (see Section 3.7).

3.1.4 General

The primary risk would be inhalation of soil contaminated by PCB oil. Respiratory protection that provides for filtration of particulates will afford adequate protection. PCBs are non-volatile. For additional information see Sections 3.1.2 and 3.7.

3.2 Physical Hazards

Due to the hazards of working with rusted drums, all personnel will have received or be current for tetanus immunizations.

3.2.1 Heavy Equipment

The heavy equipment used at Cape Lisburne will consist of a hydraulic excavator, rubber tire backhoe, loader, and dump truck. All equipment will be inspected by a competent mechanic and verified in good working order prior to being put in service on the project. This inspection will include operation of backup alarms, brakes, emergency brakes, horns, and air and hydraulic systems. Any deficiencies noted will be corrected before the equipment is put into service.

Equipment operators must possess verified experience on the specific piece of equipment they will operate, prior to operating the equipment. On-the-job training of equipment operators will not be permitted. All equipment in use will have one ground personnel assigned to the piece of equipment to act as a spotter. The spotter will be responsible for keeping other personnel out of the active working area of the piece of equipment, verifying overhead clearances, and communicating directly with the operator either by hand signals or verbally. The spotter will remain within direct eye contact with the equipment operator at all times.

3.2.2 Excavations

Prior to opening an excavation, underground utilities will be located and protected. Excavations will be inspected as necessary for safety considerations. If the possibility of cave-ins, slides, or other hazardous conditions becomes apparent, all workers will be removed from the excavation until necessary safety precautions have been implemented.

All excavations that require entry by personnel will be sloped at 1.5 horizontal to 1 vertical and will be constructed following all requirements of 29 CFR 1926.650. No shoring is anticipated for any of the excavations on this project. Any open excavations will be backfilled as soon as possible. A barricade system will be erected where excavations must remain open and unattended. All digging permits will be provided by the Air Force, prior to commencing excavation.

3.3 Temperature Stress

3.3.1 Cold Stress

Due to the climate and time of year work is to be performed at Cape Lisburne LRRS, cold stress (frostbite and hypothermia) is of great consideration. Cold stress prevention is

addressed in detail in the Program Health and Safety Plan (AF 1995e), which includes the following:

- Wear adequate layers of clothing
- Recognize the combination of wind and low temperatures (wind chill Index)
- Drink adequate fluids
- Have adequate work-rest regimens with heated rest areas
- Train employees in recognizing hypothermia symptoms in themselves and others
- Hold safety meetings at the beginning of each shift
- Work using the buddy system

3.3.2 Heat Stress

Although elevated temperatures are not expected during execution of the work at Cape Lisburne LRRS, there is a possibility that personnel working while wearing personal protective equipment are at risk of heat stress. Heat stress is caused by a number of interacting factors, including environmental conditions, clothing, workload, and the individual characteristics of the worker. Heat stress can result in health effects ranging from transient heat fatigue to serious illness or death.

Physical monitoring for heat stress will not be performed on this project. The weather conditions are not expected to present a serious heat stress risk. Project personnel will be kept alert to the symptoms of heat stress, as described in the Program QAPP.

Heat stress will be minimized by consuming one quart of water per hour, scheduling work during cooler hours, providing periodic rest breaks. Avoid consumption of alcohol and alcoholic beverages during non-work hours. Work breaks should be planned to prevent stress-related accidents and fatigue.

3.4 Biological Hazards

3.4.1 Bears

The most notable biological hazard at Cape Lisburne LRRS is the presence of bears. They are known to frequent the beach and landfill areas. The following precautions should be taken to avoid confrontations with these unpredictable and aggressive wild mammals. In general, if a bear approaches the work site, at a distance of 100 yards or closer, all personnel will be directed to seek the protection of heavy equipment or other vehicle enclosures, or building enclosures. Other rules to be mindful of include:

- Do not work or wander off alone, especially near the beach or at night
- Site superintendent to have custody of protective fire arm

3.5 Drum Handling

The LF01 site will involve the handling of buried drums that may be full, partially full or empty. These drums may be intact, partially intact or in pieces. The primary safety consideration for this element of work is the potential for contact with unidentified materials that may be hazardous. For this reason, all work in this area will be performed in level C PPE as indicated in Section 3.7.

Other considerations in handling drums is the potential for sharp edges, heavy lifting and falling or rolling drums. A site orientation will be conducted at the start of work on this site and will be attended by all contractor and subcontractor personnel. At this orientation the topics covered will include:

- Safe lifting techniques
- Mechanical lifting techniques
- Working around sharp objects
- Slip trip and fall hazards in excavations
- Buddy system
- First aid availability and reporting
- Do not sit, stand, or walk on drums
- Limiting presence of personnel at the drum handling areas to those essential to the work being done
- Use of intrinsically safe tools when working with potentially flammable substances
- Relieving/releasing drum contents slowly and evenly to account for any pressurization of the drums

All persons reporting to or requesting access to this site after the work has begun will be required to acknowledge this information before access will be allowed. No persons will be allowed within the controlled area for any reason with out proper PPE and orientation.

All tools to be used on the drums, including bung wrenches, drum piercers, etc., will be nonsparking if drums or containers being operated on are full or partially full of product.

3.6 Hazardous Material Bulking

The following guidelines will be followed to ensure the safe transfer of hazardous materials by pumping:

- Avoid contact with hazardous materials to the extent possible by donning proper PPE (see Section 3.7) and employing safe work practices.
- Follow established guidelines for PPE.
- To the extent possible, minimize handling product or transferring multiple times.

- Inspect drum into which product will be transferred to check for damage or possible leakage.
- Make sure all hoses, casings, fittings, and gaskets are compatible with the material being pumped.
- Inspect hose lines, fittings, and valves to ensure everything is intact and in good condition.
- Use duct tape, whip checks, etc. as appropriate on all hose connections.
- Have rags and sorbent pads on hand to promptly clean up any spill.
- Ensure that all electrical equipment used is properly grounded.

3.7 Personal Protective Equipment

All work conducted at all sites as a part of this project will be performed, at a minimum, in level D PPE. Level D will consist of the following:

- Chemical resistant Beta Haz-Max chemical resistant boots with steel toes
- Coveralls
- Hardhat
- Eye protection
- Ear protection

PPE requirements for product transfer, drum contents characterization, full or partially full drum handling, and working in PCB contaminated areas will be performed in level C. Level C will consist of the following additional requirements:

- Full face, air purifying, respirator combination with HEPA filters
- Tyvek coveralls with taped wrist and ankle protection
- "Best N-Dex" inner and nitrile outer gloves

A minimum of two level B emergency use only kits will be available at all times.

3.8 Emergency Facilities

There are no emergency facilities available at the Cape Lisburne site. All emergency services will be provided (if required) via medi-vac resources. The nearest hospital is:

Kotzebue Hospital: (907) 552-9637

To sequester emergency services, operations personnel and/or project staff will call 911 on a local facility telephone. The assigned project superintendent is trained in CPR and first aid procedures.

3.9 Site Control

All four sites (LF01, SS03, SS08, SS09) will have established site controls to limit the access to the site during project activities. Site access will be through the contamination reduction zone. This corridor will be delineated by physical barriers, as necessary, placed before any site excavation or demolition begins. The responsible person for each site will enforce site controls and ensure that no personnel or equipment enters or leaves the site without proper authorization. Typically the responsible person on each site will be the site superintendent however, in his absence a temporary replacement may be appointed.

3.10 Decontamination

For most activities at this site decontamination procedures will be followed to minimize the spread of contaminants. Personnel and equipment decontamination will be performed as described in the following subsections. Rinsate generated during decontamination will be handled in a similar manner as water encountered in drums during field characterization as described in Section 2.3.4.

3.10.1 Personnel Decontamination

All personnel decontamination will be performed in the contaminant reduction zone (CRZ), which will be located at the constructed decontamination areas at sites SS03, SS08, SS09, and LF01. The following sequence of decontamination steps will be performed:

- Proceed to the boot wash area in the CRZ and wash and rinse boots.
- Proceed to a wash station and clean outer gloves.
- Wash protective clothing from head to toe, in a manner that moves from top to bottom.
- Rinse gloves and protective clothing thoroughly.
- Remove outer clothing in a way that keeps the inner surfaces clean and free from contact with outer contaminated surfaces.
- Place outer clothing in used PPE storage container for future disposal.

3.10.2 Equipment Decontamination

All equipment decontamination will be performed in the constructed decontamination areas located at sites SS03, SS08, SS09, and LF01. The following sequence of decontamination steps will be performed:

- Move heavy equipment and/or vehicles to LF01 for decontamination activities.
- Scrape, brush, or wipe all heavy contamination from surfaces.
- At Site LF01, using a pressure washer, spray off equipment from the top down.

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4.0 SAMPLING AND ANALYSIS PLAN

The Sampling and Analysis Plan (SAP) is comprised of two main sections: the Quality Assurance Program Plan and the Field Sampling Plan.

4.1 Quality Assurance Program Plan (QAPP)

An essential component of an effective multidisciplinary field investigation program is a definitive QA/QC program. A comprehensive and well documented QA/QC program is necessary to obtain data that is scientifically and legally defensible, and to meet the requisite levels of precision and accuracy. Specified criteria are designed to produce useful data and to reduce the potential for error associated with field and laboratory activities. The ability of this data collection activity to meet this objective is provided through the establishment of data quality characteristics which include precision, accuracy, representativeness, comparability, and completeness.

The QA/QC descriptions and requirements were taken from the AFCEE Quality Assurance Project Plan Version 1.1 (USAF 1996) and from Columbia Analytical Services' QA Manual (Columbia Analytical Services 1996).

4.1.1 Data Categories

The two general categories of data used for this project are defined as: (1) screening data and (2) definitive data.

Screening data are generated by rapid methods of analysis with less rigorous sample preparation, calibration and/or QC requirements than are necessary to produce definitive data. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data may provide analyte identification and quantitation, although the quantitation may be relatively imprecise.

Screening methods are confirmed by analyses that generate definitive data. Confirmation samples will be selected to include both detected and nondetected results from the screening method.

Definitive data are generated using rigorous analytical methods, such as approved EPA reference methods. The data for this project will be generated in an off-site laboratory. Data are analyte-specific, and both identification and quantitation are confirmed. These methods have standardized QC and documentation requirements. Definitive data are not restricted in their use unless quality problems require data qualification.

4.1.2 Precision, Accuracy, Representativeness, Completeness, and Comparability

The basis for assessing each of these elements of data quality is discussed in the following subsections. Precision and accuracy QC limits for each method and matrix are identified in the tables in Section 4.1.8.

4.1.2.1 Precision

Precision measures the reproducibility of measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Analytical precision is the measurement of the variability associated with duplicate (two) or replicate (more than two) analyses. The laboratory control sample (LCS) is used to determine the precision of the analytical method. If the recoveries of analytes in the LCS are within established control limits, then precision is within limits. In this case, the comparison is not between a sample and a duplicate sample analyzed in the same batch, rather the comparison is between the sample and samples analyzed in previous batches. Total precision is the measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations. Field duplicate samples and matrix duplicate spiked samples will be analyzed to assess field and analytical precision, and the precision measurement is determined using the relative percent difference (RPD) between the duplicate sample results. The formula for the calculation of precision, defined as RPD, is:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where:

RPD = Relative percent difference.

*D*₁ = First sample result.

*D*₂ = Second sample result.

For replicate analyses, the relative standard deviation (RSD) is determined. The formula for the calculation of RSD is:

$$RSD = \left(\frac{S}{\bar{X}} \right) \times 100$$

where:

RSD = Relative standard deviation

S = Standard deviation

$\bar{X}$ = Mean

The mean is calculated as:

$$\bar{X} = \frac{\left(\sum_{i=1}^n x_i \right)}{n}$$

where :

$\bar{X}$ = Mean

x_i = Individual measurement

n = Total number of measurements

The standard deviation is calculated as:

$$S = \left(\frac{\sum (x_i - \bar{X})^2}{(n-1)} \right)$$

where:

S = Standard deviation

x_i = Individual measurement

$\bar{X}$ = Mean

n = Total number of individual samples

4.1.2.2 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.

Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS to a control limit. For volatile and semivolatile organic compounds, surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed. Analysis of performance evaluation (PE) samples will also be used to provide additional information for assessing the accuracy of the analytical data being produced.

Both accuracy and precision are calculated for each analytical batch, and the associated sample results are interpreted by considering these specific measurements. The formula for calculation of accuracy for a spiked compound in a pure matrix, defined as percent recovery (%R), is:

$$\%R = \left(\frac{X_{\text{measured}}}{X_{\text{true}}} \right) \times 100$$

where :

$\%R$ = Percent recovery

X_{measured} = Measured value

X_{true} = True value

The formula for %R of a spiked compound in a sample matrix is:

$$\%R = \frac{\text{Matrix Spike Concentration} - \text{Sample Result}}{\text{Quantity of Analyte Added}} \times 100$$

4.1.2.3 *Representativeness*

Objectives for representativeness are defined for each sampling and analysis task and are a function of the investigative objectives. Representativeness will be achieved through use of the standard field, sampling, and analytical procedures. Representativeness is also determined by appropriate program design, with consideration of elements such as sampling locations.

4.1.2.4 *Completeness*

Completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples. Completeness is calculated and reported for each method, matrix and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all non-qualifying results. The requirement for completeness is 95 percent for aqueous samples and 90 percent for soil samples. For any instances of samples that could not be analyzed for any reason (holding time violations in which resampling and analysis were not possible, samples spilled or broken, etc.), the numerator of this calculation becomes the number of valid results minus the number of possible results not reported.

The formula for calculation of completeness is:

$$\% \text{ Completeness} = \frac{\text{Number of Valid Results}}{\text{Number of Possible Results}}$$

4.1.2.5 *Comparability*

Comparability is the confidence with which one data set can be compared to another data set. The objective for the QA/QC program is to produce data with the greatest possible degree of comparability. The number of matrices that are sampled and the range of field conditions encountered are considered in determining comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions and using standard and comprehensive reporting formats. Complete field documentation using standardized data collection forms will support the assessment of comparability. Analysis of performance evaluation (PE) samples and reports from audits will also be used to provide additional information for assessing the comparability of analytical data produced among subcontracting laboratories. Historical comparability will be achieved through consistent use of methods and documentation procedures throughout the project.

4.1.3 Method Detection Limits, Practical Quantitation Limits, and Instrument Calibration Requirements

4.1.3.1 Method Detection Limits

The method detection limit (MDL) 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 will establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project. MDL studies will be accomplished on an annual basis. The laboratory will provide the MDL studies to AFCEE at the beginning of the project and upon request. Results less than the MDL will be reported as the MDL value and flagged with a "U".

4.1.3.2 Practical Quantitation Limits

The practical quantitation limit (PQL) is the lowest level that can be reasonably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. All results will be reported at or above the MDL values, however, for those results falling between the MDL and the PQL, an "F" flag will be applied to the results indicating the variability associated with the result.

4.1.3.3 Instrument Calibration

Analytical instruments will be calibrated in accordance with the analytical methods. All analytes reported will be present in the initial and continuing calibrations, and these calibrations must meet the acceptance criteria specified in Table 4-13. Records of standard preparation and instrument calibration will be maintained by the laboratory. Records unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards will be traceable to standard materials.

Instrument calibration will be checked using all of the analytes listed in the QC acceptance criteria tables in Section 4.1.8 for the method. This applies equally to multiresponse analytes. All calibration criteria satisfy SW-846 requirements at a minimum. The initial calibration will be checked at the frequency specified in the method using materials prepared independently of the calibration standards. Acceptance criteria for the calibration check are presented in Table 4-13. Analyte concentrations are determined with either calibration curves or response factors (RFs). For gas chromatography (GC) and gas chromatography/mass spectroscopy (GC/MS) methods, when using RFs to determine analyte concentrations, the average RF from the initial five point calibration will be used. The continuing calibration will not be used to update the RFs from the initial five point calibration.

4.1.4 Elements Of Quality Control

QC elements relevant to screening data are presented in Section 4.1.7. This section presents QC requirements relevant to analysis of environmental samples that will be followed during all analytical activities for fixed-base, mobile, and field laboratories

producing definitive data. The purpose of this QC program is to produce data of known quality that satisfy project objectives and that meet or exceed requirements of the standard methods of analysis. This program provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC materials.

Laboratory QC samples (such as blanks and laboratory control samples) will be included in the preparation batch with the field samples. An analytical batch is a number of samples (not to exceed 20 environmental samples plus the associated laboratory QC samples) similar in composition (matrix) and that are extracted or digested at the same time and with the same lot of reagents. Matrix spikes and matrix spike duplicates count as environmental samples. The term analytical batch also extends to cover samples that do not need separate extraction or digestion (such as volatile analyses by purge and trap). This analytical batch is a number of samples (not to exceed 20 environmental samples plus the associated laboratory QC samples) that are similar in composition (matrix) analyzed sequentially within a calibration period. The identity of each analytical batch will be unambiguously reported with the analyses so that a reviewer can identify the QC samples and the associated environmental samples. All references to the analytical batch in the following sections and tables in this QAPP refer to the analytical batch.

The type of QC samples and the frequency of use of these samples are discussed below and in the method-specific subsections of Section 4.1.8.

4.1.4.1 Laboratory Control Sample

The laboratory control sample (LCS) is analyte-free water (for aqueous analyses) or Ottawa sand (for soil analyses) spiked with known concentrations of all analytes listed in the QC acceptance criteria table in Section 4.1.8. The LCS will be carried through the complete sample preparation and analysis procedure.

The LCS is used to evaluate each analytical batch and to determine if the method is in control.

One LCS will be included in every analytical batch.

The performance of the LCS is evaluated against the QC acceptance limits given in the tables in Section 4.1.8.

Whenever an analyte in an LCS is outside the acceptance limit, corrective action will be performed. After the system problems have been resolved and system control has been reestablished, all samples in the analytical batch will be reanalyzed for the out-of-control analyte(s). When an analyte in an LCS exceeds the upper or lower control limit and no corrective action is performed or the corrective action was ineffective, the appropriate validation flag, as described in the QC procedure tables in Section 4.1.8, will be applied to all affected results.

4.1.4.2 Matrix Spike/Matrix Spike Duplicate

A matrix spike (MS) and matrix spike duplicate (MSD) is an aliquot of sample spiked with known concentrations of all analytes listed in the QC acceptance criteria tables in Section 4.1.8 for the method. The spiking occurs prior to sample preparation and

analysis. Only samples for this project will be used for spiking. The MS/MSD will be designated on the chain of custody.

The MS/MSD is used to document the bias of a method due to sample matrix and perform QA/QC of the sample and analysis procedures.

A minimum of one MS and one MSD sample will be analyzed for every 20 samples.

The performance of the MS and MSD is evaluated against the QC acceptance limits given in the tables in Section 4.1.8. If either the MS or the MSD is outside the QC acceptance limits, the analytes in all related samples will be qualified according to the data flagging criteria in Section 4.1.8.

4.1.4.3 *Surrogates*

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that are not normally found in environmental samples.

Surrogates are used to evaluate accuracy, method performance, and extraction efficiency.

Surrogates will 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 must be performed. After the system problems have been resolved and system control has been reestablished, the sample will be reprepared and reanalyzed. If corrective actions are not performed or are ineffective, the appropriate validation flag, as described in the QC procedure tables in Section 4.1.8, will be applied to the sample results.

4.1.4.4 *Internal Standards*

Internal standards (ISs) are measured amounts of certain compounds added after preparation or extraction of a sample. They are used in an IS calibration method to correct sample results affected by column injection losses, purging losses, or viscosity effects.

ISs will be added to environmental samples, controls, and blanks, in accordance with the method requirements.

When the IS results are outside of the acceptance limits, corrective actions will be performed. After the system problems have been resolved and system control has been reestablished, all samples analyzed while the system was malfunctioning will be reanalyzed. If corrective actions are not performed or are ineffective, the appropriate validation flag, as described in Section 4.1.8, will be applied to the sample results.

4.1.4.5 *Retention Time Windows*

Retention time windows are used in GC and high performance liquid chromatography (HPLC) analysis for qualitative identification of analytes. They are calculated from

replicate analyses of a standard on multiple days. The procedure and calculation method are given in SW-846 Method 8000A.

When the retention time is outside of the acceptance limits, corrective action will 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. If corrective actions are not performed, the appropriate validation flag, as described in Section 4.1.8, will be applied to the sample results.

4.1.4.6 *Interference Check Sample*

The interference check sample (ICS), used in inductively coupled plasma (ICP) analyses only, contains both interfering and analyte elements of known concentrations

The ICS is used to verify background and interelement correction factors. It is run at the beginning and end of each run sequence. The ICS and all affected samples will be reanalyzed if the ICS results are outside of acceptance limits.

When the interference check sample results are outside of the acceptance limits stated in the method, corrective action will be performed. After the system problems have been resolved and system control has been reestablished, reanalyze the ICS. If the ICS result is acceptable, reanalyze all affected samples. If corrective action is not performed or the corrective action was ineffective, the appropriate validation flag, as described in Section 4.1.8, will be applied to all affected results.

4.1.4.7 *Method Blank*

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 will be carried through the complete sample preparation and analytical procedure.

The method blank is used to document contamination resulting from the analytical process.

A method blank will be included in every analytical batch.

The presence of analytes in a method blank at concentrations greater than the PQL indicates a need for corrective action. Corrective action will be performed to eliminate the source of contamination prior to proceeding with analysis. After the source of contamination has been eliminated, all samples in the analytical batch will be reprepared and reanalyzed. No analytical data will be corrected for the presence of analytes in blanks. When an analyte is detected in the method blank and in the associated samples and corrective actions are not performed or are ineffective, the appropriate validation flag, as described in Section 4.1.8, will be applied to the sample results.

4.1.4.8 *Equipment Blank*

An equipment blank is a sample of ASTM Type II reagent grade water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis.

Equipment blanks are used to assess the effectiveness of equipment decontamination procedures.

The frequency of collection for equipment blanks is specified in Section 4.2 of the SAP. Equipment blanks will be collected immediately after the equipment has been decontaminated. The blank will be analyzed for all laboratory analyses requested for the environmental samples collected at the site.

When an analyte is detected in the equipment blank the appropriate validation flag, as described in Section 4.1.8, will be applied to all sample results from samples collected.

4.1.4.9 Trip Blank

The trip blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled like an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes.

Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures.

When an analyte is detected in the trip blank the appropriate validation flag, as described in Section 4.1.8, will be applied to all sample results from samples in the cooler with the affected trip blank.

One trip blank will accompany each cooler of samples sent to the laboratory for analysis of VOCs.

4.1.4.10 Field Duplicates

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned an identification number in the field such that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel performing the analysis. Specific locations are designated for collection of field duplicate samples prior to the beginning of sample collection.

Duplicate sample results are used to assess precision of the sample collection process. Precision of soil samples to be analyzed for VOCs is assessed from collocated samples because the compositing process required to obtain uniform samples could result in loss of the compounds of interest.

The frequency of collection for field duplicates is specified in Section 4.2 of the SAP.

4.1.5 Quality Control Procedures

4.1.5.1 Holding Time Compliance

All sample preparation and analysis will be completed within the method-required holding times. Holding times are given in Table 4-1. The holding time begins at the time of sample collection. Some methods have more than one holding time requirement (e.g., methods SW8080A, SW8270B, etc.). The preparation holding time is calculated from the time of sample collection to the time of completion of the sample preparation process as described in the applicable method, prior to 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 reanalysis. In methods requiring sample preparation prior to 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 reanalysis.

If holding times are exceeded and the analyses are performed, the results will be flagged according to the procedures as described in Section 4.1.9.

4.1.5.2 Confirmation

Quantitative confirmation of results at or above the PQL for samples analyzed by GC or HPLC will be required and will be completed within the method-required holding times. For GC methods, a second-column is used for confirmation. For HPLC methods, a second column or a different detector is used. The result of the first column/detector will be the result reported. If holding times are exceeded and the analyses are performed, the results will be flagged according to the procedures as described in Section 4.1.9.

4.1.5.3 Standard Materials

Standard materials used in calibration and to prepare samples will be traceable to National Institute Standards and Technology (NIST), EPA, American Association of Laboratory Accreditation (AALA) or other equivalent approved source, if available. If an NIST, EPA or AALA standard material is not available, the standard material proposed for use will be included in an addendum to the SAP and approved before use. The standard materials will be current, and the following expiration policy will be followed: The expiration dates for ampulated solutions will 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 will 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 will be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials will be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window

statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory will label standard and QC materials with expiration dates.

4.1.5.4 *Supplies and Consumables*

The laboratory will inspect supplies and consumables prior to their use in analysis. The materials description in the methods of analysis will be used as a guideline for establishing the acceptance criteria for these materials. Purity of reagents will be monitored by analysis of LCSs. An inventory and storage system for these materials will assure use before manufacturers' expiration dates and storage under safe and chemically compatible conditions.

4.1.5.5 *Sample Dilutions*

Since contamination at the site is expected to be high, it is anticipated that some samples will have to be diluted, causing the risk of raising detection limits for some analytes above ARAR or risk based goals. Specifically, there is a strong possibility of low concentrations of PCBs 'hiding' behind high concentrations of TPH.

4.1.6 Sampling Procedures

4.1.6.1 *Field Sampling*

The field sampling procedures for collecting samples and sampling methods are included in Section 4.2 of the FSP.

Sample Containers

Sample containers will be supplied by the laboratory precleaned and treated according to EPA specifications for the methods. Containers will be stored in clean areas to prevent exposure to fuels, solvents, and other contaminants.

Sample Volumes, Container Types, and Preservation Requirements

Sample volumes, container types, and preservation requirements for the analytical methods performed on for this project are listed in Table 4-1 (CAS 1996).

4.1.6.2 *Sample Handling and Custody*

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analysis and storage, data generation and reporting, and sample disposal. Records concerning the custody and condition of the samples will be maintained in field and laboratory records.

- Chain of custody records will be maintained for all field and field Quality Control(QC) samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in their possession, (2) it is in their view, after being in their possession, (3) it was in their possession and they locked it up or, (4) it is in a designated secure area.

Table 4-1. Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times

Name	Analytical Methods	Container	Preservation	Minimum Sample Volume or Weight	Maximum Holding Time
Metals	SW6010A	Polyethylene bottle (water), glass jar or brass sleeve (soil)	HNO ₃ to pH < 2 (water), 4°C (both)	500 mL (water), 8 ounces (soil)	180 days (water and soil)
Gasoline Range Organics (GRO)	AK101	Vial with Teflon-lined septum (water), glass jar with teflon lined septum (soil)	HCl to pH < 2 and protect from light, 4°C (water), methanol ^a , 25°C ^b (soil)	2x40 mL (water), 4 ounces (soil)	Analyze within 14 days after collection (water), within 28 days ^b (soil)
Diesel Range Organics (DRO)	AK102	Amber glass bottle with teflon-lined cap (water), glass jar with teflon lined lid (soil)	HCl to pH < 2 (water), 4°C (water & soil)	1 liter (water), 4 ounces (soil)	7 days until extraction, 40 days after extraction (water); 14 days until extraction, 40 days after extraction (soil)
Glycols	SW8015 (modified)	Vial with Teflon-lined septum (water), glass jar with teflon lined septum (soil)	4°C (water & soil)	2x40 mL (water), 4 ounces (soil)	7 days until extraction, 40 days after extraction (water); 14 days until extraction, 40 days after extraction (soil)
Polychlorinated biphenyls (PCBs)	SW8081B	Glass bottle with teflon-lined cap (water), glass jar or brass sleeve (soil)	4°C, pH 5-9	1 liter (water), 8 ounces (soil)	7 days until extraction, 40 days after extraction (water); 14 days until extraction, 40 days after extraction (soil)
Semivolatiles organics	SW8270B	Glass bottle with teflon-lined cap (water), glass jar or brass sleeve (soil)	HCl to pH < 2 (water), 4°C (water & soil)	1 liter (water), 8 ounces (soil)	7 days until extraction, 40 days after extraction (water); 14 days until extraction, 40 days after extraction (soil)
Volatile organics	SW8260A	Glass VOA vial with teflon-lined cap (water), glass jar or brass sleeve (soil)	HCl to pH < 2 (water), 4°C (water & soil)	2x40 mL (water), 4 ounces (soil)	14 days (water and soil); 7 days if unpreserved by acid (water)

a. Alaska method 101 requires that sample bottles are preserved with methanol before or at the time of sampling for soil collection. Methanol preservation will not be performed on this project due to shipping restrictions.

b. Soil samples not preserved with methanol at the time of sampling should be stored at 4°C and must be analyzed within 14 days.

The following information concerning the sample will be documented on all of the laboratory chain of custody (COC) forms used for this project:

- Unique sample identification
- Date and time of sample collection
- Source of sample (including name, location, and sample type)
- Designation of MS/MSD
- Preservative used
- Analyses required
- Name of collector(s)
- Pertinent field data (pH, temperature, etc.)
- Serial numbers of custody seals and transportation cases (if used)
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories
- Bill of lading or transporter tracking number (if applicable)

All samples will be uniquely identified, labeled, and documented in the field at the time of collection in accordance with Section 4.2.4 of the FSP.

Samples collected in the field will be transported to the laboratory or field testing site as expeditiously as possible. When a 4°C requirement for preserving the sample is indicated, the samples will be packed in ice or chemical refrigerant to keep them cool during collection and transportation. During transit, it is not always possible to rigorously control the temperature of the samples. As a general rule, storage at low temperature is the best way to preserve most samples. A temperature blank (a volatile organics compounds sampling vial filled with tap water) will be included in every cooler and used to determine the internal temperature of the cooler upon receipt of the cooler at the laboratory. When, in the judgment of the laboratory, the temperature of the samples upon receipt may have affected the stability of the analytes of interest, the problem will be documented in laboratory records and discussed with the Project Manager. The resolution of the problem will also be documented.

Once the samples reach the laboratory, they will be checked against information on the COC form for anomalies. The condition, temperature, and appropriate preservation of samples will be checked and documented on the COC form. Checking an aliquot of the sample using pH paper is an acceptable procedure except for VOCs where an additional sample is required to check preservation. The occurrence of any anomalies in the received samples and their resolution will be documented in laboratory records. All sample information will then be entered into a tracking system, and unique analytical sample identifiers will be assigned. A copy of this information will be reviewed by the laboratory for accuracy. Sample holding time tracking begins with the collection of samples and continues until the analysis is complete. Holding times for methods required routinely for the analyses for this project are specified in Table 4-1. Samples not

preserved or analyzed in accordance with these requirements will be resampled and analyzed. Procedures ensuring internal laboratory COC will also be implemented and documented by the laboratory. Specific instructions concerning the analysis specified for each sample will be communicated to the analysts. Analytical batches will be created, and laboratory QC samples will be introduced into each batch.

While in the laboratory, samples will be stored in limited-access, temperature-controlled areas. Refrigerators, coolers and freezers will be monitored for temperature seven days a week. Acceptance criteria for the temperatures of the refrigerators and coolers is $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Acceptance criteria for the temperatures of the freezers will be less than 0°C . All of the cold storage areas will be monitored by thermometers that have been calibrated with a NIST-traceable thermometer. Samples for volatile organics determination will be stored separately from other samples, standards, and sample extracts. Samples will be stored after analysis until disposed of in accordance with applicable local, state, and federal regulations. Disposal records will be maintained by the laboratory.

Standard operating procedures (SOPs) describing sample control and custody are maintained by the laboratory.

4.1.7 Screening Analytical Methods

The screening methods anticipated to be used for this project include PCBs by immunoassay and halogenated hydrocarbons by immunoassay. The following sections describe the analytical methods.

PCB Immunoassay Field Screening

Soil samples will be screened for total polychlorinated biphenyls (PCBs) using an Ohmicron PCB RaPID Assay® kits. An extraction of the soil sample is performed, and the extract and an enzyme conjugate reagent are added to immobilized anti-chemical antibodies. The enzyme conjugate competes with the chemicals of concern in the sample for binding to antibodies. The test is interpreted by comparing the response produced by the sample to the response produced by a standard. The powerful catalytic ability of the enzyme provides highly sensitive detection. The range of detection in soil is 0.5 mg/kg to 100 mg/kg. Results will be reported with one significant figure due to the precision of procedures used for sample collection and dilution. See Appendix D for a detailed description of the test Ohmicron test kit and the procedures used.

QC procedures to be implemented for immunoassay testing will be as follows: One field duplicate will be collected and analyzed for every ten samples. The RPD acceptance criteria will be <20%. Additionally, a calibration curve is comprised of four standards. Two repetitions of each standard point are run. QC procedures require a coefficient of variation between each repetition of a standard point to be less than 10 percent. These two repetitions of the standard are averaged to produce a value used in the calibration curve. The calibration curve must approach a linear curve with a correlation value of $R=0.990$ or less. Finally, one control sample with a known value of $3\text{ }\mu\text{g/kg}$ will be analyzed with each run. Ohmicron QC standards state that this control sample must be within $3 \pm 0.6\text{ }\mu\text{g/kg}$. Periodically, duplicate samples will be collected and shipped to the

laboratory for analysis. The laboratory results will be compared with the test kit results to verify the accuracy of the test kits.

PCB Drum Screening

Drum samples will be screened, as determined in Section 2.3.4, for PCBs using Dexsil's Clor-N-Oil® test kits. QC procedures to be implemented for immunoassay testing will be as follows: One field duplicate will be collected and analyzed for every ten samples. The RPD acceptance criteria will be <20%.

Halogenated Hydrocarbon Immunoassay Drum Screening

Drum samples will be screened, as determined in Section 2.3.4, for halogenated hydrocarbons using Dexsil's Hydroclor-Q® test kits. These kits quantitative field test kits designed to measure organic chlorine contamination in oil/water mixtures and antifreeze. QC procedures to be implemented for immunoassay testing will be as follows: One field duplicate will be collected and analyzed for every ten samples. The RPD acceptance criteria will be <20%.

4.1.8 Analytical Methods and Procedures

A brief description and three tables for each method for the analyses to be performed during this project are included in the following subsections as required by the AFCEE Quality Assurance Project Plan, February 1996. The first table presents the PQLs for each analyte in the method. The PQLs are presented for both soil and water matrices. The second table presents the acceptance criteria for the accuracy of spiked analyte and surrogate recoveries. This table also presents the acceptance criteria for the precision of matrix, field, and laboratory duplicate recoveries. The third table presents the calibration and QC procedures for each method. Corrective actions and data flagging criteria are also included in this table.

In the third table, the first two columns designate the method number and the class of analytes that may be determined by the method. The third column lists the method-required calibration and QC elements. The fourth column designates the minimum frequency for performing each calibration and QC element. The fifth column designates the acceptance criteria for each calibration and QC element. The sixth column designates the corrective action in the event that a calibration or QC element does not meet the acceptance criteria. The last column designates the data flagging criteria that will be applied in the event that the method-required calibration and QC acceptance criteria are not met.

Before the field effort begins, the laboratory will be required to provide copies of the laboratory specific QA, MDL study results (as discussed in Section 4.1.3.1), and control limit study results. Laboratory PQLs must meet or exceed PQLs listed in the following method tables. Updated MDL and control limit studies must be submitted as they are updated by the laboratory through the duration of the project.

All QA/QC descriptions and requirements (PQLs, surrogates, %R, RPDs) were obtained from the AFCEE Quality Assurance Project Plan Version 1.1 (USAF 1996) and Alaska Methods 101, 102, and 103 (ADEC Storage Tank Program Procedures Manual 1996).

4.1.8.1 Method SW6010A-Trace Elements (Metals)

Samples are analyzed for trace elements or metals using method SW6010A for water and soils. Analysis for most metals requires digestion of the sample. This digestion is performed by method SW3005A for water or method SW3050A for soil. Following digestion, the trace elements are determined simultaneously or sequentially using (Inductively Coupled Plasma Emission Spectroscopy) ICPES. The elements of concern for this project and corresponding PQLs for this method are listed in Table 4-2. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4-3 and 4-4.

Table 4-2. PQLs for Method SW6010A

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
ICP Screen for Metals SW3005A/SW6010A (W) SW3050A/SW6010A (S)	Arsenic	0.6	mg/L	60.0	mg/kg
	Cadmium	0.04	mg/L	4.0	mg/kg
	Chromium	0.07	mg/L	7.0	mg/kg
	Lead	0.5	mg/L	50.0	mg/kg

Table 4-3. QC Acceptance Criteria for Method SW6010A

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
SW6010A	Arsenic	80-120	≤ 15	80-120	≤ 25
	Cadmium	80-120	≤ 15	80-120	≤ 25
	Chromium	80-120	≤ 15	80-120	≤ 25
	Lead	80-120	≤ 15	80-120	≤ 25

Table 4-4. Summary of Calibration and QC Procedures for Method SW6010A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria ^{a, b, c}
SW6010A	ICP Metals	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Highest calibration standard	Before beginning a sample run	All analytes within $\pm 5\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration blank	After every 10 samples and at end of the analysis sequence	No analytes detected > PQL	Correct problem then analyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) in all samples associated with the blank
		Continuing calibration verification (Instrument Check Standard)	After every 10 samples and at the end of the analysis sequence	All analyte(s) within $\pm 10\%$ of expected value	Repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 4-3	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected > PQL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		Interference check solution (ICS)	At the beginning and end of an analytical run or twice during an 8 hour period, whichever is more frequent	Within $\pm 20\%$ of expected value	Terminate analysis; correct problem; reanalyze ICS; reanalyze all affected samples	Apply R to all results for specific analyte(s) in all samples associated with the ICS

Quality Program Plan Addendum
Cape Lisburne LRRS, Alaska

Table 4-4. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^{b,c}
SW6010A	ICP Metals	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 4-3	Correct problem then reprep and analyze the LCS and all samples in the affected analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects Apply J to all sample results if either of following exist: (1) new matrix check not run (2) RPD ≥ 10%
		Dilution test	Each new sample matrix	1:4 dilution must agree within ±10% of the original determination	Perform post digestion spike addition	
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 4-3	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if, (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per year ^d	Detection limits established should be < the PQLs in Table 4-2	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and PQL	none	none	none	Apply F to all results between MDL and PQL

a. All corrective actions associated with this project work will be documented, and all records will be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. When corrective action is successful, the analysis is rerun and original data is not reported.

c. Data qualifiers are defined in Table 4-26.

d. Per the AFCEE QAPP, the laboratory shall provide the MDL demonstrations to AFCEE at the beginning of the project and upon request.

4.1.8.2 Method AK101 - Gasoline Range Organics

This method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as gasoline. Detection is achieved by a FID or PID/FID in series. For soil samples, this analysis requires the introduction of methanol into the sample at the time of sampling. Due to air transportation safety regulations prohibiting the transport of flammable materials, a waiver from ADEC will be requested to use ADEC approved sample containers not containing methanol that insure sample integrity. The PQL for this method is listed in Table 4-5. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4-6 and 4-7. The complete method description is included in Appendix D.

Table 4-5. PQLs for Method AK101

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
AK101	GRO	0.1	mg/L	5	mg/kg

Table 4-6. QC Acceptance Criteria for Method AK101

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
AK101	GRO	60-120	≤ 20	60-120	≤ 20
	<i>Surrogates:</i>				
	1,4-Difluorobenzene	N/A		50-150	
	4-Bromofluorobenzene	60-120		60-120	

Table 4-7. Summary of Calibration and QC Procedures for Method AK101

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action*	Flagging Criteria**
AK101	GRO	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	%RSD < 20% for CFs or RFs	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4-6	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected > PQL	Correct problem repeat and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4-6	Repeat and analyze the LCS and all samples in the affected analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results; if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

Table 4-7. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^{b & c}
AK101	GRO	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4-6	Correct problem then reextract and analyze sample	For the samples, if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects If any surrogate recovery is < 10%, apply R to all results For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if: (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria, Table 4-6	none	
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample
		MDL study	Once per year ^d	Detection limits established should be < the PQLs in Table 4-5	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and PQL	none	none	none	Apply F to all results between MDL and PQL

a. All corrective actions associated with this project work will be documented, and all records will be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. When corrective action is successful, the analysis is rerun and original data is not reported.

c. Data qualifiers are defined in Table 4-26.

d. Per the AFCEE QAPP, the laboratory shall provide the MDL demonstrations to AFCEE at the beginning of the project and upon request.

4.1.8.3 Method AK102 - Diesel Range Organics

This method is designed to measure the concentration of diesel range organics in water and soil. It is based on a solvent extraction, gas chromatography procedure. The PQL for this method is listed in Table 4-8. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4-9 and 4-10. The complete analytical method is included in Appendix D.

Table 4-8. PQLs for Method AK102

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
AK102	DRO	0.10	mg/L	4.0	mg/kg

Table 4-9. QC Acceptance Criteria for Method AK102

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
AK102	DRO	60-120	≤ 20	60-120	≤ 20
	<i>Surrogates:</i> p-Terphenyl	60-120		60-120	

Table 4-10. Summary of Calibration and QC Procedures for Method AK102

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action*	Flagging Criteria**
AK102	DRO	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	%RSD < 20% for CFs or RFs	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4-9	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected > PQL	Correct problem reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4-9	Reprep and analyze the LCS and all samples in the affected analytical batch	For specific analyte(s) in all samples in the associated analytical batch;
						if the LCS %R > UCL, apply J to all positive results
						if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

Quality Program Plan Addendum
Cape Lisburne LRRS, Alaska

Table 4-10. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^{b & c}
AK102	DRO	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4-9	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria, Table 4-9	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if: (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second-column confirmation MDL study	100% for all positive results Once per year ^d	Same as for initial or primary column analysis Detection limits established should be < the PQLs in Table 4-8	Same as for initial or primary column analysis none	Apply R to the result for the specific analyte(s) in the sample
		Results reported between MDL and PQL	none	none	none	Apply R to all results for the specific analyte(s) in all samples analyzed
						Apply F to all results between MDL and PQL

a. All corrective actions associated with this project work will be documented, and all records will be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. When corrective action is successful, the analysis is rerun and original data is not reported.

c. Data qualifiers are defined in Table 4-26.

d. Per the AFCEE QAPP, the laboratory shall provide the MDL demonstrations to AFCEE at the beginning of the project and upon request.

4.1.8.4 Method AK103 - Residual Range Organics

This method is designed to measure the concentration of residual range organics in water and soil. It is based on a solvent extraction, gas chromatography procedure. The PQL for this method is listed in Table 4-11. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4-12 and 4-13. The complete analytical method is included in Appendix D.

Table 4-11. PQLs for Method AK103

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
AK103	RRO	0.4	mg/L	40	mg/kg

Table 4-12. QC Acceptance Criteria for Method AK103

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
AK103	RRO	60-120	≤ 20	60-120	≤ 20
	Surrogates: p-Terphenyl	60-120		60-120	

Table 4-13. Summary of Calibration and QC Procedures for Method AK103

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action*	Flagging Criteria ^{a, b, c}
AK103	RRO	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	%RSD < 20% for CFs or RFs	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analytes of a QC check sample	Once per analyst	QC acceptance criteria, Table 4-12	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected > PQL	Correct problem reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4-12	Reprep and analyze the LCS and all samples in the affected analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

Table 4-13. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^{b, c}
AK103		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4-12	Correct problem then reextract and analyze sample	For the samples, if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria, Table 4-12	none	If any surrogate recovery is < 10%, apply R to all results
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if: (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per year ^d	Detection limits established should be < the PQLs in Table 4-11	none	Apply R to the result for the specific analyte(s) in the sample
		Results reported between MDL and PQL	none	none	none	Apply R to all results for the specific analyte(s) in all samples analyzed Apply F to all results between MDL and PQL

a. All corrective actions associated with this project work will be documented, and all records will be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. When corrective action is successful, the analysis is rerun and original data is not reported.

c. Data qualifiers are defined in Table 4-26.

d. Per the AFCEE QAPP, the laboratory shall provide the MDL demonstrations to AFCEE at the beginning of the project and upon request.

4.1.8.5 Method SW8015M - Glycols

This method, a modified version of SW 8015, is designed to measure the concentration of ethylene glycol and propylene glycol in water and soil. For this project, samples will only be analyzed for ethylene glycol. Samples are analyzed via a GC/FID. The PQL for this method is listed in Table 4-14. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4-15 and 4-16.

Table 4-14. PQLs for Method 8015M

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
8015M	Ethylene Glycol	10	mg/L	10	mg/kg

Table 4-15. QC Acceptance Criteria for Method 8015M

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
8015M	Ethylene Glycol	50-150	30	50-150	40
	<i>Surrogate:</i> Hexafluoroisopropanol	50-150		50-150	

Table 4-16. Summary of Calibration and QC Procedures for Method 8015M

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action*	Flagging Criteria**
8015M	Glycols	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	%RSD < 20% for CFs or RFs	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4-15	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected > PQL	Correct problem reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4-15	Reprep and analyze the LCS and all samples in the affected analytical batch	For specific analyte(s) in all samples in the associated analytical batch;
						if the LCS %R > UCL, apply J to all positive results
						if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

**Quality Program Plan Addendum
Cape Lisburne LRRS, Alaska**

Table 4-16. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^{b & c}
8015M	Glycols	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4-15	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria, Table 4-15	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if: (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample
		MDL study	Once per year ^d	Detection limits established should be < the PQLs in Table 4-14	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and PQL	none	none	none	Apply F to all results between MDL and PQL

a. All corrective actions associated with this project work will be documented, and all records will be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. When corrective action is successful, the analysis is rerun and original data is not reported.

c. Data qualifiers are defined in Table 4-26.

d. Per the AFCEE QAPP, the laboratory shall provide the MDL demonstrations to AFCEE at the beginning of the project and upon request.

4.1.8.6 Method SW8081 - Polychlorinated Biphenyls (PCBs)

PCBs in water and soil samples are analyzed using method SW8081. This analytical method involves extraction of water samples using a separatory funnel (method SW3510B). Extraction of solid samples is accomplished using ultrasonic extraction (method SW3550A) procedures. The PCBs are separated and quantified by GC using electron capture detection. PQLs for this method are presented in Table 4-17. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4-18 and 4-19.

Table 4-17. PQLs for Method SW8081

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
PCBs	AROCLOR-1016	1.0	µg/L	0.70	mg/kg
SW3510B/SW8081 (W)	AROCLOR-1221	1.0	µg/L	0.70	mg/kg
SW3550A/SW8081 (S)	AROCLOR-1232	1.0	µg/L	0.70	mg/kg
	AROCLOR-1242	1.0	µg/L	0.70	mg/kg
	AROCLOR-1248	1.0	µg/L	0.70	mg/kg
	AROCLOR-1254	1.0	µg/L	0.70	mg/kg
	AROCLOR-1260	1.0	µg/L	0.70	mg/kg

Table 4-18. QC Acceptance Criteria for Method SW8081

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
SW8081	AROCLOR-1016	54-125	≤ 30	44-127	≤ 50
	AROCLOR-1221	41-126	≤ 30	31-136	≤ 50
	AROCLOR-1232	41-26	≤ 30	31-136	≤ 50
	AROCLOR-1242	39-150	≤ 30	29-160	≤ 50
	AROCLOR-1248	41-126	≤ 30	31-136	≤ 50
	AROCLOR-1254	29-131	≤ 30	25-141	≤ 50
	AROCLOR-1260	41-126	≤ 30	31-136	≤ 50
	Surrogates:				
	DCBP	34-133		25-143	
	TCMX	45-125		35-135	

Table 4-19. Summary of Calibration and QC Procedures for Method SW8081

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action*	Flagging Criteria ^{b,c}
SW8081	PCBs	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	%RSD < 20% for CFs or RFs	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
		Breakdown check (Endrin and DDT)	Daily prior to analysis of samples	Degradation $\leq 20\%$	Repeat breakdown check	Apply J to all positive DDT, DDE, DDD, endrin, endrin ketone and endrin aldehyde results; apply R to the analytes listed above if minimum frequency is not met
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 4-18	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected > PQL	Correct problem reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch

Quality Program Plan Addendum
Cape Lisburne LRRS, Alaska

Table 4-19. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^{b,c}
SW8081	PCBs	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4-18	Reprep and analyze the LCS and all samples in the affected analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4-18	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 4-18	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample
		MDL study Results reported between MDL and PQL	Once per year ^d none	Detection limits established should be < the PQLs in Table 4-17 none	none none	Apply R to all results for the specific analyte(s) in all samples analyzed Apply F to all results between MDL and PQL

a. All corrective actions associated with this project work will be documented, and all records will be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. When corrective action is successful, the analysis is rerun and original data is not reported.

c. Data qualifiers are defined in Table 4-26.

d. Per the AFCEE QAPP, the laboratory shall provide the MDL demonstrations to AFCEE at the beginning of the project and upon request.

4.1.8.7 *Method SW8260A - Volatile Organics*

Volatile (or purgeable) organics in water and soil samples are analyzed using method SW8260A. This method uses a capillary column GC/MS technique. Volatile compounds are introduced into the GC by purge and trap (SW5030A). An inert gas is bubbled through the water samples (or a soil-water slurry for soil samples) to transfer the purgeable organic compounds from the liquid to vapor phase. Soil samples with higher contaminant levels are extracted before purging. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is backflushed and heated to desorb the purgeable organics onto a capillary GC column where they are separated and then detected with a mass spectrometer. The analytes detected and PQLs for this method are listed in Table 4-20.

Calibration — The mass spectrometer is tuned daily to give an acceptable spectrum for bromofluorobenzene (BFB). The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- 50-15 percent to 40 percent of mass 95
- 75-30 percent to 60 percent of mass 95
- 95-base peak, 100 percent relative abundance
- 96-5 percent to 9 percent of mass 95
- 173-0 percent to less than 2 percent of mass 174
- 174-greater than 50 percent of mass 95
- 175-5 percent to 9 percent of mass 174
- 176-greater than 95 percent, but less than 101 percent of mass 174
- 177-5 percent to 9 percent of mass 176

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4-21 and 4-22.

Table 4-20. PQLs for Method SW8260A

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
VOCs SW5030A/SW8260A (W, S)	1,1,1,2-Tetrachloroethane	0.5	µg/L	0.003	mg/kg
	1,1,1-TCA	0.8	µg/L	0.004	mg/kg
	1,1,2,2-Tetrachloroethane	0.4	µg/L	0.002	mg/kg
	1,1,2-TCA	1.0	µg/L	0.005	mg/kg
	1,1-DCA	0.4	µg/L	0.002	mg/kg
	1,1-DCE	1.2	µg/L	0.006	mg/kg
	1,1-Dichloropropene	1.0	µg/L	0.005	mg/kg
	1,2,3-Trichlorobenzene	0.3	µg/L	0.002	mg/kg
	1,2,3-Trichloropropane	3.2	µg/L	0.02	mg/kg
	1,2,4-Trichlorobenzene	0.4	µg/L	0.002	mg/kg
	1,2,4-Trimethylbenzene	1.3	µg/L	0.007	mg/kg
	1,2-DCA	0.6	µg/L	0.003	mg/kg
	1,2-DCB	0.3	µg/L	0.002	mg/kg
	1,2-Dibromo-3-chloropropane	2.6	µg/L	0.01	mg/kg
	1,2-Dichloropropane	0.4	µg/L	0.002	mg/kg
	1,2-EDB	0.6	µg/L	0.003	mg/kg
	1,3,5-Trimethylbenzene	0.5	µg/L	0.003	mg/kg
	1,3-DCB	1.2	µg/L	0.006	mg/kg
	1,3-Dichloropropane	0.4	µg/L	0.002	mg/kg
	1,4-DCB	0.3	µg/L	0.002	mg/kg
	1-Chlorohexane	0.5	µg/L	0.003	mg/kg
	2,2-Dichloropropane	3.5	µg/L	0.02	mg/kg
	2-Chlorotoluene	0.4	µg/L	0.002	mg/kg
	4-Chlorotoluene	0.6	µg/L	0.003	mg/kg
	Benzene	0.4	µg/L	0.002	mg/kg
	Bromobenzene	0.3	µg/L	0.002	mg/kg
	Bromochloromethane	0.4	µg/L	0.002	mg/kg
	Bromodichloromethane	0.8	µg/L	0.004	mg/kg
	Bromoform	1.2	µg/L	0.006	mg/kg
	Bromomethane	1.1	µg/L	0.005	mg/kg
	Carbon tetrachloride	2.1	µg/L	0.01	mg/kg
	Chlorobenzene	0.4	µg/L	0.002	mg/kg
	Chloroethane	1.0	µg/L	0.005	mg/kg
	Chloroform	0.3	µg/L	0.002	mg/kg
	Chloromethane	1.3	µg/L	0.007	mg/kg
	Cis-1,2-DCE	1.2	µg/L	0.006	mg/kg
	Cis-1,3-Dichloropropene	1.0	µg/L	0.005	mg/kg
	Dibromochloromethane	0.5	µg/L	0.003	mg/kg
	Dibromomethane	2.4	µg/L	0.01	mg/kg
	Dichlorodifluoromethane	1.0	µg/L	0.005	mg/kg
	Ethylbenzene	0.6	µg/L	0.003	mg/kg
	Hexachlorobutadiene	1.1	µg/L	0.005	mg/kg

Table 4-20. Continued

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
VOCs SW5030A/SW8260A (W, S) (continued)	Isopropylbenzene	0.5	µg/L	0.008	mg/kg
	m-Xylene	0.5	µg/L	0.003	mg/kg
	Methylene chloride	0.3	µg/L	0.002	mg/kg
	n-Butylbenzene	1.1	µg/L	0.005	mg/kg
	n-Propylbenzene	0.4	µg/L	0.002	mg/kg
	Naphthalene	0.4	µg/L	0.002	mg/kg
	o-Xylene	1.1	µg/L	0.005	mg/kg
	p-Isopropyltoluene	1.2	µg/L	0.006	mg/kg
	p-Xylene	1.3	µg/L	0.007	mg/kg
	Sec-Butylbenzene	1.3	µg/L	0.007	mg/kg
	Styrene	0.4	µg/L	0.002	mg/kg
	TCE	1.0	µg/L	0.01	mg/kg
	Tert-Butylbenzene	1.4	µg/L	0.007	mg/kg
	Tetrachloroethene	1.4	µg/L	0.007	mg/kg
	Toluene	1.1	µg/L	0.005	mg/kg
	Trans-1,2-DCE	0.6	µg/L	0.003	mg/kg
	Trans-1,3-Dichloropropene	1.0	µg/L	0.005	mg/kg
	Trichlorofluoromethane	0.8	µg/L	0.004	mg/kg
	Vinyl chloride	1.1	µg/L	0.009	mg/kg

Table 4-21. QC Acceptance Criteria for Method SW8260A

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
SW8260A	1,1,1,2-Tetrachloroethane	72-125	≤ 20	62-108	≤ 30
	1,1,1-TCA	75-125	≤ 20	65-135	≤ 30
	1,1,2,2-Tetrachloroethane	74-125	≤ 20	64-135	≤ 30
	1,1,2-TCA	75-127	≤ 20	65-135	≤ 30
	1,1-DCA	72-125	≤ 20	62-135	≤ 30
	1,1-DCE	75-125	≤ 20	65-135	≤ 30
	1,1-Dichloropropene	75-125	≤ 20	65-135	≤ 30
	1,2,3-Trichlorobenzene	75-137	≤ 20	65-147	≤ 30
	1,2,3-Trichloropropane	75-125	≤ 20	65-135	≤ 30
	1,2,4-Trichlorobenzene	75-135	≤ 20	65-145	≤ 30
	1,2,4-Trimethyl Benzene	75-125	≤ 20	65-135	≤ 30
	1,2-DCA	68-127	≤ 20	58-137	≤ 30
	1,2-DCB	75-125	≤ 20	65-135	≤ 30
	1,2-Dibromo-3-chloropropane	59-125	≤ 20	49-135	≤ 30
	1,2-Dichloropropane	70-125	≤ 20	60-135	≤ 30
	1,2-EDB	75-125	≤ 20	65-135	≤ 30
	1,3,5-Trimethylbenzene	72-112	≤ 20	62-135	≤ 30
	1,3-DCB	75-125	≤ 20	65-135	≤ 30
	1,3-Dichloropropane	75-125	≤ 20	65-135	≤ 30
	1,4-DCB	75-125	≤ 20	65-135	≤ 30
	1-Chlorohexane	75-125	≤ 20	65-135	≤ 30
	2,2-Dichloropropane	75-125	≤ 20	65-135	≤ 30
	2-Chlorotoluene	73-125	≤ 20	63-135	≤ 30
	4-Chlorotoluene	74-125	≤ 20	64-135	≤ 30
	Benzene	75-125	≤ 20	65-135	≤ 30
	Bromobenzene	75-125	≤ 20	65-135	≤ 30
	Bromochloromethane	73-125	≤ 20	63-135	≤ 30
	Bromodichloromethane	75-125	≤ 20	65-135	≤ 30
	Bromoform	75-125	≤ 20	65-135	≤ 30
	Bromomethane	72-125	≤ 20	62-135	≤ 30
	Carbon Tetrachloride	62-125	≤ 20	52-135	≤ 30
	Chlorobenzene	75-125	≤ 20	65-135	≤ 30
	Chloroethane	65-125	≤ 20	55-135	≤ 30
	Chloroform	74-125	≤ 20	64-135	≤ 30
	Chloromethane	75-125	≤ 20	65-135	≤ 30
	Cis-1,2-DCE	75-125	≤ 20	65-135	≤ 30
	Cis-1,3-Dichloropropene	74-125	≤ 20	64-135	≤ 30
	Dibromochloromethane	73-125	≤ 20	63-135	≤ 30
	Dibromomethane	69-127	≤ 20	59-137	≤ 30
	Dichlorodifluoromethane	75-125	≤ 20	65-135	≤ 30
	Dichloropropene	75-125	≤ 20	65-135	≤ 30
	Ethylbenzene	75-125	≤ 20	65-135	≤ 30
	Hexachlorobutadiene	75-125	≤ 20	65-135	≤ 30
	Isopropylbenzene	75-125	≤ 20	65-135	≤ 30
	m-Xylene	75-125	≤ 20	65-135	≤ 30
	Methylene chloride	75-125	≤ 20	65-135	≤ 30
	n-Butylbenzene	75-125	≤ 20	65-135	≤ 30

Table 4-21. Continued

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
SW8260A (Continued)	n-Propylbenzene	75-125	≤ 20	65-135	≤ 30
	Naphthalene	75-125	≤ 20	65-135	≤ 30
	o-Xylene	75-125	≤ 20	65-135	≤ 30
	p-Isopropyltoluene	75-125	≤ 20	65-135	≤ 30
	p-Xylene	75-125	≤ 20	65-135	≤ 30
	Sec-Butylbenzene	75-125	≤ 20	65-135	≤ 30
	Styrene	75-125	≤ 20	65-135	≤ 30
	TCE	71-125	≤ 20	61-135	≤ 30
	Tetrachloroethene	71-125	≤ 20	61-135	≤ 30
	Toluene	74-125	≤ 20	64-135	≤ 30
	Trans-1,2-DCE	75-125	≤ 20	65-135	≤ 30
	Trans-1,3-Dichloropropene	66-125	≤ 20	56-135	≤ 30
	Trichlorofluoromethane	67-125	≤ 20	57-135	≤ 30
	Vinyl Chloride	46-134	≤ 20	36-144	≤ 30
	Surrogates:				
	Dibromofluoromethane	75-125		65-135	
	Toluene-D8	75-125		65-135	
	4-Bromofluorobenzene	75-125		65-135	
	1,2-DCA-D4	62-139		52-149	

Table 4-22. Summary of Calibration and QC Procedures for Method SW8260A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action*	Flagging Criteria**
SW8260 A	Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF $\geq 0.30^d$; and %RSD for all calibration analytes $\leq 30\%$	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Calibration verification	Daily, before sample analysis, every 12 hours of analysis time, and at end of analysis sequence	SPCCs average RF $\geq 0.30^d$; and CCCs $< 20\%$ drift; and all calibration analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4-21	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (Section 4.1.8.5)	Retune instrument and verify	Apply R to all results for all samples associated with the tune
		ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to $+100\%$ of last calibration verification (12 hours) for each	Inspect mass spectrometry or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Apply R to all results for specific analytes for all samples associated with the IS
		Method blank	One per analytical batch	No analytes detected > PQL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch

Table 4-22. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^{b,c}
SW8260 A	Volatile Organics	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4-21	Correct problem then reprep and analyze the LCS and all samples in the affected analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4-21	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 4-21	none	If any surrogate recovery is < 10%, apply R to all results For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3) MS/MSD RPD > CL
		MDL study	Once per year ^e	Detection limits established should be < the PQLs in Table 4-20	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and PQL	none	none	none	Apply F to all results between MDL and PQL

a. All corrective actions associated with this project work will be documented, and all records will be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. When corrective action is successful, the analysis is rerun and original data is not reported.

c. Data qualifiers are defined in Table 4-26.

d. Except > 0.10 for bromoform

e. Per the AFCEE QAPP, the laboratory shall provide the MDL, demonstrations to AFCEE at the beginning of the project and upon request.

4.1.8.8 *Method SW8270B-Semivolatile Organics*

Semivolatile organics (also known as base/neutral and acid extractables) in water and soil samples are analyzed using method SW8270B. This technique determines quantitatively the concentration of a number of SVOCs. Aqueous samples are prepared using method SW3510B, solid samples are prepared by method SW3550A. Samples are extracted and both base/neutral and acid extracts are then concentrated through evaporation. Compounds of interest are separated and quantified using a capillary column GC/MS. The PQLs are listed in Table 4-23.

The mass spectrometer is tuned every 12 hours to give an acceptable spectrum for decafluorotriphenylphosphine (DFTPP). The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- 51-30 percent to 60 percent of mass 198
- 68-less than 2 percent of mass 69
- 70-less than 2 percent of mass 69
- 127-40 percent to 60 percent of mass 198
- 197-less than 1 percent of mass 198
- 198-base peak, 100 percent relative abundance
- 199-5 percent to 9 percent of mass 198
- 275-10 percent to 30 percent of mass 198
- 365-greater than 1 percent of mass 198
- 441-present, but less than mass 443
- 442-greater than 40 percent of mass 198
- 443-17 percent to 23 percent of mass 442

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4-24 and 4-25.

Table 4-23. PQLs for Method SW8270B

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
Semivolatile organics Base/Neutral SW3510B/SW8270B (W) SW3550A/SW8270B (S)	1,2,4-Trichlorobenzene	10.0	µg/L	0.7	mg/kg
	1,2-DCB	10.0	µg/L	0.7	mg/kg
	1,3-DCB	10.0	µg/L	0.7	mg/kg
	1,4-DCB	10.0	µg/L	0.7	mg/kg
	2,4-DNT	10.0	µg/L	0.7	mg/kg
	2,6-DNT	10.0	µg/L	0.7	mg/kg
	2-Chloronaphthalene	10.0	µg/L	0.7	mg/kg
	2-Methylnaphthalene	10.0	µg/L	0.7	mg/kg
	2-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3,3'-Dichlorobenzidine	20.0	µg/L	1.3	mg/kg
	4-Bromophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Chloroaniline	20.0	µg/L	1.3	mg/kg
	4-Chlorophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Nitroaniline	50.0	µg/L	3.3	mg/kg
	Acenaphthylene	10.0	µg/L	0.7	mg/kg
	Acenaphthene	10.0	µg/L	0.7	mg/kg
	Anthracene	10.0	µg/L	0.7	mg/kg
	Benz (a) anthracene	10.0	µg/L	0.7	mg/kg
	Benzo (a) pyrene	10.0	µg/L	0.7	mg/kg
	Benzo (b) fluoranthene	10.0	µg/L	0.7	mg/kg
	Benzo (g,h,i) perylene	10.0	µg/L	0.7	mg/kg
	Benzyl alcohol	20.0	µg/L	1.3	mg/kg
	Bis (2-chloroethoxy) methane	10.0	µg/L	0.7	mg/kg
	Bis (2-chlorethyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-chloroisopropyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-ethylhexyl) phthalate	10.0	µg/L	0.7	mg/kg
	Butyl benzylphthalate	10.0	µg/L	0.7	mg/kg
	Chrysene	10.0	µg/L	0.7	mg/kg
	Di-n-butylphthalate	10.0	µg/L	0.7	mg/kg
	Di-n-octylphthalate	10.0	µg/L	0.7	mg/kg
	Dibenz (a,h) anthracene	10.0	µg/L	0.7	mg/kg
	Dibenzofuran	10.0	µg/L	0.7	mg/kg
	Diethyl phthalate	10.0	µg/L	0.7	mg/kg
	Dimethyl phthalate	10.0	µg/L	0.7	mg/kg
	Fluoranthene	10.0	µg/L	0.7	mg/kg
	Fluorene	10.0	µg/L	0.7	mg/kg
	Hexachlorobenzene	10.0	µg/L	0.7	mg/kg
	Hexachlorobutadiene	10.0	µg/L	0.7	mg/kg
	Hexachlorocyclopentadiene	10.0	µg/L	0.7	mg/kg
	Hexachloroethane	10.0	µg/L	0.7	mg/kg
	Indeno (1,2,3-cd) pyrene	10.0	µg/L	0.7	mg/kg
	Isophorone	10.0	µg/L	0.7	mg/kg

Table 4-23. Continued

Parameter/Method	Analyte	Water		Soil	
		PQL	Unit	PQL	Unit
Semivolatile organics	n-Nitrosodiphenylamine	10.0	µg/L	0.7	mg/kg
Base/Neutral	n-Nitrosodi-n-propylamine	10.0	µg/L	0.7	mg/kg
SW3510B/SW8270B (W)	Naphthalene	10.0	µg/L	0.7	mg/kg
SW3550A/SW8270B (S)	Nitrobenzene	10.0	µg/L	0.7	mg/kg
(continued)	Phenanthrene	10.0	µg/L	0.7	mg/kg
	Pyrene	10.0	µg/L	0.7	mg/kg
Semivolatile organics	2,4,5-Trichlorophenol	50.0	µg/L	3.3	mg/kg
Acid Extractables	2,4,6-Trichlorophenol	10.0	µg/L	0.3	mg/kg
SW3510B/SW8270B (W)	2,4-Dichlorophenol	10.0	µg/L	0.3	mg/kg
SW3550A/SW8270B (S)	2,4-Dimethylphenol	10.0	µg/L	0.3	mg/kg
	2,4-Dinitrophenol	50.0	µg/L	3.3	mg/kg
	2-Chlorophenol	10.0	µg/L	0.3	mg/kg
	2-Methylphenol	10.0	µg/L	0.3	mg/kg
	2-Nitrophenol	10.0	µg/L	0.3	mg/kg
	4,6-Dinitro-2-methylphenol	50.0	µg/L	3.3	mg/kg
	4-Chloro-3-methylphenol	20.0	µg/L	1.3	mg/kg
	4-Methylphenol	10.0	µg/L	0.3	mg/kg
	4-Nitrophenol	50.0	µg/L	1.6	mg/kg
	Benzoic acid	50.0	µg/L	1.6	mg/kg
	Pentachlorophenol	50.0	µg/L	3.3	mg/kg
	Phenol	10.0	µg/L	0.3	mg/kg

Table 4-24. QC Acceptance Criteria for Method SW8270B

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
SW8270B	1,2,4-Trichlorobenzene	44-142	20	34-152	30
	1,2-DCB	42-155	20	32-135	30
	1,3-DCB	36-125	20	26-135	30
	1,4-DCB	30-125	20	25-135	30
	2,4-DNT	39-139	20	29-149	30
	2,6-DNT	51-125	20	41-135	30
	2-Chloronaphthalene	60-125	20	50-135	30
	2-Methylnaphthalene	41-125	20	31-135	30
	2-Nitroaniline	50-125	20	40-135	30
	3,3'-Dichlorobenzidine	29-175	20	25-175	30
	3-Nitroaniline	51-125	20	41-135	30
	4-Bromophenyl phenyl ether	53-127	20	43-137	30
	4-Chloroaniline	45-136	20	35-146	30
	4-Chlorophenyl phenyl ether	51-132	20	41-142	30
	4-Nitroaniline	40-143	20	30-153	30
	Acenaphthylene	47-125	20	37-135	30
	Acenaphthene	49-125	20	39-135	30
	Anthracene	45-165	20	35-175	30
	Benz (a) anthracene	51-133	20	41-143	30
	Benzo (a) pyrene	41-125	20	31-135	30
	Benzo (b) fluoranthene	37-125	20	27-135	30
	Benzo (g,h,i) perylene	34-149	20	25-159	30
	Benzyl alcohol	35-125	20	25-135	30
	Bis (2-chloroethoxy) methane	49-125	20	39-135	30
	Bis (2-chloroethyl) ether	44-125	20	34-135	30
	Bis (2-chloroisopropyl) ether	36-166	20	26-175	30
	Bis (2-ethylhexyl) phthalate	33-129	20	25-139	30
	Butyl Benzyl Phthalate	26-125	20	25-135	30
	Chrysene	55-133	20	45-143	30
	Di-n-Butyl Phthalate	34-126	20	25-136	30
	Di-n-Octyl Phthalate	38-127	20	28-137	30
	Dibenz (a,h) Anthracene	50-125	20	40-135	30
	Dibenzofuran	52-125	20	42-135	30
	Diethyl Phthalate	37-125	20	27-135	30
	Dimethyl Phthalate	25-175	20	25-175	30
	Fluoranthene	47-125	20	37-135	30
	Fluorene	48-139	20	38-149	30
	Hexachlorobenzene	46-133	20	36-143	30
	Hexachlorobutadiene	25-125	20	25-135	30
	Hexachlorocyclopentadiene	41-125	20	31-135	30
	Hexachloroethane	25-153	20	25-163	30
	Indeno (1,2,3-c,d) Pyrene	27-160	20	25-170	30
	Isophorone	26-175	20	25-175	30
	n-Nitrosodi-n-propylamine	37-125	20	27-135	30
	n-Nitrosodiphenylamine	27-125	20	25-135	30
	Naphthalene	50-125	20	40-135	30
	Nitrobenzene	46-133	20	36-143	30

Table 4-24. Continued

Method	Analyte	Accuracy Water (% R)	Precision Water (RPD)	Accuracy Soil (% R)	Precision Soil (RPD)
SW8270B (Continued)	Phenanthrene	54-125	20	44-135	30
	Pyrene	47-136	20	37-146	30
	2,4,5-Trichlorophenol	25-175	20	25-175	30
	2,4,6-Trichlorophenol	39-128	20	29-138	30
	2,4-Dichlorophenol	46-125	20	36-135	30
	2,4-Dimethylphenol	45-139	20	35-149	30
	2,4-Dinitrophenol	30-151	20	25-161	30
	2-Chlorophenol	41-125	20	31-135	30
	2-Methylphenol	25-125	20	25-135	30
	2-Nitrophenol	44-125	20	34-135	30
	4,6-Dinitro-2-Methyl	26-134	20	25-144	30
	4-Chloro-3-Methyl Phenol	44-125	20	34-135	30
	4-Methylphenol	33-125	20	25-135	30
	4-Nitrophenol	25-131	20	25-141	30
	Benzoic Acid	25-162	20	25-172	30
	Pentachlorophenol	28-136	20	38-146	30
	Phenol	25-125	20	25-135	30
	<i>Surrogates:</i>				
	2,4,6-Tribromophenol	25-134		25-144	
	2-Fluorobiphenyl	43-125		34-135	
	2-Fluorophenol	25-125		25-135	
	Nitrobenzene-D5	32-125		25-135	
	Phenol-D5	25-125		25-135	
	Terphenyl-D14	42-126		32-136	

Table 4-25. Summary of Calibration and QC Procedures for Method SW8270B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action*	Flagging Criteria ^{a, b, c}
SW8270B	Semi-volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF $\geq$ 0.05; and %RSD for all calibration analytes $\leq$ 30%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Calibration verification	Daily, before sample analysis, every 12 hours of analysis time, and at end of analysis sequence	SPCCs average RF $\geq$ 0.05; and CCCs $<$ 20% drift; and all calibration analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4-24	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (Section 4.1.8.6)	Retune instrument and verify	Apply R to all results for all samples associated with the tune
		ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each	Inspect mass spectrometry or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Apply R to all results for specific analytes for all samples associated with the IS
		Method blank	One per analytical batch	No analytes detected > PQL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch

Table 4-25. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^{a, b, c}
SW8270 B	Semi-volatile Organics	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4-24	Correct problem then reprep and analyze the LCS and all samples in the affected analytical batch	For specific analyte(s) in all samples in the associated analytical batch: if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4-24	Correct problem then reextract and analyze sample	For the samples, if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 4-24	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if: (1) %R for MS or MSD > UCL or (2) %R for MS or MSD < LCL or (3) MS/MSD RPD > CL
		MDL study	Once per year ^d	Detection limits established are < the PQLs in Table 4-23	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and PQL	none	none	none	Apply F to all results between MDL and PQL

a. All corrective actions associated with this project work will be documented, and all records will be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. When corrective action is successful, the analysis is rerun and original data is not reported.

c. Data qualifiers are defined in Table 4-26.

d. Per the AFCEE QAPP, the laboratory shall provide the MDL demonstrations to AFCEE at the beginning of the project and upon request.

4.1.9 Data Review, Validation, and Reporting

All analytical data generated by the laboratory will be extensively reviewed prior to report generation to assure the validity of the reported data. The data validation process consists of data generation, reduction, and three separate reviews. The first review is performed by the person generating the data. This review ensures that the work is done correctly the first time. The second review is an independent technical review of the work to ensure the data is error-free and to provide a mechanism to correct errors that are missed during the first review. The third review serves to ensure that the completed project meets the client's specifications. In each stage, the review process is documented.

The analyst who generates the analytical data has the prime responsibility for the correctness and completion of the data. All data are generated and reduced following protocols specified in laboratory SOPs. Each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample preparation information is correct and complete
- Analysis information is correct and complete
- The appropriate SOPs have been followed
- Analytical results are correct and complete
- QC samples are within established control limits
- Blanks are within appropriate QC limits
- Analytical and/or preparation holding times are met
- Special sample preparation and analytical requirements have been met
- All required documentation is complete.

The data reduction and validation steps are documented, signed, and dated by the analyst. The analyst then passes the data package to an independent reviewer, who performs the second review.

The second review is performed by a supervisor or data reviewer, whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

- Calibration data is appropriate to the method and is completely documented
- QC samples are within established guidelines
- Qualitative identification of sample components is correct
- Quantitative results are correct
- Documentation is complete and correct
- The data package is complete and ready for data archive

Data qualifiers will be added by the laboratory supervisor of the respective analytical section, after the first and second level of laboratory data reviews have been performed. Analytical batch comments will be added to the first page of the definitive data report packages to explain any nonconformance or other issues. When data are qualified, the laboratory supervisor will apply a final qualifier to any data that have been affected by multiple qualifiers. This final qualifier will reflect the most severe qualifier that was applied to the data, i.e., all data will have only one data qualifying flag associate with it. The allowable final data qualifiers for definitive data and the hierarchy of data qualifiers, listed in order of the most severe through the least severe, are R, M, F, J, B, and U. The definitions of the data qualifiers are shown in Table 4-26. Table 4-27 lists the general flagging conventions. Table 4-28 lists the flagging conventions specific to organic methods. Table 4-29 lists the flagging conventions specific to inorganic methods.

The one exception to these data flagging criteria rules applies to the tentatively identified compounds (TICs) that are identified only in the GC/MS methods. These TICs numerical results will always be qualified with one and only one flag for any reason, and that is the "T" flag.

Table 4-26. Data Qualifiers

Qualifier	Description
J	The analyte was positively identified, the quantitation is an estimation.
U	The analyte was analyzed for, but not detected. The associated numerical value is at or below the MDL.
F	The analyte was positively identified but the associated numerical value is below the PQL.
R	The data are unusable due to deficiencies in the ability to analyze the sample and meet QC criteria.
B	The analyte was found in an associated blank, as well as in the sample.
M	A matrix effect was present.
S	To be applied to all field screening data.
T	Tentatively identified compound (using GC/MS)

Table 4-27. General Flagging Conventions

QC Requirement	Criteria	Flag	Flag Applied To
Holding Time	Time exceeded for extraction or analysis	R	All analytes in the sample
LCS	% R > UCL %R < LCL	J for the positive results J for the positive results, R for the nondetects	The specific analyte(s) in all samples in the associated analytical batch
Method Blank	Analyte(s) detected > PQL	B	The specific analyte(s) in all samples in the associated analytical batch
Equipment Blank	Analyte(s) detected > PQL	B	The specific analyte(s) in all samples with the sampling date
Field duplicates	Field duplicates > PQLs AND RPD outside CL	J for the positive results R for the nondetects	The specific analyte(s) in all samples collected on the same sampling date
MS/MSD	MS or MSD % R > UCL OR MS or MSD % R < LCL OR MS/MSD RPD > CL	M for all results	The specific analyte(s) in all samples collected from the same site as the parent sample
Sample Preservation/Collection	Preservation/collection requirements not met	R for all results	All analytes in the sample
Sample Storage	< 2°C or > 6°C	J for the positive results R for the nondetects	All analytes in the sample
Quantitation	Analyte(s) detected ≥ MDL but < PQL	F	All affected results

Table 4-28. Flagging Conventions Specific to Organic Methods

QC Requirement	Criteria	Flag	Flag Applied To
Ambient Blank (VOC samples only)	Analyte(s) detected > PQL	B	The specific analyte(s) in all samples with the same matrix and sampling date
Trip Blank (VOC samples only)	Analyte(s) detected > PQL	B	The specific analyte(s) in all samples shipped in the same cooler.
Initial Five Point Calibration (GC methods)	RSD > CL	R for all results	The specific analyte(s) in the sample associated with the calibration
Initial Five Point Calibration (GC/MS methods)	SPCC criteria not met	R for all results	All analytes in all samples associated with the calibration
Second Source Calibration Verification (GC and GC/MS methods)	RF outside $\pm$ CL	R for all results	The specific analyte(s) in the sample associated with the calibration
Initial Daily Calibration Verification (GC methods)	RF outside $\pm$ CL	R for all results	The specific analyte(s) in the sample associated with the calibration
Initial Daily Calibration Verification (GC/MS methods)	SPCC and CCC criteria not met	R for all results	The specific analyte(s) in the sample associated with the calibration
	RF outside $\pm$ CL	R for specific results	All analytes in all samples associated with the calibration
Continuing Calibration (GC methods)	RF outside $\pm$ CL	R for all results	The specific analyte(s) in the sample associated with the calibration
Retention time	Retention time of analyte outside of established retention time window	R	The specific analyte(s) in the samples
Surrogates	any surrogate % R > UCL OR any surrogate % R < LCL OR any surrogate recovery < 10%	J for the positive results J for the positive results R for the nondetects R for all results	All analytes in the sample
Mass Spectrometer Tune	Ion abundance criteria not met	R for all results	All analytes in all samples associated with the tune
Tentatively Identified Compounds (TICs)		T	All TICs

Table 4-29. Flagging Conventions Specific to Inorganic Methods

QC Requirement	Criteria	Flag	Flag Applied To
Initial multipoint calibration	Correlation coefficient ≤ 0.995	R	All results for specific analyte(s) for all samples associated with the calibration
Highest calibration standard	result not within 95–105% of expected value	R	All results for specific analyte(s) for all samples associated with the calibration
Calibration blank	not within ± 3 standard deviations of mean blank value	B	All results for specific analyte(s) in all samples associated with the blank
Continuing calibration verification (Instrument Check Standard)	result not within 90–110% of expected value	R	All results for the specific analyte(s) in all samples since the last acceptable calibration
Interference check solution (ICS)	result not within 80–120% of expected value	R	All results for specific analyte(s) in all samples associated with the ICS
Dilution test	dilution not within 90–110% of the original determination	J	Apply to all sample results if the new matrix check was not run or $RPD \geq 10\%$
New matrix check	result > 10% of original result	J	All samples in digestion batch if analytical spike not performed
Recovery test	% R not within 85–115% of expected value	J	All samples in digestion batch if method of standard addition is not performed
Post digestion spike addition	% R of expected value not within 75–125%	J	All sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition
Second source calibration verification	RPD outside $\pm CL$	R	All samples
Method of standard addition	Method of standard addition not done OR Method of standard addition spike levels inappropriate OR correlation coefficient < 0.995	J to all positive results	Sample

The second data review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data is then approved for release and a final report is prepared.

Before the report is released to the client, the project is reviewed for completeness and to ensure that the data meet the overall objectives of the project. This review is labeled the third review. Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality is generated consistently.

Upon receipt of final laboratory reports, the data will be reviewed inhouse or by a third party to ensure all contract analytical requirements are met. Specifically,

- All preparation and analytical holding times were met
- All method blank samples were free of contamination
- All surrogate recoveries are within specified limits
- All MS/MSD analyses are within the specified ranges
- All QC sample RPDs are within specified limits

Summaries of this information will be presented in the final project report.

4.1.10 Corrective Actions

Items and processes that do not meet established project requirements will be identified, controlled, and corrected as specified according to written procedures. Correction will be focused on determining the cause of the deficiency and instituting actions which, not only correct the deficiency, but also prevent the recurrence of a similar condition.

4.1.10.1 Response to Corrective Actions

Non-conforming equipment, items, activities, conditions, and unusual incidents that could affect compliance with project requirements will be identified, controlled, and reported in a timely manner. A non-conformance is defined as a malfunction, failure, deficiency, or deviation which renders the quality of an item unacceptable or indeterminate. The originator of a Non-Conformance Report (NCR) will describe the finding on the form provided for this purpose and notify the project manager and site CQC officer. Each non-conformance will be reviewed and a disposition given for the item, activity, or condition. The disposition of a non-conformance will be documented and approved by the organization responsible for the issuance of the non-conformance. Project manager and site CQC officer concurrence will be evidenced by signing the NCR.

4.1.10.2 Re-establishment of Control

The modification, repair, re-work, or replacement of non-conforming equipment, items, or activities will require the re-verification of acceptability. In certain instances, as

determined by the program manager, project manager or QA manager, these steps may require that corrective action be completed and verified before site work continues.

The equipment, item, or activity that has a deficiency may be temporarily stopped while the non-conformance is being investigated. If, in the opinion of the project manager and the QA Manager, the non-conformance does not significantly affect the technical quality or use of the work, the work may continue, pending resolution of the non-conformance. The basis for such decisions will be documented on the NCR and submitted to the QA manager for review and approval. The documentation will include a statement that the decision was made before continuing with the work. The records of non-conformance and their dispositions will be kept in the project central files. In addition, the project manager will notify the Air Force of any significant non-conformance that could impact the schedule or work results. The documentation will indicate the corrective action taken or planned.

4.1.11 Quality Assurance Reports

Fundamental to the success of this QA/QC Program is the active participation of management in the project. Management will be apprised of project activities and will actively participate in the development, review, and operation of the project. Management will be informed of QA activities through the receipt, review, and/or approval of daily QA reports. The site QC officer will generate these reports and the project manager will sign them. These reports will become part of the project central files.

Weekly reports will be prepared summarizing the daily reports of each week. These reports will include copies of the daily reports. Daily and weekly reports will be generated by onsite personnel and transmitted via FAX or electronic mail to the Air Force.

4.2 Field Sampling Plan

This section provides site specific procedures that supplement the sampling procedures described in the Program SAP. Table 4-30 provides a summary of samples to be collected and their respective analytes.

4.2.1 Final Characterization Sampling

After stockpiling contaminated soil is complete, and before placing of covers, the stockpiles will be sampled in accordance with parameters shown in Table 4-30.

The samples will be collected following the procedures described below:

1. For the specified analyses, the following number of samples will be collected:
 - VOCs: 1 per 100 cy (minimum of 3)
 - SVOCs and PCBs: 1 per 100 cy (composite, minimum of 3)
 - GRO, DRO, and: 1 per 500 cy (composite, minimum of 3)

- Geotechnical Parameters: 1 per 200 cy (minimum of 3, maximum of 10, collected after soil is placed in the cell)
- 2. The samples will be collected using disposable instruments. A new set of instruments will be used for each collection.
- 3. Soil samples will be placed in 8-ounce glass jars with a Teflon-lined cap provided by the laboratory.
- 4. Wipe samples will be collected by wiping a laboratory-prepared, hexane-soaked gauze wipe on a measured area of debris. After analysis by 8080, results will be given in units of weight per wiped area (e.g. mg/sq. inch).
- 5. Each sample will be given a unique sample number written on the label in indelible ink.
- 6. Each sample will be logged by the sampling team indicating the date, time, and location of the point of collection.
- 7. The samples will be placed in a cooler with "Blue Ice" packs. At the end of the collection, the samples will be transported to the composite building and will be placed in a refrigerator pending final packing for shipment to the laboratory.
- 8. When all of the samples have been collected from the stockpiles, the samples will be placed in zip lock bags, wrapped in bubble wrap and placed in coolers with frozen chemical ice packs to maintain a temperature of 4 degrees C.
- 9. The Chain of Custody for each cooler will be placed in a zip lock bag and fastened to the inside top of the cooler with duct tape. The cooler will then be closed and sealed with duct tape.
- 10. The coolers will be flown via Cape Smythe Air to Kotzebue and then via Alaska Airlines to Anchorage. In Anchorage, the coolers will be picked up by a laboratory representative and hand carried to the designated laboratory.

4.2.2 Soil Sampling for Confirmation of Cleanup Levels in Excavations

All of the excavation sites (LF01, SS03, SS08 and SS09) will be sampled prior to backfill placement to confirm field screening results and to ensure cleanup levels have been met.

A minimum of three samples will be collected from each excavation at SS03, SS08, and SS09 locations. Sample locations will be distributed across the entire excavation in order to obtain representative samples. One sample per 100 square feet will be collected at site LF01. All samples will be collected as described in Section 4.2.1.

4.2.3 Sampling of Bulk Drum Contents

To establish shipping and disposal requirements; a discrete sample will be drawn from each drum that contains product transferred from excavated drums. Each sample will be analyzed as shown in Table 4-30.

All of the material to be sampled is expected to be liquid. The following procedure will be followed for the collection of these samples:

Table 4-30. Sample Summary

MATRIX	LOCATION	NUMBER OF SAMPLES		ANALYSIS													
		Primary	Field Duplicates	Turnaround Time	VOCs 8260	SVOCs 8270	GRO AK101	DRO AK102	RRO AK103	PCBs 8080	Burn RCRA	Glycols 8015	Moisture Content	Total Organic Carbon	Grain Size	Bulk Density	
SOILS	LF01																
	Field Screening	8	1	2 Days				X	X								
	Drum Excavation	20	2	2 Weeks				X	X								
	Confirmation	20	2	2 Days			X	X	X	X					X		
	SSO3																
	Field Screening	2		2 Days				X	X								
	Confirmation	4		2 Days			X	X	X	X					X		
	SSO8																
	Field Screening	2		2 Days				X	X	X							
	Confirmation	1		2 Days			X	X	X	X					X		
	SSO9																
	Field Screening	2		2 Days				X	X	X							
	Closure	2		2 Days			X	X	X	X					X		
STOCKPILES (characterization)	1 per 100cy	Min. 3		2 Weeks	X	X				X							
	1 per 200 cy																
	1 per 500cy	Min. 3		2 Weeks			X	X	X						X	X	X
DRUM CONTENTS	Used/Waste Oil	95		2 Weeks							X						
	Failed Burn Specs: F	30		2 Weeks	X												
	List Antifreeze Mixes	20		2 Weeks											X		
RINSATE	Equipment Rinsate	4		2 Weeks	X	X	X	X	X								

1. Each sample will be collected using a glass drum thief. The bung will be removed from the drum and placed on an absorbent pad placed on top of the drum. Also located on the absorbent pad will be the sample containers with the lids removed and ready to accept the sample material. The drum thief will be inserted all the way to the bottom of the drum. After a slight hesitation to allow the product to level in the drum thief, the thumb of the sampler will be placed over the exposed end of the drum thief and the drum thief will be quickly extracted from the drum. The other hand of the sampler will hold an absorbent pad tightly placed around the outside surface of the drum thief. As the thief is extracted from the drum, the excess material clinging to the outside of the thief will be wiped clean with this absorbent pad thus reducing the potential for spilling some of the contents. When the thief is extracted, the bottom end of the drum thief.

will be placed in the sample container and the thumb plugging the top of the drum thief will be slowly removed to allow controlled placement of the sample in the sample container and prevent overfilling or material splashing. This procedure will be repeated until the sample container is full.

2. The drum thief will be re-inserted in the drum with the lower hand still holding the absorbent pad around the drum thief at the drum bung. When the thief is approximately two thirds of the way in the drum, the drum thief will be sharply forced directly away from the sampler to snap the drum thief into two pieces. The piece of the drum thief remaining in the sampler's hand will be placed in the drum. The bung will be replaced and tightened. The top of the drum will be wiped clean with the absorbent pads and the pads will be placed in 10 mil plastic bags. The absorbent pads may be placed in bags of absorbent pads used previously for cleaning drums of compatible material.
3. The samples will be placed in containers provided by the laboratory for this exclusive purpose.
4. Each sample will be given a unique sample number written on the label in indelible ink.
5. Each sample will be logged by the sampling team indicating the date, time, and location of the point of collection. Sample nomenclature will be in accordance with section 3 of this document.
6. The samples will be placed in a cooler with frozen chemical ice packs. At the end of the collection, the samples will be transported to the composite building and placed in a refrigerator pending final packing for shipment to the laboratory.
7. When all of the samples have been collected from the stockpiles, the samples will be placed in zip lock bags, wrapped in bubble wrap and placed in coolers with frozen chemical ice packs to maintain a temperature of 4 degrees C.
8. The Chain of Custody (COC) for each cooler will be placed in a zip lock bag and fastened to the inside top of the cooler with duct tape. The cooler will then be closed and sealed with duct tape.

9. The coolers will be flown via Cape Smythe Air to Kotzebue and then via Alaska Airlines to Anchorage. In Anchorage the coolers will be picked up by a laboratory courier and hand carried to the laboratory.

4.2.4 Sample Labels

Sample containers (including those for quality assurance and quality control samples) will be labeled at the time of sampling and before the sample is placed in the container. Sample numbers will be recorded in the field logbook at the time of sampling.

A 14 character sample identification system will be used. The first two digits represent the year. The following three letters represent the specific site designation for the job. The next three digits represent the individual sample number. The final two letters represent the matrix type for the sample. An example of a sample identification number is provided below, along with explanations of letter designations.

<u>YEAR</u>	<u>JOB</u>	<u>LOCATION</u>	<u>SAMPLE NUMBER</u>	<u>MATRIX</u>
96	CPL	LF01	001	ES

where:

LF01	=	Landfill and Waste Accumulation Area	SS03	=	White Alice Site
SS08	=	Upper Camp Transformer Building	SS09	=	Lower Camp Transformer
ES	=	Excavation Soil	DC	=	Drum Contents
SS	=	Stockpiled Soil	FW	=	Filtered Water

4.2.5 Sample Packing

Samples will be shipped or hand delivered to the appropriate analytical laboratory with sufficient time allowed for the laboratory to extract the sample within the holding time requirements of the test methods. Samples will be packaged and shipped according to the following procedures:

- One trip blank for each matrix will accompany each cooler containing samples to be analyzed for VOCs and GRO. VOC samples will be packaged in a separate cooler
- One temperature blank will accompany each cooler
- Sample containers will be preserved and transported on ice in a sealed, waterproof, metal (or equivalent strength plastic) cooler
- Inert absorbent material will be placed in the bottom of the cooler to a depth of two to four inches
- Sample containers will be labeled with the sample number, test method requested, and name of the receiving laboratory. Field personnel will verify that all sample

container caps are tight. Additional inert absorbent packing material will be placed around and on top of the containers to prevent breakage. Glass containers will be wrapped in shock-absorbent packaging material and sealed in a plastic bag. The containers will be placed upright in the cooler

- Sealed bags of "Blue Ice" will be placed around, among, and on top of the sample containers. Sample temperature will be maintained at approximately 4 degrees C
- The cooler drain will be taped shut
- The cooler lid will be secured with tape. The cooler will be wrapped with strapping tape at a minimum of two locations, without obscuring any labels
- All sample shipments will be accompanied by a chain of custody form. The completed chain-of-custody form will be enclosed and sealed in a plastic bag and taped to the inside lid of the cooler
- The completed shipping label will be affixed to the top of the cooler
- Labels indicating which side should be up will be displayed on four sides and "Fragile" labels will be displayed on at least two sides
- Numbered, signed, and dated custody seals will be adhered to the front right and back left of the cooler. Custody seals should not be covered with tape
- Coolers will not exceed the weight limits set by the shipper

Note that individual custody tapes will not be placed over each sample container.

4.2.6 Field Logbook

The key aspect of documenting sample custody is thorough record keeping. A daily field logbook will be maintained to document all field activities, including the collection of every sample. The daily field logbook will be bound with consecutively numbered pages and all field notes will be entered in indelible ink. If any changes are made to the field record, the original notation will be crossed out with a single line and initialed and dated by the person making the correction. At a minimum, daily field logbooks will contain the following information:

- Date and time of work commencement
- Name and location of site
- Description of work area
- Purpose of proposed work effort
- Date and times of sample collection or event
- Name of the leader of the field team, names of all field personnel, and the names, addresses and telephone numbers of all pertinent project contacts
- Field observations (weather conditions, field instrument readings)
- Summary of equipment preparation procedures

- Number and type of samples taken and sample identification numbers
- Sample locations
- Description of sample container, preservatives, special handling procedures, analytical tests, etc.
- Explanations of any deviations from the work plan, with rationale for the deviation
- Problems encountered and their resolution
- Date, time and specifics of any instruction received from the Air Force
- Date, time and specifics of any unusual or unanticipated events or conditions

The site CQC officer will review the logbooks of field personnel within 48 hours of field activities. Logbooks will be reviewed for accuracy, completeness, and legibility.

4.2.7 Chain of Custody

At the end of each day on which samples are obtained, and prior to the transfer of the samples to the laboratory, chain of custody documentation will be completed for each sample. The chain of custody form will be used to document sample custody. Information on the container labels will be verified to ensure that the information provided on the chain of custody form is consistent with information on the container label, the Sampling Information Form, and in the field logbook.

4.2.8 Soil Sampling Analysis

Field screening samples will be analyzed in the laboratory for extended range DRO (AK 102). Excavation confirmation samples will be analyzed for GRO (AK 101), DRO (AK 102), and PCBs (EPA 8080). Soil stockpile characterization samples will be analyzed for GRO, DRO, VOCs (EPA 8240), SVOCs (EPA 8270), and PCBs (EPA 8080).

4.2.9 Liquid Waste Analysis

The liquid waste samples will be analyzed to determine the appropriate disposal method for the filled drums of consolidated waste. The samples will initially be tested for RCRA Burn Specifications comprised of the following analyses:

- Flash Point (EPA 1010)
- pH/Corrosivity (EPA 9040)
- Metals (arsenic, chromium, cadmium, and lead, EPA 6000/7000 series)
- PCBs (EPA 8080)

Those drums in which glycols are determined to be present (from drum bulking activities) will be tested for glycols by EPA 8015 and TCLP lead. Those samples failing the RCRA Burn Specifications because of F List Solvents will be tested for VOCs by EPA 8240.

4.2.10 Quality Control

A minimum of four equipment rinsate blanks will be collected, one for each anticipated day of sampling. Each rinsate blank will be analyzed for DRO, GRO, PCBs, VOCs, and SVOCs by the methods listed above.

Five field duplicate samples will be collected from the drum excavation area and analyzed for the same constituents as their respective duplicates.

Chemical data validation will include review of chain of custody forms, holding times, sample containers, preservation, method calibration limits, method blanks, laboratory verification of quantitation limits, preparatory batch control records, corrective actions, formulas used for analyte quantitation, and completeness. Data validation will be performed by an environmental professional experienced in environmental chemistry methods and procedures.

Field records will be reviewed for completeness, identification of valid samples, correlation of field test data, identification of anomalous data, and assessment of accuracy and precision of field test data and measurements. All data will be evaluated for completeness by personnel familiar with this project.

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APPENDIX A – References

REFERENCES

- Alaska Department of Environmental Conservation (ADEC). 1991. *Interim Guidance for Non-UST Contaminated Soil Cleanup Levels*. Guidance No. 001 - Revision No. 1, 17 July 1991. Table 2 (North Slope Numeric Cleanup Levels).
- ADEC. 1995. *Underground Storage Tanks Procedures Manual*. Appendix D (Methods AK101, AK102, and AK103).
- Department of Health Services (Kenneth Kizer, M.D.), Department of Industrial Relations (Ron Rianldi), Division of Occupational Safety and Health (David M. Vaoff). 1986. *A System of Field Identification and Classification of Commonly Spilled Materials*. Interagency Agreement 83-81664, Emergency Responders Risk Assessment Unit, Cal OSHA.
- National Institute for Occupational Safety and Health (NIOSH). 1994. *NIOSH Pocket Guide To Chemical Hazards*. U.S. Department of Health and Human Services, Washington, D.C.
- U.S. Air Force. 1993. *Handbook for the Installation Restoration Program (IRP) Remedial Investigations and Feasibility Studies (RI/FS)*. AFCEE, Brooks Air Force Base, Texas (September 1993).
- USAF. 1994. *Work Plan and Sampling and Analysis Plan for Interim Remedial Actions, Cape Lisburne Radar Station, Alaska*. Prepared for U.S. Air Force Center of Environmental Excellence, Environmental Restoration Program Office, Brooks Air Force Base, Texas. Prepared by ICF Technology Incorporated.
- USAF. 1995a. *Final Interim Remedial Action Report, Cape Lisburne Radar Installation, Alaska*. Prepared for U.S. Air Force Center of Environmental Excellence, Environmental Restoration Program Office, Brooks Air Force Base, Texas. Prepared by ICF Technology Incorporated.
- USAF. 1995b. *Environmental Sampling and Analysis Plan*. Prepared for AFCEE, Environmental Restoration Program Office, Brooks Air Force Base, Texas. Prepared by ACCI, Contract No. F4162494-8070-0009, Delivery Order No. 0001.
- USAF. 1995c. *Standard Operating Procedures for Hazardous and Non-Hazardous Waste (Waste Plan)*. Prepared for AFCEE, Environmental Restoration Program Office, Brooks Air Force Base, Texas. Prepared by ACCI, Contract No. F4162494-8070-0009, Delivery Order No. 0001.
- USAF. 1995d. *Environmental Sampling and Analysis Plan*. Prepared for AFCEE, Environmental Restoration Program Office, Brooks Air Force Base, Texas. Prepared by ACCI, Contract No. F4162494-8070-0009, Delivery Order No. 0001.
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- USAF. 1996a. *Final Remedial Investigation and Feasibility Study, Cape Lisburne Radar Installation, Alaska*. Prepared for AFCEE, Environmental Restoration Program Office, Brooks Air Force Base, Texas. Prepared by ICF Technology Incorporated.

Quality Program Plan Addendum
Cape Lisburne LRRS, Alaska

USAF. 1996b. *Final Risk Assessment, Cape Lisburne Radar Installation, Alaska*. Prepared for AFCEE, Environmental Restoration Program Office, Brooks Air Force Base, Texas. Prepared by ICF Technology Incorporated.

USAF. 1996c. *Quality Assurance Project Plan*. Version 1.1. Prepared by AFCEE.

Woodward-Clyde Consultants. 1992. *Draft Site Investigation Report*. Cape Lisburne Long Range Radar Stations, Alaska.

APPENDIX B - MEMO FROM ADEC TO THE AIR FORCE

9608
File 301



DEPARTMENT OF THE AIR FORCE
PACIFIC AIR FORCES

9 October 1995

MEMORANDUM FOR: ALASKA DEPARTMENT OF ENVIRONMENTAL CONSERVATION
ATTN: MR. KALU A. KALU

FROM: 611th Civil Engineer Squadron
21885 2nd St
Elmendorf AFB AK 99506-4420

SUBJECT: Request for Concurrence on the One-Time Discharge of 100,000 Gallons of
Conditioned Water at Kotzebue Long Range Radar Site (LRRS), Alaska

1. This memorandum is in follow-up to your discussion with Mr. Chris Williams, Jacobs Engineering Group, Inc. on 6 Oct 95 and 9 Oct 95, and the technical proposal presented to you on 6 Oct 95 (attached).

2. Based on this information, we are requesting the States' concurrence on the one-time discharge of approximately 100,000 gallons of water conditioned by granular activated carbon. The water is a result of the soil washing project performed this summer at Kotzebue LRRS.

3. Per your request, conditioned water will meet the 10 part per million (ppm) total BETX requirement and will be discharged onto the gravel pads. Also, benzene concentrations will not exceed 5 parts per billion (ppb). Total BETX and benzene will be analyzed by method SW8020 as opposed to method 602. You indicated to Chris Williams that this would be acceptable. Furthermore, there is no visible sheen or visible hydrocarbons present on the water.

4. Attached please find results for the first two effluent BETX samples. Both samples represent the first 30,000 gallons of water to be discharged. The results are well below the 10ppm BETX and 5ppb benzene limit. We will collect one more sample at the 70,000 gallon stage. These sample results will be forwarded to you for your records.

5. Discharge of water will commence on 9 Oct 95 and will be completed by 13 Oct 95.

6. If you have questions, please feel free to call me at (907) 552-4532.

Michael I. Rhoads
MICHAEL I. RHOADS, GS-12
IRP Project Manager

Table 1
Conditioned Water Discharge Results
Kotzebue LRR8, Alaska
Soil Washing Project

Sample No.	K-EM-002	K-EM-003
Date Collected	10/4/95	10/6/95
Analytical Method	8020	8020
Representative Volume to Discharge (gallons)	0 - 5,000	5,000 - 30,000
Benzene (ppb)	<10	<0.5
Toluene (ppb)	9	<1.0
Ethylbenzene (ppb)	<10	27
Xylenes (ppb)	120	68
Total BTEX (ppm)	0.129	0.095
Discharge Requirement Total BTEX (ppm)*	10	10
Discharge Requirement Benzene (ppb)*	5	5

Post-Test Fee Note	7671
To	Lewis Inc.
From	Bret + Julie
Co.	
Phone	278-7915
Fax	

* Per discussion with Kalu A. Kalu on 10/9/95

APPENDIX C - OHMICRON IMMUNOASSAY DESCRIPTION AND PROCEDURES



Product Profile

General Description

Polychlorinated biphenyls (PCBs) were marketed under the trade name Aroclor® from 1930 to 1977 for use in transformers, capacitors, printing inks, paints, pesticides and many other applications. The Toxic Substances Control Act (TOSCA, 1976), declared PCBs to be toxic and carcinogenic and banned manufacture.

PCBs are classified by the US EPA as Group B2 contaminants, a probable human carcinogen based on sufficient evidence in animals and inadequate data in human studies (US EPA, 1991). PCBs do not readily degrade in the environment and tend to bioaccumulate. The EPA has set a Maximum Contaminant Level for PCB in drinking water of 0.5 ppb and various levels in soil.

Current EPA-approved methods for the detection of PCBs are costly and require lengthy sample preparation, large volume extraction and solvent exchange. The PCB RaPID Assay® eliminates the need for clean-up steps and GC/ECD instrumentation.

The PCB RaPID Assay applies the principles of enzyme linked immunosorbent assay (ELISA) to the determination of PCBs. ELISAs use selective antibodies which are attached to solid supports, in combination with sensitive enzyme reactions. The immunochemical reaction provides high selectivity due to the extraordinary discriminatory capabilities of antibodies. The powerful catalytic ability of the enzyme provides highly sensitive detection. These features produce an analytical system capable of detecting very low levels of chemicals.

PCB RaPID Assay®



Features

- Rapid** – 60 results in less than 45 minutes.
- Precise** – within and between assay %CV <5% at 4.4 ppb.
- Accurate** – highly selective immunochemical method.
- Efficient** – rapid results can cut costs by allowing better personnel and equipment utilization.
- Sensitive** – least detectable dose in water of 0.2 ppb as Aroclor 1254 (90% B/Bo).
- Test Range** – water: 0.5 to 10.0 ppb as Aroclor 1254.
soil: 0.5 to 10.00 ppm.

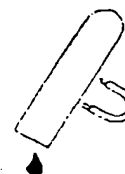
PCB RaPID Assay® — Assay Protocol



1. Add 200 μ L of prepared sample, 250 μ L enzyme conjugate, and 500 μ L antibody coupled magnetic particles.



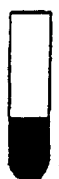
2. Incubate for 15 minutes.



3. Using the RaPID magnetic separator, decant, wash and vortex (2x).



4. Add 500 μ L color reagent.



5. Incubate 20 minutes. Blue color develops.



6. Stop the reaction and read color at 450 nm. Solution turns yellow.

Performance

Specificity

The proprietary antibody used in this assay was developed to detect PCBs. The cross-reactivity with different Aroclors can be expressed as the least detectable dose (LDD) which is estimated at 90% B/B0.

Compound	LDD Water (ppb)	LDD Soil (ppm)	50% B/B0 Water (ppb)	50% B/B0 Soil (ppm)
Aroclor 1254	0.20	0.50	3.60	3.60
Aroclor 1260	0.20	0.30	2.30	2.30
Aroclor 1248	0.22	0.42	4.22	4.22
Aroclor 1242	0.34	0.80	8.80	8.80
Aroclor 1262	0.36	0.64	4.74	4.74
Aroclor 1232	0.84	2.24	18.76	18.76
Aroclor 1268	0.92	2.30	21.80	21.80
Aroclor 1016	0.94	2.70	25.60	25.60
Aroclor 1221	13.54	28.60	162.60	162.60

Precision

Water samples were fortified with Aroclor 1254 at different levels and assayed in singlicate five times over five days. The following results were obtained (n=25):

Sample #	1	2	3	4
Mean	0.86	3.10	4.36	8.04
% CV *	8.5	6.2	4.6	4.9
% CV *	15.7	6.1	0.6	2.5

* within assay

* between assay

Recovery

Four water samples, taken from a small creek, local pond, well and municipal water source were fortified with various levels of Aroclor 1254 and assayed with this system. The following results were obtained (n=12):

Spike (ppb)	Recovery Mean $\pm$ S.D.	%
1.0	0.86 $\pm$ 0.08	86
3.0	3.18 $\pm$ 0.28	106
4.0	4.32 $\pm$ 0.36	108
8.0	7.68 $\pm$ 0.68	96
Average		99

Solvent Tolerances

The following solvents do not interfere with this assay when their concentration in the sample is at, or lower, than those listed.

Solvent	Maximum Conc. Tolerated
Acetone	5%
Acetonitrile	2%
DMF	5%
Methanol	10%
Methylene Chloride	0.2%

Results

When using the RPA-I RaPID Analyzer™, results are reported in ppb PCB. If read in a standard spectrophotometer, results from the calibrators are plotted on graph paper and used to determine final results. It is recommended that a control be included in each run. A positive control (3.0 ppb) is supplied with the PCB RaPID® Assay kit.

As with any analytical technique (GC, HPLC, etc.), positive results requiring some action should be confirmed by an alternate technology.

Ordering Information

PCB Products

RaPID Assay kit, 30 and 100 tests
Sample Diluent, 100 mL
Proficiency Samples
Sample Extraction kit, 20 tests

For ordering or technical assistance contact:

Sales Department
Ohmicron Environmental Diagnostics, Inc.
1-800-544-8881
1-215-860-5115
Fax 1-215-860-5213

L20043
6-98

RaPID Assays[®]

PCB in Soil

• Intended Use

For detection of Polychlorinated Biphenyls (PCBs) in soil.

• Materials Required but Not Provided

RaPID Prep[™] Soil Collection Kit and PCB Sample Extraction Kit.

• Procedural Notes and Precautions

Prepare soil samples for analysis according to the procedure given in the PCB Sample Extraction Kit. Then, follow the immunoassay procedure as described in the PCB RaPID Assay[®] Kit package insert.

As with all immunoassays, a consistent technique is the key to optimal performance. To obtain the greatest precision, be sure to treat each tube in an identical manner.

Add reagents directly to the bottom of the tube while avoiding contact between the reagents and the pipet tip. This will help assure consistent quantities of reagent in the test mixture.

Avoid cross-contaminations and carryover of reagents by using clean pipets for each sample addition and by avoiding contact between reagent droplets on the tubes and pipet tips.

• Quality Control

A control solution at approximately 3 ppb of PCB (as Aroclor 1254) is provided with the PCB RaPID Assay kit. It is recommended that it be included in every run and treated in the same manner as unknown samples. Once the control results are corrected for the dilution factors (see Results section) an acceptable result should be 2000 times the value stated on the vial, i.e. 8.0 - 1.2 ppm.

• Results

Multiply the sample and control results by the appropriate dilution factor introduced by the collection, extraction and extract dilution steps. When the collection/extraction/dilution procedure described in the PCB Sample Extraction Kit is performed with a ten gram soil sample, the RaPID Assay result is multiplied by 2000 to determine the soil PCB concentration. Alternatively, program the RPA-1 Analyzer as listed below to automatically correct for this dilution factor.

Using the RPA-1[™] RaPID Analyzer, calibration curves can be automatically calculated and stored. Refer to the RPA-1 operating manual for detailed instructions. To obtain results from the PCB RaPID Assay on the RPA-1 the following parameter settings are recommended:

Data Reduct : Lin. Regression
Xformation : Ln(LangeitB)
Read Mode : Absorbance
Wavelength : 450 nm
Units : PPM
Rpt Bk : 0

Calibration:
of Cals : 4
of Rpts : 2

Concentrations:
#1: 0.00 PPM
#2: 0.50 PPM
#3: 2.00 PPM
#4: 10.00 PPM

Range : 0.20 - 10.00
Correlation : 0.990
Rep. %CV : 10%

• Expected Results

In a study with 18 samples including both field contaminated soils and analytically spiked soil samples, the RaPID Prep PCB Sample Extraction Kit results were shown to correlate well with a gas chromatographic method $r = 0.8052$.

• Performance Data

Range of Detection

The PCB RaPID Assay has a range of detection in soil of 500 ppb to 10 ppm when used in conjunction with the PCB Sample Extraction Kit.

Recovery

PCB recoveries will vary depending on soil type, retention mechanism, solvent and extraction apparatus used, length of extraction period and levels of potentially interfering substances in the soil.

Two (2) soils of the clay and loam type were fortified with PCB (Aroclor 1254) to final soil concentrations of 5, 50 and 500 ppm. Average recovery of added PCB was 85%. Results ranged from 74 to 101%.

Precision

The overall coefficient of variation (%CV) for PCB measurement in soil spiked at 4 ppm using the RaPID Prep components and PCB RaPID Assay is less than 20%. This represents the amount of variability expected when a homogeneous soil sample undergoes ten replicate collections, extractions and dilutions generating ten immunoassay results from a single run.

Method	Sample Collection	
	by weight	by volume
# of replicates	10	10
mean results (ppm)	3.20	3.67
% CV	15.2	18.3

• Assistance

For ordering or technical assistance contact:
Omicron Environmental Diagnostics
Sales Department
Newtown, Pennsylvania 18940
(800)544-8881 • Fax (215)880-5213

• Availability

Omicron
PCB RaPID Assay
30 Test Kit
100 Test Kit
PCB Sample Diluent
PCB Proficiency Samples
RaPID Prep Soil Collection Kit
RaPID Prep PCB Sample Extraction Kit

20075 •

©1997 Omicron



PCB in Water

• Intended Use

For detection of Polychlorinated Biphenyls (PCBs) in water samples: groundwater, surface water, well water, effluent.

• Materials Required but Not Provided

Methanol HPLC Grade or equivalent.

• Sample Information

Water samples should be collected in glass vessels (Nalco in the cap liner). Immediately upon collection, samples should be diluted with an equal volume (1:1) of methanol HPLC grade to prevent absorptive losses to the glass containers.

After samples are diluted, those samples containing gross particulate matter should be filtered (e.g. 0.2 μ m Anopore™ 25 Plus, Whatman, Inc.) to remove particles.

• Procedural Notes and Precautions

Prepare water samples as described above. Follow the assay procedure as described in the PCB RaPID Assay® kit package insert.

As with all immunoassays, a consistent technique is the key to optimal performance. To obtain the greatest precision, be sure to treat each tube in an identical manner.

Add reagents directly to the bottom of the tube while avoiding contact between the reagents and the pipet tip. This will help assure consistent quantities of reagent in the test mixture.

Avoid cross-contaminations and carryover of reagents by using clean pipets for each sample addition and by avoiding contact between reagent droplets on the tubes and pipet tips.

• Results

Multiply the sample and control results by a factor of 2 to account for the initial 1:1 dilution of sample with methanol or alternatively program the RPA-I Analyzer as listed below to automatically correct for the dilution factor.

Using the RPA-I™ RaPID Analyzer, calibration curves can be automatically calculated and stored. Refer to the RPA-I operating manual for detailed instructions. To obtain results from the PCB RaPID Assay on the RPA-I the following parameter settings are recommended:

Data Reduct : Lin. Regression
X/formation : LnLogit8
Read Mode : Absorbance
Wavelength : 450 nm
Units : PPB
Rpt Blk : 0

Calibration:
of Cals : 4
of Rpts : 2

Concentrations:

#1: 0.00 PPB
#2: 0.50 PPB
#3: 2.00 PPB
#4: 10.0 PPB

Range : 0.20 - 10.0
Correlation : 0.990
Rep. %CV : 10%

• Expected Results

In a study with 30 water samples from locations across the U.S., the PCB RaPID Assay was shown to correlate well with gas chromatography (EPA SW-846 Method 8082) $r = 0.910$.

• Performance Data

Sensitivity

The PCB RaPID Assay has an estimated minimum detectable concentration in water, based on a 90% B/E of 200 ppt.

Recovery

Four (4) samples, including a municipal water source, drinking water from a local well, a sample from a local pond and a small creek were spiked with various levels of PCB (Aroclor 1254), diluted 1:1 with methanol, and then assayed using the PCB RaPID Assay. The following results were obtained:

Amount of PCB Added (ppb)	Recovery		
	Mean (ppb)	S.D. (ppb)	%
1.0	3.68	0.08	98
3.0	3.18	0.28	108
4.0	4.32	0.38	108
8.0	7.68	0.68	98
Average			99

Precision

The following results were obtained:

Control	1	2	3	4
Replicates	5	5	5	5
Days	5	5	5	5
n	25	25	25	25
Mean (ppb)	0.98	3.10	4.38	8.04
% CV (within assay)	8.5	8.2	4.8	4.9
% CV (between assay)	15.7	8.1	0.8	2.5

• Assistance

For ordering or technical assistance contact:

Chimicon Environmental Diagnostics
Sales Department
Newtown, Pennsylvania 18848
(800) 544-8881 • Fax (215) 860-5213

• Availability

Chimicon

PCB RaPID Assay
30 Test Kit
100 Test Kit
PCB Proficiency Sample
PCB Sample Kit
RaPID Prog™ Soil Collection Kit
RaPID Prog PCB Sample Extraction Kit



RaPID Prep™

PCB Sample Extraction Kit

• Intended Use

For use in conjunction with RaPID Prep™ Soil Collection Kit and the PCB RaPID Assay® Kit for determination of PCB in soil.

• Principle

Before the 1975 requirements of the Toxic Substances Control Act were put in place, polychlorinated biphenyls (PCBs) were produced in the United States for use in a wide variety of industrial applications including electrical transformers and capacitors, paints, inks and pesticides. The chemical and physical stability of these compounds can lead to long term environmental problems. The non-polar structure of this class of compounds imparts a hydrophobicity that allows PCBs to adsorb readily to soil and other solid surfaces. Accurate determination of the PCB content of soils suspected of contamination is necessary to make appropriate decisions regarding site cleanup and remediation.

The reagents contained in the RaPID Prep PCB Sample Extraction Kit have been optimized for fast, efficient removal of PCB from soil and convenient preparation of the sample for immunoassay or levels of interest to the investigator. The system allows for reliable, convenient and cost effective determinations at the field testing or remediation site.

• Description of Contents

1. PCB Extraction Solution
Methanol with soil dispersion agent.
per kit: 20 bottles containing 20 ml. each
2. PCB Extract Diluent
buffered saline solution containing preservatives and stabilizers without any detectable PCB.
per kit: 20 vials containing 25 ml. each
3. Twenty five microliter precision pipet.
4. Pipet tips
per kit: 21 disposable plastic tips
5. Chain of custody container labels.
per kit: 30 labels for diluent vials

can use for 1:1 dilution of 100 sample

• Reagent Storage and Stability

Store all reagents and components in a dry well ventilated area at 2-30°C. Reagents may be used until the expiration date shown on the vials.

Consult local, state and federal regulations for proper disposal of all reagents.

• Materials Not Provided

In addition to the materials provided, the following items will be necessary for the performance of this procedure:

- RaPID Prep Soil Collection Kit
- stopwatch or clock with second hand
- permanent marking pen
- protective gloves
- digital balance (optional, available from OhiMicron)

• Sample Information

This kit was validated for use with soil samples. Other types of sample matrices and solid wastes may require different procedures to extract PCB.

• Procedural Notes and Precautions

Do not use any reagent beyond its stated shelf life.

Sixty seconds of continuous agitation of the soil sample in the

presence of the extraction solution is important for good extraction efficiency. Use of a one minute timer or stopwatch to assure adequate shaking time is recommended.

Avoid contact of extraction solution (methanol) with skin and mucous membranes. If this reagent comes in contact with skin wash with water.

The twenty five microliter pipet is considered disposable and should be discarded after the kit reagents are depleted.

Due to the large dilution factor used, the accuracy of the final result will depend in part on the care taken in pipetting the soil extract into the diluent.

• Limitations

The PCB Sample Extraction Kit, when used in conjunction with RaPID Prep Soil Collection Kit and the PCB RaPID Assay, will provide screening results. Positive results may need to be confirmed by a non-immunological method.

• Extraction/Filtration Procedure

Read the Procedural Notes and Precautions and the RaPID Prep Soil Collection kit package insert before proceeding. Various soil sampling options are presented in the Soil Collection Kit package insert.

1. Write sample information on the labels provided for soil collection devices, extract collection vials and PCB Extract Diluent vials. Apply labels to appropriate vessels.

2. **Sampling:** Remove the screw cap from the soil collector and collect soil by volume or by weight, as follows:

2.1 **By volume:** With the plunger fully depressed (pushed to the top of the tube), pack soil into the open end of the collection tube. Unscrew the plunger rod from its plunger by turning the handle counterclockwise. Level the soil flush with the top of the collector tube using the plunger rod. Using the base portion of the handle, push the soil sample and the plunger to the bottom.

2.2 **By weight using digital balance:**

Option 1. Remove screw cap. Tare the soil collector with its plunger rod. Collect the soil "By volume," level it off and push the soil and plunger to the bottom of the tube. Reattach plunger rod and weigh the tube containing the soil. Subtract original weight from final weight to determine soil weight. Record the weight of the soil.

Option 2. Remove the screw cap and plunger rod from an empty collection tube. Position the plunger at the bottom of the collection tube. Attach the red base piece provided and place the tube in an upright position on the balance and tare weight. Weigh 10 ± 0.1 gram of soil into the tube. Record the soil weight.

3. Extraction

3.1 Position the soil collection tube containing a soil sample upright in the Styrofoam rack.

3.2 Pour the contents of one vial of PCB Extraction Solution into the collector. Screw the cap (with/without filter) on tightly and make sure that the base cap is secured.

3.4 **SHAKE VIGOROUSLY AND CONTINUOUSLY FOR AT LEAST 60 SECONDS.** Additional shaking may be required to break up large or dry soil aggregates.

3.5 Position the collection tube upright in the rack and allow the mixture to settle at least five minutes.

If batch processing is desired, up to 21 soil samples with added



extraction solution can be loaded into the rack inside the Soil Collection Kit box base; the box lid is put in place and the box is shaken vigorously for at least 30 seconds.

4. Filtration

- 4.1 Remove the screw cap and attach the filter cap. Hand tighten until resistance is felt.
 - 4.2 Attach the plunger rod to the plunger of the soil collector.
 - 4.3 Remove the filter cap and invert the soil collector so that the filter cone is positioned over a collection vial. Keep inverted for a few seconds to wet the filter and to allow the filtrate to drip through the filter into the filter cone.
 - 4.4 Apply slight pressure to the plunger handle. The filtrate will begin to flow more quickly as gentle pressure is continuously applied.
 - 4.5 Fill the vial with approximately 20 drops (1 mL of the filtrate). Cap the vial.
- This amount of filtrate is sufficient to perform multiple replicate analyses with RaPID Assay kits. The vial will hold up to 5 mL of filtrate if additional extract volume is desired. The filtrate containing PCB is stable when stored in the extract collection vial for one week at room temperature (15 to 30 °C).

• Dilution Procedure

Using the pipet provided, transfer 25 µL of the extract directly into a vial of PCB Extract Diluent (25 mL). Mix by inverting several times.

This mixture can now be measured as "sample" according to the package insert of the PCB RaPID Assay PCB RaPID Assay kit procedure step #3.)

• Calculation of Results

Calculate the PCB concentration in soil by multiplying the RaPID Assay result by the factors introduced by the procedure.

$$\text{RaPID Assay result (ppb)} \times \frac{\text{vol. Extractant (mL)}}{\text{wt. of soil (g)}} \times \text{dilution factor} =$$

$$\text{RaPID Assay result (ppb)} \times \frac{20}{\text{wt. of soil (g)}} \times 1000 =$$

• PCB soil concentration (ppb)

* NOTE:

$$\text{Dilution factor} = \frac{\text{vol. extract (mL)} + \text{vol. diluent (mL)}}{\text{vol. extract (mL)}}$$

$$= \frac{0.025 + 25}{0.025} = 1000$$

When the extraction/dilution procedure described above is performed with a ten gram soil sample the RaPID Assay result is multiplied by 2000 to determine the soil PCB concentration.

EXAMPLE: For a soil sample weighing 10.0 grams giving a PCB RaPID Assay result of 2.5 ppb:

$$2.5 \text{ ppb} \times 2000 = 5,000 \text{ ppb or} \\ 5.0 \text{ ppm PCB soil concentration}$$

• Range of Detection

When this extraction/dilution procedure is used in conjunction with RaPID Prep Soil Collection Kit and the PCB RaPID Assay kit, the range of detection in soil is 500 ppb to 10 ppm.

For samples with expected PCB concentrations greater than 10 ppm on initial screen, the diluted extract should be further diluted with PCB RaPID Assay Diluent/Zero Standard before testing. A discussion of dilution schemes for optimal interpretation of other

PCB soil concentrations is given in the PCB User's Guide available from the Obstruction Technical Service Department.

Immunoassay results that are above or below the limits of the RaPID Assay Kit standard curve are considered estimated concentrations. Extrapolated assay concentrations should never be multiplied by the dilution factor and reported as a soil PCB concentration.

Screening results

The PCB Sample Extraction Kit can be utilized as a screening test for a soil contamination level of interest. Immunoassay results like all analytical results possess an amount of variability that can be expressed as a confidence interval around the result. Data characterizing the method variation can be shown as normal statistical probabilities and the utility of a selected cutoff concentration can be estimated. The following table shows the frequency of positive and negative results for a screening scheme with a 3 ppm cutoff that assures less than 1% false negatives at a level of 5 ppm PCB in soil:

True soil PCB value (ppm)	Estimated Rate of Positive Results (%)	Estimated Rate of Negative Results (%)
2.0	< 0.1	> 99.9
2.5	0.4	99.6
3.0 (cutoff)	50.0	50.0
3.5	87.4	12.6
4.0	94.9	5.1
4.5	98.5	1.5
5.0 (detection level)	99.4	0.6
5.5	99.8	0.2
6.0	> 99.9	< 0.1
7.5	> 99.9	< 0.1

Similar estimates can be made for other PCB detection levels of interest. The PCB User's Guide provides additional information regarding utility of the method as a screening tool.

• Expected Results

In a study with 16 samples including both field contaminated soils and analytically spiked soil samples, the RaPID Prep PCB Sample Extraction kit results were shown to correlate well with a gas chromatographic method ($r = 0.905$).

Recovery

PCB recoveries will vary depending on soil type, retention mechanism, solvent and extraction apparatus used, length of extraction period, amount of agitation and levels of potentially interfering substances in the soil.

Two soils of the clay and loam type were fortified with PCB (Aroclor 1254) to final soil concentrations of 5, 50 and 500 ppm. All soils were then subjected to the above extraction/dilution procedure followed by additional dilutions if necessary to expand the detection range. Average recovery of added PCB was 95%. Results ranged from 74 to 101%.

Soil Contaminants

Some contaminants found in soils that also contain PCB's can interfere with the analysis and cause false positives, false negatives or both when the compound is present at elevated concentrations. Interferences were assessed by adding increasing concentrations of some relevant contaminants to blank and PCB spiked soils prior to the extraction procedure. The concentration of compound shown below produced no evidence of interference in a positive or negative direction in the 500 ppb to 10 ppm detection range of the procedure described above.

soil contaminant

trichloroethylene
gasoline
transformer oil
1-chloronaphthalene
1,2,4-trichlorobenzene
diesel fuel

concentration in soil producing no interference

100,000 ppm or 10%
25,000 ppm or 2.5%
5,000 ppm or 0.5%
2,000 ppm or 0.2%
1,000 ppm or 0.1%
1,000 ppm or 0.1%

If additional dilutions of the soil extract are made to detect soil PCB concentrations greater than 10 ppm, these interferences are diminished in direct proportion to the dilution made.

Aroclor Specificity

The PCB RaPID Assay kit has been calibrated with Aroclor 1254. The kit antibody binds with differing affinity to the other PCB Aroclors. Aroclors 1254 and 1260 react most strongly in the system while Aroclors 1248, 1262 and 1263 also give a somewhat significant response in the assay system. Percent cross reactivity of the common aroclors with the antibody is given in the PCB RaPID Assay package insert. Equivalent concentrations of the other Aroclors in terms of Aroclor 1254 can be obtained from information provided in the PCB User's Guide.

• Performance Data

Precision

The overall coefficient of variation (%CV) for PCB measurement in soil spiked at 4 ppm using the RaPID Prep components and PCB RaPID Assay is less than 20%. This represents the amount of variability expected when a homogeneous soil sample undergoes ten replicate collections, extractions and dilutions generating ten immunoassay results from a single run.

Sample Collection Method by weight by volume

	by weight	by volume
no. of replicates	10	10
mean assay result (ppm)	3.20	3.47
%CV	15.2	18.3

• Availability

From Obstruction

Description	Part Number
PCB Sample Extraction Kit (20 units)	A00137
RaPID Prep Soil Collection Kit (20 units)	A00127
Portable Digital Balance	A00131
PCB RaPID Assay	
100 tests	A00134
10 tests	A00133

• Assistance

For ordering or technical assistance contact:
Obstruction Environmental Diagnostics
Sales Department
Newtown, Pennsylvania 18840
(800)544-8791 • Fax (215)880-5213

200255

A107394





OHMICRON
... The Quality Difference

Product Profile

General Description

AUTOMATIC REPORTS

The microprocessor controlled RPA-I RaPID Analyzer™ automatically converts immunoassay optical readings to sample concentrations (ppb). Programs for every RaPID Assay method are stored along with previous calibration results for convenient instant operation. The fully automatic data reduction capability is important to integrate the logit and log-linear enzyme immunoassay procedures and produce directly reportable results.

SPECIAL OPTICAL SYSTEM

The specially designed optical system and dual wavelength path in the RPA-I RaPID Analyzer allow for direct readings through the round plastic RaPID Assay reaction tubes. This minimizes diffraction due to the curved surface and interference from scratches or finger marks. Readings, data reduction, storage and result printout are completed within one second of tube insertion, thus providing high throughput capability.

SIMPLE OPERATION

The self-prompting display facilitates quick and easy use. Method entry, test steps and error messages are all displayed. Output is through a back lighted liquid crystal display, a simultaneous thermal printer, or the RS-232 interface.

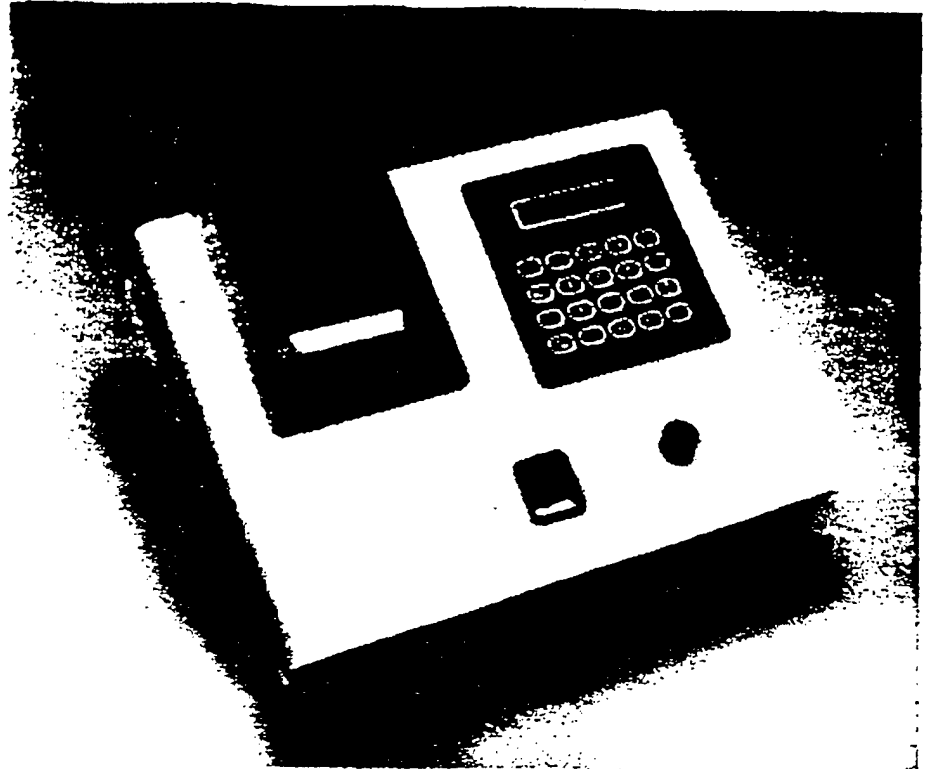
EASY PROGRAM EXPANSION

The RPA-I is pre-programmed to perform all current RaPID Assay methods. Program loading for new RaPID Assay kits is accomplished through a customer removable download cartridge or by a keypad procedure. The operating program software is contained in a similar cartridge for easy field update or software modification. Automatic battery back-up is incorporated to protect all protocols and data if power is lost or disconnected.

CONVENIENT

The compact size of the instrument makes it ideal for laboratories of any size. The robust design makes it easily transportable to other lab sites for convenient use.

The RPA-I RaPID Analyzer™



Features

- Easy** - Self prompting display for all steps.
- Fast** - Read times less than 1 second per tube with immediate printout.
- Convenient** - Automatic data reduction and readout in concentration units.
- Software Driven** - Microprocessor memory maintains all assay protocol and calibration data.
- Sophisticated** - Dual wavelength measurements produce interference free readings.
- Flexible** - Output on backlit liquid crystal display, thermal printer or RS-232.



Specifications

Mechanical

System configuration	Compact bench top instrument.
Tube types	Glass or plastic 12mm round tubes.
Read rate	1 sample per second.

Electro-Optical

Light source	High intensity quartz-halogen lamp.
Detector	Solid state photodiodes.
Wavelengths	340 nm to 630 nm.
Optics	Bichromatic.
Linear range	0-2.0A.
Voltage range	Adaptors available for 100/120 and 220/240 volts, 50 or 60 cycles.

Data Handling

Input	Membrane keyboard.
Display	Liquid crystal display.
Printer	Thermal dot matrix.
Data storage	Data storage capability for: — test protocols — calibration curves
Interface	RS232.

Ordering Information

Products

RPA-I Analyzer
Printer Paper (12 Rolls)
Filter Blocks
Service Contract

For ordering or technical
assistance call:

(800) 544-8881 • (215) 860-5115 • Fax (215) 860-5213



APPENDIX D - ANALYTICAL METHODS AK101, AK102, AND AK103

APPENDIX D

**Alaska Series Laboratory Methods
for the Analysis of**

**Gasoline Range Organics (AK101),
Diesel Range Organics (AK102), and
Residual Range Organics (AK103)**

Forward for All AK Series Methods

The Alaska Department of Environmental Conservation (ADEC) has published these laboratory methods in order to provide ADEC-approved laboratory test methods and related information for laboratory analysts, data users, and other interested parties. The test methods may be used, without permission, for laboratory testing to provide measurements relative to regulations in ADEC programs. Except where explicitly specified in a regulation, the use of these test methods is not mandatory.

These test methods have been written to provide comprehensive guidance for analysts attempting to analyze samples. However, ADEC does not intend for users to follow all details of a method in a prescriptive, rote fashion. Rather, except where specifically indicated by the words "shall," "must," or "required," analysts have the flexibility to modify method procedures, parameters, equipment, reagents, etc. for all method steps, as long as the changes do not adversely affect the method performance needed to achieve the data quality needs of the study being conducted. Examples of the types of flexibility allowed include changes in chromatographic conditions, columns, traps, sample extraction conditions, glassware and sample size.

The flexibility is intended to provide laboratories a way to improve test methods (e.g., reduce the generation of laboratory wastes, utilize existing equipment, reduce costs) without having to undergo elaborate studies and a time-consuming approval process. In exercising this flexibility, laboratories must be able to demonstrate and document that the changes implemented can produce results that are consistent with the data quality needs of the intended application, based on the results of initial and ongoing quality control activities.

Chapter One of EPA's publication SW-846 describes a variety of quality control activities that may be used to evaluate the appropriateness of any method modification and of the sample results. Additional quality control activities are described in the individual methods.

The test methods provide information relative to the expected performance (accuracy, precision and sensitivity) of the method when applied by a well operated laboratory. These performance data should be used both to assist in the selection of a method for a given application and to evaluate whether a modification is appropriate.

In summary, the test methods included in this appendix provide comprehensive guidance which may be used by laboratories, individual analysts, and the regulated community. The results from quality control sample analyses are used to evaluate the quality of sample results relative to the intended use of the data.

Method AK101
For the Determination of Gasoline Range Organics
Revision 3.0, 1/31/96

1. Scope and Application

1.1 Analytes

1.1.1 This method is designed to measure the concentration of Gasoline Range Organics (GRO) in water and soil. This corresponds to an alkane range from the peak start of C_6 to the peak start of C_{10} and a boiling point range between approximately 60°C and 170°C.

1.1.2 Components greater than or equal to C_{10} present in products such as diesel or fuel oil are detectable under the conditions of the method.

1.1.3 With the optional photo ionization detector (PID), this method can be extended for specific determination of volatile aromatics (BTEX) as specified in EPA methods 602 and 8020.

1.2 Quantitation Limits

1.2.1 The Practical Quantitation Limit (PQL) of this method for GRO is approximately 5 mg/kg GRO as gasoline for soils and 0.1 mg/L GRO as gasoline for water.

1.3 Dynamic Range

1.3.1 Dilutions should be performed as necessary to put the chromatographic envelope within the linear range of the method. In general, the approximate range is 0.5 to 2 mg/L of gasoline.

1.4 Experience

1.4.1 This method is based on a purge-and-trap, Gas Chromatography (GC) procedure. This method should be used by, or under supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatographs as a quantitative tool.

2. Summary of Method

- 2.1 This method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as gasoline. Other non-petroleum compounds, with similar characteristics and boiling points, may also be detected with this method. Samples must be analyzed utilizing purge-and-trap sample concentration. The gas chromatograph is temperature programmed to facilitate separation of organic compounds. Detection is achieved by a flame ionization detector (FID) or PID/FID in series. Quantitation must be performed by comparing the total chromatographic area between and including C₆ (hexane) and C₉ (nonane), to the peak start time of C₁₀, including resolved and unresolved components, based on FID response compared to a blended commercial gasoline standard (paragraph 3.2) and using forced baseline-baseline integration. See Table 1, for suggestions regarding purge and trap operating parameters.
- 2.2 Water samples can be analyzed directly for GRO by purge-and-trap extraction and gas chromatography. Soil or waste samples are dispersed in methanol to dissolve the volatile organic constituents. (See Table 2). A portion of the methanol solution is injected into water and then analyzed.
- 2.3 Special field sampling techniques are required to minimize the loss of volatiles from soil resulting from conventional sampling and sample handling techniques.
- 2.4 This version of the method was developed by Dr. Mary Jane F. Pilgrim, and is based in part on U.S. EPA SW-846 [1] methods 5030, 8000, 8020, 8015, a single laboratory method evaluation study conducted by the American Petroleum Institute (API) [2], work by the EPA Total Petroleum Hydrocarbons Methods Committee [3], and work by the Alaska Department of Environmental Conservation, State Chemistry Laboratory, with support from the Storage Tank Program.

3. Definitions

- 3.1 Gasoline Range Organics (GRO): All chromatographic peaks, both resolved and unresolved, eluting between the peak start time for C₆ (hexane) and the peak start time for C₁₀ (decane). Quantitation is based on a direct comparison of the baseline - baseline integrated area within this range to the total area of the calibration standard over the same C₆ - C₁₀) range using FID response.
- 3.2 Gasoline Calibration Standard (GCS): An equal-weight mixture of unleaded, leaded, and premium commercial gasolines mixed and diluted to appropriate concentrations, used to prepare a standard curve. In areas where leaded gasoline is not available, a second unleaded regular gasoline may be used to prepare the calibration standard. Leaded gasoline is generally available from retailers of aviation fuels.

- 3.3 Calibration Verification Standard (CVS): A gasoline quality control standard (preferably ERA Certified, or equivalent) prepared as in 3.2 but with product from a source other than that used to prepare the Gasoline Calibration Standard. It is used by the laboratory as a quality control check to verify the accuracy of calibration.
- 3.4 Continuing Calibration Standard (CCS): A mid-range working standard diluted from the Gasoline Calibration Standard, used to verify that the analytical system is operating in a manner comparable to that at the time of calibration.
- 3.5 Surrogate Control Standard (SCS): Either bromofluorobenzene or trifluorotoluene, or a mixture of both, used as a laboratory data quality control.
- 3.6 Surrogate Control Sample: A method blank sample spiked with the surrogate used in the method. The surrogate recovery is used to evaluate method control (see 7.3).
- 3.7 Laboratory Fortified Blank (LFB): A method blank sample spiked with a commercial gasoline other than the ones blended to prepare the GCS. The spike recovery is used to evaluate method control. The CVS is used in the Laboratory Fortified Blank.
- 3.8 Retention Time Window Standard : A normal alkane standard containing n-hexane and n-decane (C_6 and C_{10}) which is analyzed once per 24 hour day or with each batch of samples, whichever is less frequent, not to exceed 20 samples per batch. This standard is used to establish the retention time window for quantitation of GRO. The compounds of BTEX can be included if all quality control criteria are met (see section 10).
- 3.9 Other terms are as defined in SW-846 [1].

4. Interferences

- 4.1 High levels of heavier petroleum products such as diesel or heating fuel may contain some volatile components producing a response within the retention time range for GRO. Other organic compounds, including chlorinated solvents, ketones, and ethers are also detectable by this method. As defined in the method, the GRO results include these compounds.
- 4.2 Samples contaminated with a single compound which is detectable using this method (e.g., some solvents,) and which are quantitated against the GCS, may result in a value which is biased high for that compound. This is caused by the difference in response factors seen with the GCS and various solvents. An alternative detection and quantitation procedure may be more appropriate if the identity and quantity of the compound are specific project concerns.
- 4.3 Samples can become contaminated by diffusion of volatile organics during shipment and storage. A trip blank prepared from reagent water (for water samples) or methanol (for soil and sediment samples) and carried through sampling and subsequent storage and

handling is recommended to serve as a check for such contamination.

- 4.4 Contamination by carryover can occur when high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and purging device should be rinsed between samples with reagent water and methanol. If an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank or reagent water to check for contamination. For volatile samples containing high concentrations of water-soluble materials, suspended solids, high boiling compounds or organohalides, it may be necessary to wash the syringe or purging device with a detergent solution, rinse with distilled water and methanol, and then dry in a 105 °C oven between analyses. The trap and other parts of the system are also subject to contamination. Therefore, frequent bake-out and purge of the entire system may be necessary. A screening of all samples prior to analysis is recommended to protect analytical instrumentation (see 9.6.1).

5. Safety Issues

- 5.1 The toxicity or carcinogenicity of each reagent used 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. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in chemical analyses. Additional references to laboratory safety should be made available and identified for the information of the analyst. Some data (i.e., on methanol) is available from the Department.

6. Apparatus and Materials

(Unless otherwise indicated, all apparatus and materials are representative, not required.)

6.1 Glassware

6.1.1 40 mL glass vials with Teflon-lined septa and screw caps (a.k.a., VOA or VOC vials).

6.1.2 4 oz. amber glass wide mouth jars with Teflon-lined septa which are fused to the screw caps.

6.1.3 Volumetric flasks, class A: 10 mL, 50 mL, 100 mL, 500 mL and 1000 mL with ground glass stoppers.

6.1.4 Disposable pipettes: Pasteur.

6.2 Syringes

- 6.2.1 5 mL Luerlock glass syringe and 5 mL gas-tight syringe with shutoff valve.
- 6.2.2 For purging large sample volumes for low detection limit analysis, 25 or 50 mL syringes may be used. Remember to adjust other volumes as necessary throughout the method.
- 6.2.3 Microsyringes: 1, 5, 10, 25, 100, 250, 500 and 1000 μ L.
- 6.3 Analytical balance, capable of accurately weighing to the nearest 0.0001 g for preparation of standards and per cent moisture determinations, and a top-loading balance capable of weighing to the nearest 0.1 g for samples.
- 6.4 Stainless steel spatula
- 6.5 Gas Chromatography
 - 6.5.1 Gas Chromatograph: Analytical system complete with gas chromatograph suitable for purge-and-trap sample introduction and all required accessories, including detectors (FID required, additional PID optional), column supplies, gases and syringes. A data system capable of determining peak areas using a forced baseline and baseline projection is required. A data system capable of storing and reintegrating chromatographic data is recommended.
 - 6.5.2 Columns:
 - 6.5.2.1 Column 1: 105-m x 0.53 mm ID. Restek RTX 502.2, 0.3 micron film thickness or equivalent.
 - 6.5.2.2 Capillary columns may be essential to achieve necessary resolution. The column must resolve C_6 from the methanol solvent front in a mid-range LCS standard and, if BTEX is to be done simultaneously, must resolve ethylbenzene from m/p-xylene.
 - 6.5.2.3 The column must be capable of separating typical gasoline components from the surrogate and (optional) internal standard.
 - 6.5.3 Purge-and trap device: The purge-and-trap device consists of three separate items: the sample purger (sparging device), the trap, and the desorber (furnace). Several complete assemblies are commercially available. See Method AK 101.0, Table 1 for summary.

6.5.3.1 Purging chamber: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3-cm deep. The gaseous headspace between the water column and the trap should have a total volume of less than or equal to 15 mL. In any case, the purge chamber must be configured so that the quality assurance requirements specified in section 10 of this method are met.

6.5.3.2 Trap: The trap must be capable of retaining GCS components at the highest concentration of the calibration curve, and concomitantly meet the quality assurance requirements specified in section 10 of this method. Before initial use, the trap should be conditioned as specified by the manufacturer. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned, according to manufacturer's specifications, with back flushing. The trap may be vented to the analytical column during daily conditioning; however, the column should be run through the temperature program before analysis of samples to assure that any contamination from trap conditioning has been removed.

An alternate trap uses 7.6-cm Carboxpack B and 1.3-cm Carboxsieve S-III (Supelco Cat# 2-0321R). This trap should be desorbed at 240°C and baked to 300°C. Another useful trap is the "J" trap, and should be conditioned and used according to manufacturer's specifications.

6.5.3.3 Desorber (Furnace): The desorber should be capable of rapidly heating the trap to the required temperature for desorption. The trap should not be heated higher than the manufacturer specified tolerances.

6.5.4 The purge-and-trap device may be assembled as a separate unit or may be coupled to a gas chromatograph, as long as complete transfer of the sample is assured.

7. Reagents and Standards

7.1 Reagent Water: Carbon-filtered, purged water which has been shown to be free from purgable compounds (this has also been called organic-free water). Nitrogen or helium may serve as purge gas.

7.2 Methanol: Pesticide grade or equivalent. Store away from other solvents. At a minimum, the methanol must not show GRO contamination above the PQL.

7.3 Stock Standard Solutions - Prepare the following stock standards. Unless otherwise noted, all are prepared using the methanol listed in 7.2 as solvent. Standard preparation should follow guidelines in SW 846 [1]. All standards prepared by the laboratory must be stored without headspace at -10 to -20°C and protected from light. Standards must be replaced within 6 months of preparation. Standards should be checked regularly to assure their integrity. Standards which are purchased pre-made from commercial suppliers may be kept for the life, and under conditions, specified by the manufacturer if different than described in this paragraph.

7.3.1 Internal Standard: An internal standard (1-chloro-4-fluorobenzene) is recommended for 602/8020 quantitation on the PID. Due to potential interferences, the internal standard is not recommended for GRO (FID) quantitation.

7.3.2 Recommended Surrogate: 50 ug/mL of bromofluorobenzene and /or trifluorotoluene . Add 5.0 uL of this surrogate directly into the 5 mL syringe with every water sample and reference standard analyzed. Surrogate is spiked into soil samples during the extraction step (see 8.2.1). A second surrogate may be used in addition to, but not in place of, the surrogate sent to the field (8.2.1).

7.3.3 Retention Time Window Standard: This mixture of hexane and decane serves as a retention time window defining mix for GRO. The concentration of the individual components should not be less than 500 ug/mL and not more than 1000 ug/mL. Additional standards may be added to this mix if 602 or 8020 is to be done concomitantly.

7.3.4 Calibration Standards: A mixture of equal weights of leaded, unleaded and supreme gasolines serves as the Gasoline Calibration Standard. No fewer than 3 concentrations of the GCS are diluted directly into a 5 mL syringe (linear range approx. 0.5 to 2.0 mg/L) at the time of calibration. BTEX calibration should meet the criteria specified in EPA method 602 for waters [11] and in SW846 method 8020 for soils [1]. Other than one standard concentration near the practical quantitation limit, the expected range of concentrations found in real samples should define the working range of the GC (see 9.3.2).

7.3.5 Stock Standard for Calibration Verification:: From a blend of commercial gasolines other than those used to prepare the GCS, make an equal weight mixture as described in 7.3.4. Prepare a dilution of 500 ug/mL in methanol. Addition of the following amounts yields the indicated concentrations when preparing LFBs:

0.005 mL added to 5 mL water:	0.5 mg/L
0.5 mL added to 10 g soil:	25 mg/kg

When verifying the BTEX calibration curve, the criteria set forth in EPA method 602 should be met [11].

8. Sample Collection, Preservation, Handling, and Holding Times

8.1 Aqueous Samples:

8.1.1 Aqueous samples should be collected without agitation and without headspace in contaminant-free, amber glass 40-mL vials with Teflon-lined septa in the caps. A sufficient number of samples should be collected to provide for quality control criteria, and for back-up in the event of breakage. If amber glass vials are not available, clear glass may be substituted if the samples are protected from light. The Teflon layer must contact the sample (zero headspace). Sample vials should contain 200 μ L of 50% HCl as a preservation for volatile analytes. Refrigerated samples ($4^{\circ} \pm 2^{\circ}$ C) must be analyzed within 14 days of collection.

8.1.2 A trip blank (contaminant-free amber glass 40-mL vial with Teflon-lined septum, filled to zero headspace with purged, organic free water) must accompany all sampling kits, at a recommended ratio of 1 for every 10 samples collected, and should be stored and analyzed with the field samples.

8.2 Soils and Sediments: Soil and sediment samples require special procedures to minimize the loss of volatiles during transit from the field to laboratory.

8.2.1 Soil or sediment samples must be collected into appropriately sized containers and submerged in surrogate methanol.

8.2.2 Solid samples should be collected with minimum disturbance into tared 4 oz (or larger, if appropriate) jars with a Teflon-lined septum fused to the lid. 25 mL aliquots of methanol (includes 1.2 mL of a surrogate solution at 50 μ g/mL) should be carefully added to the undisturbed soil until the sample is submerged.

8.2.3 It is extremely important that the weight of the jar, the weight of the methanol/surrogate solution and the weight of the sample collected be known. These must either be measured directly, or sufficient information documented so that these weights can be calculated.

8.2.4 The ratio of soil to methanol used to calculate the MDL and PQL offered in this method was 1:1 (w:w). However, absorbent, organic soils such as muskeg and tundra will require a higher methanol-to-sample ratio, while beach sand may tolerate a lower ratio.

8.2.5 Soil for volatiles analysis can be collected using any coring device that minimizes

soil disturbance. Any scraping, stirring or similar activity will result in a loss of volatiles during sampling. A sufficient number of samples should be collected to provide for backup in the event of breakage.

- 8.2.6 Although it is not necessary to refrigerate all preserved samples at $4\pm 2^{\circ}\text{C}$ after collection and until analysis is complete, collected samples must be kept below 25°C .
- 8.2.7 A second surrogate added to the methanol and soil mixture after sample collection may be used in addition to, but not in place of, the surrogate with which the field methanol was prepared.
- 8.2.8 A reagent methanol trip blank should be prepared in the same manner as the sample vials, and should contain surrogated methanol. Trip blanks must accompany all sampling kits, at a recommended ratio of 1 for every 10 samples collected, and should be stored and analyzed with the field samples.
- 8.2.9 Field blanks may be added to the sampling protocol and are prepared in the field by addition of surrogated methanol to the prepared container, as required by the Assessment Firm or the Project Manager.
- 8.2.10 A sample of the same soil to be analyzed for GRO should be collected into a moisture-proof container for % moisture determination. This sample should be processed as soon as possible upon arrival at the laboratory to assure that the resulting moisture determination is representative of the preserved sample as surveyed.
- 8.2.11 Trip blanks, field blanks, method blanks, etc. should be prepared from the same batch of solvent, reagents and vials as are used for sample collection.
- 8.3 28 days is the maximum holding time for soil and sediment samples collected into methanol.
- 8.4 Because the jars are pre-weighed, it is extremely important that the sampler put evidence tape on the kit ONLY and not on the individual bottles. Removal of evidence tape is extremely difficult and the additional weight biases final results. Also, the glue on the evidence tape can contribute to the volatiles concentration in the sample (per Rocky Mountain Analytical).
- 8.5 Trip blanks, field blanks and bottle blanks should be prepared as appropriate to meet the quality assurance goals of the project plan.

9. Procedure

9.1 Volatile compounds are introduced into the gas chromatograph by purge-and-trap. Purge gas should be set at a flow rate of 25 - 40 mL/min. and purge time at 12 min., or conditions necessary to optimize the resulting chromatography.

9.2 Waters:

9.2.1 Purge-and-trap may be used directly on most water samples.

9.2.2 Water samples high in dispersed sediments (non-settling or slow settling solids) must NOT be filtered before analysis, as this results in loss of volatiles. In most cases, a muddy water sample can be left undisturbed until the solids settle out. An aliquot of the sample can then be taken with a 5 mL gas tight syringe, being careful not to disturb the sediment layer. Introduction of sediment into the purge device can result in occlusion of the frit, leading to incomplete purging of the sample and low-biased results. In any case, sample preparation should be noted, and an approximate volume given for the solids, if present.

9.3 Soils and Sediments:

9.3.1 Soils and solids are methanol extracted. An aliquot of the extract is added to reagent water and analyzed as in 9.10.

9.3.2 For best retention of volatile compounds, samples should be collected into tared, methanol and surrogate containing sample jars (see 8.2).

9.3.3 The entire volume of soil must be submerged in a methanol and surrogate solution.

9.3.4 Weigh the sample jar upon receipt and record the total filled weight. Swirl the jar gently for 2 minutes to be sure that the soil sample is dispersed into the methanol, and allow the sediment to settle. It is recommended that the meniscus of the methanol be marked and dated on the outside of the jar.

9.3.5 Best results are obtained by allowing the sample volatiles to equilibrate with the methanol for at least 48 hours before continuing with the analysis. However, this is not always possible. In any case, note the time difference between when the methanol was delivered into the soil sample and when analysis was initiated.

9.4 Soils and Sediments Collected without Methanol Preservation:

9.4.1 When solids are collected by the sampling techniques in SW-846 [1], volatile results are biased low. Therefore, data from these samples (collected without methanol preservative) must be reported as "greater than or equal to" the calculated mg/kg GRO as gasoline and may not be accepted as valid by state project managers.

9.4.2 To prepare extracts from these types of collection containers, gently mix the contents of the sample container with a narrow metal spatula. Do not discard any supernatant liquids, as the entire contents of the sample container must be represented.

9.4.3 For sediment/soil and waste that are insoluble in methanol, weigh 10 g (wet weight) of sample into a tared 20 mL vial, using a top loading balance. Note and record the actual weight to 0.1 g.

9.4.4 Quickly add 9.5 mL of methanol and 0.5 mL of the 50 ug/mL surrogate spiking solution to the vial (or, after adding spiking solution, fill to the line on the volumetric flask), cap and swirl (do not shake) for 2 min.

9.4.5 Allow sediment to settle. Note alternate sample preparation procedure on data transmittal.

Note: To avoid loss of volatile organics or cross contamination, these steps must be performed rapidly and without interruption, in a laboratory free from solvent fumes.

9.5 Methanol Soluble Solids:

9.5.1 For waste that is soluble in methanol weigh 1 g (wet weight), to the nearest 0.1 g, into a tared 10 mL volumetric flask.

9.5.2 Quickly add 9.5 mL of methanol and 0.5 mL of the 50 ug/mL surrogate spiking solution to the vial (or, after adding spiking solution, fill to the line on the volumetric flask), cap and swirl for 2 minutes, to disburse the waste into the methanol.

9.5.3 Allow sediment to settle, pipette an aliquot to an amber glass vial for storage at $4\pm 2^{\circ}\text{C}$ (zero headspace).

9.6 Sample Screening:

9.6.1 It is highly recommended that all samples be screened prior to analysis, as these samples may contain enough petroleum product to overload the column and/or detector(s). This screening step may be analysis of a solid sample's methanol extract (diluted) using AK101, the headspace method (SW-846 Method 3810 [1]) or the hexadecane extraction and screening method (SW-846 Method 3820 [1]).

9.7 Gas Chromatography Conditions (recommended)

9.7.1 Column 1: Set helium column pressure to 20#. Set column temperature to 30°C for 1 min., then ramp at a rate of $5^{\circ}\text{C}/\text{min.}$ to 100°C , then $8^{\circ}\text{C}/\text{min.}$ to 240°C and

hold for 7.5 min. Conditions may be altered to improve the resolution of GRO.

9.7.2 Other columns: Set GC conditions to meet the criteria in 6.5.2.2.

9.8 Calibration:

9.8.1 The GC system should be set up as in Section 6.5. This should be performed prior to calibration or to final preparation of the samples or sample extracts for analysis.

9.8.2 The GRO calibration curve must be represented by no less than 3 concentrations of GCS (a 5 point calibration curve is recommended). Prepare final solutions of GCS and Surrogate directly in a 5-mL glass syringe containing reagent water in the following manner: Using a microsyringe, add the aliquot of calibration standard directly to the reagent water in the glass syringe (refer to 9.10.7) by inserting the needle through the syringe opening. When discharging the contents of the microsyringe, be sure that the tip of the needle is well beneath the surface of the reagent water to prevent escape of calibration standard components. Similarly, add 5.0-uL of the 50 ug/mL SCS. Inject the prepared dilution(s) into the purge vessel(s) through the two way valve, and proceed with calibration.

9.8.3 Choose GCS concentrations to cover the GRO range expected in the samples or the linear range of the instrument, whichever is less. One of the concentrations must be near the practical quantitation limit. Due to potential carry over, it is recommended that not more than 10 ug of gasoline in 5 mL of water (2 mg/L) be purged. A calibration concentration at 0.01 mg/L (0.5 to 1.5 ug/L for individual volatiles) is recommended for additional quantitation if BTEX is to be included.

9.8.4 Tabulate the area response of the gasoline against mass injected. The ratio of the amount injected to the response, the response factor (RF), can be calculated for the standard at each concentration. If the percent relative standard deviation (%RSD) is less than 25% over the working range, linearity through the origin can be assumed, and the continuing calibration response factor can be used in place of a calibration curve. Use the average response factor from the calibration curve as reference.

External Standard Response Factor =
$$\frac{\text{Total peak area of standard}}{\text{Mass injected}}$$

Internal Standard Response Factor =
$$\frac{(A_x)(C_{is})}{(C_x)(A_{is})}$$

Where: A_x = Peak area response of analyte

A_{is} = Peak area response of internal standard

C_{is} = Concentration of internal standard

C_x = Concentration of analyte

9.8.5 The calibration curve must be confirmed using the CVS. This second source standard (7.5.3) verifies the accuracy of the calibration. The concentration of the CVS should be within the expected concentration range of the samples to be analyzed.

9.8.6 The working calibration curve or response factor must be verified on each working day by the injection of a midpoint CCS. The CCS is a diluted aliquot of the same standard used to initially calibrate the instrument. If the response factor for the CCS varies from the average response factor from the calibration curve (9.8.4) by more than 25% a new calibration curve must be prepared.

$$\text{Percent difference} = \frac{R_1 - R_2 \times 100}{R_1}$$

where:

R_1 = Average RF from the calibration curve.

R_2 = Response factor from CCS.

9.9 Retention Time Window

9.9.1 Before establishing windows, be certain that the GC system is within optimum operating conditions (6.5). Make three injections of the Retention Time Window Standard (7.3.3) throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

9.9.2 Calculate the standard deviation of the three absolute retention times for each component and for the surrogate.

9.9.2.1

The retention time window for individual peaks is defined as plus or minus three times the standard deviation of the absolute retention time for each component.

9.9.2.2

In those cases where the standard deviation for a particular analyte is zero, the laboratory should use ± 0.05 min. as a retention time window.

9.9.3 The laboratory must calculate retention time windows for each standard on each GC column and when a new GC column is installed or instrument conditions changed. The laboratory must retain the data and update it at least once a year.

9.10 Gas Chromatograph Analysis:

- 9.10.1 Samples are analyzed by GC/FID. Water, with or without methanol extract, to be analyzed for GRO is introduced into the programmed gas chromatograph (section 9.2) using purge-and-trap sample concentration.
- 9.10.2 If initial calibration (9.8) has been performed, verify the calibration by analysis of a mid-point CCS (9.8.6). With each day's run, open a 24 hour analysis window. This is done by running the Retention Time Window Standard.
- 9.10.3 A LFB at a concentration representative of the field samples being analyzed must also be run once every 20 samples and at the end of each sequence or twice in each batch, whichever is more frequent. If the result does not fall within the range specified in Table 3, corrective action must be performed. A matrix spike/matrix spike duplicate may be used in place of the LFBs if the quality control criteria specified in Table 1 for LFBs is met.
- 9.10.4 Calculate the percent difference of the response factor from the mid-point CCS from the mean response factor for each analyte to be quantitated (as in 9.8.4). This is done for GRO as a "group" from the CCS if GRO only is to be quantitated and for each of the components in the Retention Time Window Standard if additional quantitation for BTEX is required. If the response factors have a difference greater than 25%, corrective action must be taken.
- 9.10.5 A reagent water blank must be analyzed each day to determine the area generated from normal baseline noise under the conditions prevailing within the 24 hour period. Add 100 uL of methanol to the blank when soil or sediment extracts are to be analyzed. The noise area is generated by projecting a horizontal baseline between the retention times observed between the beginning of hexane and the beginning of decane. This lab control sample is integrated over the GRO area in the same manner as for the field samples and is reported as the reagent blank. **Do not blank subtract. This information is for data interpretation purposes only.**
- 9.10.6 Blanks should also be run after samples suspected of being highly concentrated, to prevent carryover. If the blank analysis shows contamination above the practical quantitation limit, the trap and column must be baked out and subsequent blanks analyzed until the system is shown to retain contaminants at concentrations less than the PQL.
- 9.10.7 Water samples may be introduced into the system in the following manner:
 - 9.10.7.1 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature and pour the sample into the syringe using

caution not to agitate the sample which would result in loss of volatiles. Replace the plunger and compress the sample. Invert the syringe so that the air bubble rises to the top (valve end) of the syringe. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Add 5 uL surrogate spiking solution through the valve bore of the syringe and proceed with analysis.

9.10.7.2 This process of taking an aliquot destroys the validity of the liquid sample for future analysis. Therefore, if there is only one 40-mL vial of sample, the analyst should fill a second syringe at the same time the first one is prepared, in the same manner, to protect against possible loss of sample integrity. This second sample is maintained at $4\pm 2^{\circ}\text{C}$ with valve closed only until such time as the analyst has determined that the first sample has been analyzed successfully. If a second analysis is needed, it must be from the second syringe and must be analyzed within 24 hours of the opening of the original sample vial. Care must be taken to prevent air from leaking into (and to prevent volatiles from leaking out of) the syringe containing the backup aliquot.

9.10.8 Methanol extracts from soils or sediments must be diluted into reagent water for analysis, as are methanol soluble dilutions. Table 2 is provided at the end of the method to help determine the volume of methanol extract to add to the 5 mL volume of reagent water, in order to keep the response of the major constituents in the upper half of the linear range of the curve. The maximum volume of methanol extract usable per 5 mL purge volume is usually 100 uL (this is used in calculating the PQL, section 1.2).

9.10.8.1 Follow directions for filling a syringe as outlined in 9.10.7.1, except use reagent water instead of sample. Introduce desired volume of methanol extract by inserting the needle of a microsyringe through the valve opening of the reagent water filled 5 mL syringe and depressing the micropipette plunger when the needle is well below the surface of the reagent water. The surrogate has already been added (see 8.2). Proceed with analysis.

9.10.9. Dilutions:

9.10.9.1 If the product concentration exceeds the linear range of the method as defined by the range of the calibration curve, the sample (or extract or dilution) must be diluted and reanalyzed. The response of the major peaks should be kept in the upper half of the linear range of the calibration curve.

9.10.9.2 It is most desirable to adjust the volume of extract introduced into the reagent water as in 9.10.8.1 in order to compensate for concentrated sample extracts. However, if that is not possible, the following procedure is appropriate for diluting samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe:

9.10.9.3 Dilutions may be made in class A volumetric flasks (10 mL to 100 mL seem most useful). Select the volumetric flask that will allow for the necessary dilution. Although intermediate dilutions may be necessary for highly concentrated samples, remember that the more transfers the sample makes, the greater the chance components will be lost.

9.10.9.4 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this to the flask.

9.10.9.5 Inject the proper aliquot of sample from the syringe prepared in Paragraph 9.10.7.2 into the flask. Aliquots of less than 1-mL are not recommended for dilution of water samples using this method. Make sure aliquot is introduced well below the surface of the reagent water in the volumetric flask to minimize sample loss.

9.10.9.6 Dilute the sample to the mark with reagent water, disturbing the surface as little as possible. Cap the flask and invert three times. Repeat the above procedure for additional dilutions. Continue as in 9.10.7.

9.10.10 Alternative Dilution Technique:

9.10.10.1 Alternatively, the dilutions can be made directly in the glass syringe to avoid loss of volatiles. If diluting methanol extracts, follow 9.10.8 using a smaller volume of extract in the 5 mL purge volume or the procedure outlined for the dilution of water samples.

9.10.10.2 Attach a syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject sample into the purging chamber. Proceed with the analysis. For more information, refer to purge-and-trap methods in SW-846 [1].

9.11. Moisture Determination for Solids

- 9.11.1 Moisture determinations must accompany all soils data (reported in mg/dry kg) so the client can, at will, determine the results in the original soil condition. Reporting in mg/dry kg can best be done if an unpreserved portion of the sample (collected without methanol) is provided. Because of the potential for high gasoline or related compound concentrations in the soil, all drying should be done under a functioning hood.
- 9.11.2 To determine percentage of moisture, pre-weigh an aluminum weighing boat. Weigh 5-10 g of the sample into the boat and record both weights to the nearest 0.001 g. Dry the sample overnight in a warm ($100 \pm 5^\circ\text{C}$) oven.
- 9.11.3 Remove the sample from the oven and cool in a desiccator until the sample reaches room temperature, and weigh to the nearest 0.001g. Record the weight.
- 9.11.4 Return the soil sample to the oven for an additional time period (not less than 2 hours), cool again in the desiccator until the sample reaches room temperature, and weigh to the nearest 0.001g.
- 9.11.5 If the weight of the sample has remained constant ($\pm 4\%$) from the initial "dry" weight (9.11.4), use this number for the moisture determination (see 9.12.2). If the second weighing shows that the sample has lost further weight, continue drying and weighing the sample until the weight becomes constant, then proceed to 9.12.2.
- 9.11.6 If a sample contains a high concentration of petroleum product, constant weight may be difficult to attain. If, after several tries, the $\pm 4\%$ criteria cannot be reached an estimated % moisture may be reported with appropriate explanation.

9.12 Calculations:

9.12.1 External Standard Calibration:

The concentration of Gasoline Range Organics in the sample is determined by calculating the absolute weight of analyte purged, from a summation of peak response for all chromatographic peaks, resolved and unresolved, eluting between the peak start time for C_6 (hexane) and the peak start time for C_{10} (decane), using the calibration curve or the calibration factor determined in 9.8 and baseline-baseline projection. Refer to Section 9.9 (Retention Time Window.)

The concentration of GRO may be calculated as follows [Method 8000A, 1]:

Aqueous Samples:

$$C_s (\text{mg/L}) = \frac{(A_x)(D)}{(Rf)(V_s)}$$

Where:

- C_s = Concentration of Gasoline Range Organics
 Rf = Response factor, as described in 9.8.4
 A_x = Response for the Gasoline Range Organics in the sample, units in area
 V_s = Volume of sample purged, in liters.
 D = Dilution factor, if dilution was performed on the sample prior to analysis. If no dilution was made, $D = 1$, dimensionless.

Solid samples (methanol extraction):

$$C_s (\text{mg/kg}) = \frac{(A_x)(V_t)(D)}{(Rf)(W)(V_i)}$$

Where:

- V_t = Volume of total extract (uL) (use 10000 uL for standard 10 mL extract volume).
 V_i = Volume of extract actually purged (uL)
 W = Weight of sample extracted, kg. The wet weight is used.
 A_x , Rf , and D have the same definition as above.

Note: Some chromatographic software programs are capable of performing these calculations with minimal analyst intervention.

9.12.2 Moisture Determination (%)

$$\text{Moisture (\%)} = (A-C)/(A-B) \times 100$$

Where:

- A = weight of aluminum boat + wet sample
 B = weight of boat
 C = weight of boat + dry sample

9.12.3 Internal Standard Calibration.

If internal standard calibration is used, please refer to SW 846 Method 8000A[1].

10. Quality Control (See Method AK 101.0, Table 3)

- 10.1 The laboratory must demonstrate, through the analysis of quality control check standards,

that the operation of the measurement system is in control. This must include the analysis of QC check samples plus the calculation of average recovery and the standard deviation of the recovery as outlined in Method 8000, Section 8.0, and in this method.

- 10.2 After successful calibration (Section 9.3), analyze a reagent blank sample. The reagent blank must be analyzed with every analytical batch. The surrogate recovery must be within established limits (see Table 3), or within the limits established by the project plan (whichever is more stringent). Also, the mid-point CCS must be analyzed at the end of each sequence and once per 20 samples, and compared to the successful calibration as described in 9.8.6, and fall within established limits (see Table 3).
- 10.3 With every batch, duplicate LFBs must be analyzed. The matrix for these samples should be reagent water for batches of aqueous samples or methanol/Ottawa sand (or other appropriate standard soil) for soil sample batch analyses. The accuracy and precision of the duplicates must be within established limits (see Table 3).
- 10.4 With every batch of samples extracted, the reagent blank must be analyzed. The reagent blank must have GRO less than the practical quantitation limit.
- 10.5 If any of the criteria in 9.3, 10.2, 10.3 and 10.4 are not met, corrective action must be taken before samples are analyzed.
- 10.6 Calculate the surrogate recovery in each sample. If recoveries are outside established limits (Table 3), verify calculations, dilutions, and standard solutions. Verify instrument performance.
 - 10.6.1 High recoveries may be due to a coeluting matrix interference -examine the sample chromatogram.
 - 10.6.2 Low recoveries may be due to adsorption by the sample matrix (i.e., high humus soils).
 - 10.6.3 Low recoveries may be due to a poor purge (clogged purge tube or frit). If this is suspected, check the purge tube with a blank before reanalyzing the sample.
 - 10.6.4 If the surrogate recovery is outside established limits due to suspected matrix effects, GRO results must be flagged. If the surrogate recovery is less than 50%, and the calculated GRO results are within a factor of 2 of the action limit, the laboratory should recommend that the client resubmit the sample for matrix spike and matrix spike duplicate analysis. This is a recommendation, not a requirement of the method, and therefore, the onus is not on the analytical laboratory to absorb the cost of the additional analyses.
- 10.7 Bottle blanks and matrix spikes are recommended for specific sampling programs. Field blanks, trip blanks, field duplicates are required as stated in Chapter 2, section 9 of the UST Procedures Manual.

- 10.8 Minimum quality control acceptance criteria are set forth in section 10 of this method. More stringent quality control criteria may be required by specific project plans.

10.9 Corrective Action

10.9.1 Calibration

10.9.1.1 If the initial calibration does not meet the criteria set forth in 9.8.4 and 9.8.5, the instrument must be recalibrated.

10.9.1.2 If the continuing calibration does not meet the criteria set forth in 9.8.6 and Table 3, the instrument must be recalibrated.

10.9.2 Surrogates

10.9.2.1 If surrogates are outside established control limits (Table 3), the following assessments and/or correction actions must occur:

A) Check to be sure there are no errors in calculations and that the concentration of the surrogate and internal standard solution are correct.

B) Check instrument performance to determine if it is within acceptable guidelines.

C) Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

D) Reprepare and reanalyze the sample if none of the above resolves the problem.

10.9.2.2 If the surrogate recoveries that are outside the control limits cannot be attributed to lab error, the decision to reanalyze or flag the data should be made in consultation with the client. Provided all other QC acceptance criteria are met (section 10), it is only necessary to reprepare/reanalyze a sample one time to demonstrate that a poor surrogate recovery is due to matrix effects. A relationship can be established between surrogate recovery and moisture content of organic soils, which may help in diagnosing the cause of poor surrogate recoveries.

10.9.3 Blanks: Additional laboratory and field quality control blanks may be necessary for certain projects to meet the goals of Chapter 2, section 9 of the UST Procedures Manual.

10.9.3.1 Instrument Blanks:

Instruments must be evaluated with each batch (or daily,

whichever is more frequent) and must demonstrate that the analytical system is free from contamination. This is best accomplished by analyzing an Instrument Blank.

10.9.3.2 Trip Blank:

Trip Blanks must be analyzed with each sampling batch IF the results of the field samples show contamination above the MCL. The Trip Blank for AK101 may also serve as the Method Blank and Reagent Blank in some cases.

10.9.3.3 Field Blank:

If the field samples yield GRO above the MCL, and contamination is found above the PQL in the Trip Blank, a Field Blank must be analyzed to identify whether the source of contamination originated in the field sample collection procedure, during travel or during storage in the laboratory.

(Note: Blanks are reported by value. DO NOT BLANK SUBTRACT. This information is for data quality assessment purposes only.)

10.9.4 Laboratory Fortified Blanks

10.9.4.1 If the analyte recovery from the LFBs is outside the established recovery limits (Table 3), the following assessments and/or corrective actions must occur:

- A) Check to be sure there are no errors in calculations and that the concentration of the analyte solution is correct.
- B) Check instrument performance to determine if it is within acceptable guidelines.
- C) Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.
- D) Reprepare and reanalyze the samples if none of the above resolves the problem.

10.9.4.2 If the relative percent difference between the LFB results exceeds the control limits, but meets the percent recovery criteria (Table 3), the following assessments and/or corrective actions must occur:

- A) Check to be sure that there are no errors in calculations, and that the same amount and source of analyte solution, solvent and water were used for both samples in the set.

B) Check to determine if instrument performance is still within acceptable guidelines, and that conditions did not change during the course of the batch analysis.

C) Recalculate the data if calculation error is suspected.

D) Repeat the LFB duplicate extraction and analysis, along with a representative number of samples (10% of the samples from the batch OR 1 sample, whichever is more) from the analytical batch with the failed LFB RPD. The re-analysis of the field samples is to demonstrate comparability of the extraction/analysis conditions at the time of re-extraction and analysis to those at the time of the failed QC.

11. Method Performance

11.1 Single-lab method performance data for the methanol extraction method in Ottawa Sand and other soil types is presented below. Additional method performance data is available through the State of Alaska, Department of Environmental Conservation.

11.2 Results for gasoline spikes (Methanol extraction purge and trap, soils)

<u>Matrix</u>	<u>Gasoline Spike Amount</u> <u>mg/kg</u>	<u>Percent</u> <u>Recovery</u>
Ottawa Sand ¹	50	70
Ottawa Sand ¹	50	78
Houston Black Clay ¹	50	68
Houston Black Clay ¹	50	66
Norwood Loam ¹	50	60
Norwood Loam ¹	50	57
Ottawa Sand ²	50	97
Ottawa Sand ²	50	96
Marine Sand ²	50	94
Glacial Clay ²	50	68
River Sediment ²	50	53
Marine Sediment ²	50	132
Forest Loam, muskeg, tundra ^{2,3}	50	28

1 Analyses performed by Rocky Mountain Analytical. Gasoline used = API PS6.

2 Analyses performed by State of Alaska, DEC Laboratory. Gasoline used = GCS.

3 All highly organic, high moisture soil matrices showed less than 30% analyte recovery.

11.3 The method detection limit calculated according to 40 CFR, Part 136, Appendix B was 0.5 mg/kg GRO as gasoline for the methanol extraction of soils and .01 mg/L GRO as gasoline for waters. The recommended Practical Quantitation Limit (PQL) is 5 mg/kg GRO as gasoline for soil and 0.1 mg/L GRO as gasoline for water. For purposes of this method, the PQL is defined as 10 times the MDL.

11.4 The PQL must be no more than 0.1 times the Maximum Contaminant Limit for the project.

12. References

- 12.1 USEPA "SW-846 Test Methods for Evaluating Solid Waste", 3rd Edition; Methods 5030, 8000, 8015 and 8020.
- 12.2. "Sampling and Analysis of Gasoline Range Organics in Soils," American Petroleum Institute Pub. #4516, October 1991.
- 12.3. "Evaluation of Proposed Analytical Methods to Determine Total Petroleum Hydrocarbons in Soil and Groundwater" prepared by Midwest Research Institute for USEPA Office of Underground Storage Tanks, August 14, 1990.
- 12.4 Urban, M.J., J.S. Smith, E.K. Schultz, R.K. Dickson, "Volatile Organic Analysis for a Soil, Sediment or Waste Sample" in Fifth Annual Waste Testing and Quality Assurance Symposium; USEPA, July 24-28, 1989.
- 12.5. Siegrist, R.L., and P.D. Jenssen, "Evaluation of Sampling Method Effects on Volatile Organic Compound Measurements in Contaminated Soils", Environmental Science and Technology, Vol. 24, November 1990.
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- 12.8. Hughes, B.M., D.E. McKenzie, C.K. Trang, L.S.R. Minor, "Examples of the Use of and Advanced Mass Spectrometric Data Processing Environment for the Determination of Sources of Wastes" in Fifth Annual Waste Testing and Quality Assurance Symposium; USEPA, July 24-28, 1989.
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- 12.10. "Volatile Organic Analysis for a Soil, Sediment or Waste Sample (The Methanol Method)," a symposium prepared by Dr. James S. Smith for the State of Alaska, Department of Environmental Conservation, Underground Storage Tank/Leaking Underground Storage Tank program, August 16, 1993.

Method AK 101.0 - Table 1
Purge and Trap Operating Parameters
For GRO/602/8020

<u>Parameter</u>	<u>Setting</u>
Purge Gas	Nitrogen or Helium
Purge Gas Flow Rate (mL/min.)	40
Purge Time (min.)	12.0 $\pm$ 0.1
Purge Temperature (°C)	Ambient
Desorb Temperature (°C)	180
Back Flush Inert Gas Flow (mL/min.)	20-60
Desorb Time	4
Trap Bake-out Time	10 min.

Method AK 101.0 - Table 2

Quantity of Methanol Extract Needed for
Analysis of Soils and Sediments

<u>Approximate Concentration, GRO (mg/kg)^a</u>	<u>Volume of Methanol Extract (uL)^b</u>
5-100	100
200	50
1000	10
5000	100 uL of 1/50 dilution ^c

Calculate appropriate dilution factor for concentrations exceeding this table.

- a This number is determined by sample pre-screening.
- b The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a total volume of 100 uL of methanol for each blank, sample and control.
- c Dilute an aliquot of the methanol extract and then take 100 uL for analysis.

Method AK 101.0 - Table 3

Acceptance Criteria for Quality Control

<u>ANALYTE</u>	<u>SPIKE CONCENTRATION</u>		<u>% Recovery</u>	<u>CONTROL LIMITS</u> Relative <u>% Difference</u>
	<u>Water (mg/L)</u>	<u>Soil (mg/kg)</u>		
Lab Fortified Blanks				
Gasoline Range Organics	0.5	25	60-120	20
Laboratory Sample Surrogate Recovery Trifluorotoluene or Bromofluorobenzene	0.05	2.5	60-120	
Field Sample Surrogate Recovery Trifluorotoluene or Bromofluorobenzene	0.05	2.5	50-150	
Continuing Calibration/ Calibration Verification Standards See 7.3	1.0		75-125	

The quality control criteria listed in this table represent the minimum acceptable levels, using highly organic soil matrices. Higher performance may be required on some projects.

Method AK 102
For Determination of Diesel Range Organics

Revision 3, 1/31/96

1. Scope and Application

1.1 Objectives

1.1.1 This method is designed to measure the concentration of Diesel Range Organics (DRO) in water and soil. This corresponds to an n-alkane range from the beginning of C₁₀ to the beginning of C₂₅, and a boiling point range of approximately 170° C to 400° C. (See Method AK 102.0, Table 1).

1.1.2 Components greater than C₂₄ present in products such as motor oils or lubricating oils are detectable under the conditions of the method.

1.2 Quantitation Limits

Practical quantitation limits (PQL) for this method for analysis of DRO are based on 100 ug/mL of diesel #2 in the extract and are approximately 0.10 mg/L for waters and 4.0 mg/kg for soils.

1.3 Dynamic Range

Dilutions should be performed as necessary to put the chromatographic envelope within the linear range of the method. Linear range is dependent in part upon column type, detector sensitivity, and injection volume. Typically, the approximate range is 0.01 mg/L to 100 mg/L as diesel.

1.4 Experience

This method is based on a solvent extraction, gas chromatography (GC) procedure. This method should be used by, or under the supervision of, analysts experienced in the use of solvent extractions and gas chromatographs as quantitative tools.

2. Method Summary

2.1 This method provides gas chromatographic conditions for the detection of semi-volatile petroleum products such as diesels. Other, non-petroleum compounds, with similar characteristics and boiling points, may also be detected with this method. One liter of Samples must be spiked with a surrogate compound and extracted with methylene chloride. The extract is dried and concentrated. An aliquot of the extract must be injected into a capillary column gas chromatogram equipped with a flame ionization

detector (FID), which has been temperature programmed to facilitate separation of organic compounds. Quantitation must be 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 components, based on FID response compared to a blended commercial diesel standard (see paragraph 3.2). Integration must be performed using forced baseline-baseline integration.

- 2.2 This version of the method was developed by Dr. Mary Jane Pilgrim, 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 [11], 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 based in part on US Environmental Protection Agency Methods 8000 and 8100, SW-846, "Test Methods for Evaluating Solid Waste," 3rd Edition [1], 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. Definitions

- 3.1 Diesel Range Organics (DRO): All chromatographic peaks, 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.
- 3.2 Diesel Calibration Standard (DCS): A blend of equal weights of arctic diesel, diesel #1 and diesel #2 (1:1:1), diluted to appropriate concentrations in methylene chloride or acetone, as appropriate. In those areas where arctic diesel is unavailable, kerosene-K2 may be used to prepare the calibration standard. This deviation must be noted on the final report. The DCS mixture serves as a calibration standard for DRO.
- 3.3 Surrogate Control Standard (SCS): Ortho-terphenyl or equivalent, used as a laboratory data quality control.
- 3.4 Surrogate Control Sample: A method blank sample spiked with surrogate. The surrogate recovery is used to evaluate method control (see Method AK 102.0, Table 2).
- 3.5 Calibration Verification Standard (CVS): A quality control standard (preferably Environmental Resources Association (ERA) Certified, or equivalent), prepared as in 3.2 but with products from a source other than those used to prepare the Diesel Calibration Standard. It is used by the laboratory as a quality control check to verify the accuracy of calibration.
- 3.6 Laboratory Fortified Blank (LFB): A method blank sample spiked with a commercial diesel fuel other than those blended to make the Diesel Calibration Standard (3.2). The spike recovery is used to evaluate method control (see Method AK 102.0, Table 2). The CVS may be used in the LFB.

- 3.7 Retention Time Window Standard: A mixture of the normal alkanes n-decane and n-pentacosane (C_{10} and C_{25}) which is analyzed once every 24 hour "day" or with each batch of samples, whichever is less frequent, not to exceed 20 samples per batch. This standard serves to define the retention time window for DRO.
- 3.8 Internal Standard: Alpha androstane, used to normalize DRO concentrations. Use of an internal standard is recommended, but not required.
- 3.9 Standard Soil: Ottawa sand, Norwood loam, Houston black clay, or other standard soil with characteristics which match the field samples as closely as possible, used in quality control standards.
- 3.10 Continuing Calibration Standard (CCS): A mid-range working standard diluted from the Diesel Calibration Standard, used to verify that the analytical system is operating in a manner comparable to that at the time of calibration.
- 3.11 Other terms are as defined in SW-846 [1].

4. Interferences

- 4.1 Other organic compounds including, but not limited to, animal and vegetable oil and grease, chlorinated hydrocarbons, phenols, phthalate esters, and biogenic terpenes are measurable under the conditions of this method. Heavier petroleum products such as lubricating oil and crude oils also produce a response within the retention time range for DRO. As defined in the method, the DRO results include these compounds.
- 4.2 Method interferences may be reduced by washing all glassware with hot soapy water and then rinsing it with tap water, methanol, and methylene chloride. Heating the glassware to reduce contaminants should not be necessary if this cleaning method is followed. At least one blank must be analyzed with each extraction batch to demonstrate that the samples are free from method interferences.
- 4.3 High purity reagents such as Burdick and Jackson GC² methylene chloride or Baker capillary grade methylene chloride must be used to minimize interference problems.
- 4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by a solvent blank to check for instrument contamination.

5. Safety Issues

- 5.1 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. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should also be made available to all personnel involved in chemical analysis. Additional references to laboratory safety should be available and identified for use by the analyst.

5.2 A hearing protection device should be used when performing sonication.

6. Apparatus and Materials

(Unless otherwise indicated, all apparatus and materials are suggested.)

6.1 Glassware

6.1.1 4 oz. amber glass wide mouth jars with Teflon-lined screw caps

6.1.2 Separatory funnel - 2000 mL with Teflon stopcock

6.1.3 Continuous liquid-liquid extractor - equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, New Jersey, P/N6841-10, or equivalent).

6.1.4 Concentrator tube. Kuderna-Danish 10 mL graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

6.1.5 Evaporative flask, Kuderna-Danish 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

6.1.6 Snyder column, Kuderna-Danish three ball macro (Kontes K-503000-0121 or equivalent). Rotary evaporation set-up may be used alternatively.

6.1.7 Jars: One liter amber glass, with Teflon lined screw caps.

6.1.8 Two mL glass vials with Teflon-lined cap (autosampler vials).

6.1.9 Disposable pipettes: Pasteur.

6.1.10 Graduated cylinders: 250 mL.

6.1.11 Glass or Teflon funnels.

6.2 Boiling chips - Approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.

6.3 Micro syringes: 1 ul, 5 ul, 10 ul, 25 ul, and 100 ul.

6.4 Water bath - Heated with concentric ring cover, capable of temperature control ($\pm 2^{\circ}\text{C}$). The bath should be used in a hood.

6.5 An analytical balance capable of accurately weighing 0.0001 g should be used for preparing standards and % moisture determinations. A top-loading balance capable of weighing to the nearest 0.1 g should be used for sample preparation.

6.6 Stainless steel spatula.

6.7 Gas Chromatography

6.7.1 Gas Chromatograph: Analytical system including appropriate gas supply and all required accessories, including a Flame Ionization Detector (FID), column supplies, gases, and syringes. A data system capable of determining peak areas using a forced baseline - baseline projection is required. A data system capable of storing and reintegrating chromatographic data is recommended.

6.7.2 Columns

6.7.2.1 Column 1: 25 M x 0.25 mm Quadrex 007 5% methyl phenyl 0.5 micron film thickness.

6.7.2.2 Alternate column: 30 M x 0.53 mm ID Restek RTX-5, 1.5 micron film thickness

6.7.2.3 Other Columns may be used - capillary columns may be essential to achieve the necessary resolution. The column must resolve C_{10} from the solvent front in a midrange DCS or CVS and, if AK103 is to be done simultaneously, must resolve C_{24} from C_{25} .

6.8 Sonication

6.8.1 Ultrasonic cell disrupter: A horn-type sonicator equipped with a titanium tip should be used. A Heat Systems-Ultrasonics, Inc. Model W-385 (475 watt) sonicator or equivalent (power wattage must be a minimum of 375 with pulsing capability and No. 200 $\frac{1}{2}$ inch Tapped Disrupter Horn) plus No. 207 $\frac{3}{4}$ inch Tapped Disrupter Horn, and No. 419 $\frac{1}{8}$ inch Standard tapered Microtip probe.

6.8.2 A Sonabox or equivalent is recommended with the above disrupter for decreasing sound (Heat Systems-Ultrasonics, Inc. Model 432 13 or equivalent).

6.9 Soxhlet extraction apparatus as described in SW-846 Method 3540 [1].

6.10 Nitrogen evaporator with high purity (grade 4.5 or equivalent) nitrogen gas source.

7. Reagents and Standards

- 7.1 Reagent Water: Water that has been shown to be free from DRO compounds - a Millipore system or equivalent is recommended.
- 7.2 Methylene Chloride, Hexane, Acetone - pesticide grade or equivalent. At a minimum, the solvents must be shown to be free from DRO.
- 7.3 Sodium Sulfate - (ACS grade) granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray or by extracting three times with methylene chloride and drying at 100±5° C. Incomplete cleaning of sodium sulfate can result in DRO contamination of samples.
- 7.4 Stock Standard Solutions - Prepare the following stock standards. Unless noted, all are prepared in the methylene chloride listed in 7.2 above. Standard preparation should follow guidelines in SW846 [1]. All standards prepared by the laboratory must be stored without headspace at -10 to -20°C and protected from light. Marking of the meniscus is helpful in maintaining stock standard integrity. Standards must be replaced within 6 months of preparation. Standards should be checked regularly to assure their integrity. Standards which are purchased pre-made from commercial suppliers may be kept for the life, and under the conditions, specified by the manufacturer if different than described in this paragraph.
 - 7.4.1 Optional Stock Internal Standard: 1000 ug/mL 5 alpha-androstane. Other internal standards may be used provided they do not interfere with the DRO components.
 - 7.4.2 Recommended Surrogate Control Standard: 200 ug/mL ortho-terphenyl (OTP). A working solution is made at 20 ug/mL (recommended concentration) in acetone.
 - 7.4.3 Diesel Calibration Standard: A blend of equal weights of diesel fuel, mixed together to form a composite diesel fuel (1:1:1, arctic diesel : diesel #1 : diesel #2) is used to prepare stock calibration standards in methylene chloride. No fewer than 3 concentrations of this DCS are used for instrument calibration. A five point calibration curve is recommended. Other than one standard concentration near the practical quantitation limit, the expected range of concentrations found in project samples should define the working range of the GC. If arctic diesel is not available, kerosine-k2 may be used in its place. This substitution, if used, must be noted on the final data reports. A mid-range dilution of this blend serves as the Continuing Calibration Standard.
 - 7.4.4 Retention Time Window Standard: A stock solution of C₁₀ and C₂₅ each at a level of at least 2000 ug/mL. This blend of alkanes serves as a retention time window defining mix for DRO.
 - 7.4.5 Stock CVS: From a blend of commercial diesels other than those used to prepare

the DCS, make an equal weight mixture as described in 7.4.3. A working solution is made at a recommended concentration of 5000 ug/mL in acetone.

8.0 Sample Collection, Preservation, Containers, and Holding Times

- 8.1 Water samples are collected, in duplicate, in one liter amber glass containers with Teflon-lined screw caps and acidified to pH 2 or less with HCl.
- 8.2 Soils are collected in a core tube, or 4 or 8 oz amber glass jar with Teflon-lined lid. The samples are stored at $4^{\circ} \pm 2^{\circ}$ C from the time of collection until extraction. Extraction must be performed on waters within 7 days and soils within 14 days [1]. All analyses of extracts must take place within 40 days.
- 8.3 Soil samples to be analyzed for both volatiles and DRO may be collected in the same, methanol preserved container and stored as for GRO (AK101). If this option is selected, the mechanics of the collection, preservation and container should be discussed with the client before sampling kit preparation. DRO extraction and analysis must still meet the requirements of 8.2, above.

9. Procedure

9.1 Sample Preparation

The preferred method for water extraction is SW-846 Method 3510 (Separatory Funnel Liquid-Liquid Extraction), and for soil samples Method 3540 (Soxhlet Extraction). However, any sample extraction technique which meets the quality assurance requirements specified in Section 10 and Table 1 of this method may be used.

9.1.1 Water extraction - Separatory Funnel.

9.1.1.1 Measure a 1 L portion of the sample and transfer to a 2 L separatory funnel. If the sample is in a 1 L or smaller bottle, mark the water meniscus on the side of the sample bottle. Measure the exact volume by adding tap water to the bottle to the marked level, and then transferring the volume of tap water to a 1 L graduated cylinder. If the sample is in a larger bottle, use a 1 L graduated cylinder to measure the volume of the sample. Use no more than 1 L of sample per 2 L separatory funnel. For blanks and quality control standards, pour 1 L of reagent water (7.1) into the separatory funnel.

9.1.1.2 Check and note the pH of the sample. If the field samples have been preserved with HCl, it is recommended that the quality control samples and blanks be preserved in the same way.

9.1.1.3 Add 1 mL of surrogate standard (7.4.2, recommended level of 20 ug/mL if o-terphenyl is used).

- 9.1.1.4 For every batch or 20 samples extracted (whichever is more frequent), prepare duplicate LFBs by adding 1 mL of 5000 ug/mL CVS (7.4.5) to each of two 1 L volumes of reagent water. Daily or for every 20 samples (whichever is more frequent), prepare a method blank using 1 L of reagent water. Surrogate must be added to both the LFBs and the method blank.
- 9.1.1.5 For samples, add 60 mL methylene chloride to the sample bottle to rinse the inner walls after the sample has been transferred to the separatory funnel. **Do Not** cap and shake the bottle, rinse the glass only; then transfer the solvent to the separatory funnel. Extract the sample by shaking it for no less than two minutes with frequent ventilation.
- 9.1.1.6 Allow the layers to separate (approx 10 minutes rest after shaking). If there is an emulsion, break it. If the emulsion cannot be broken (recovery of <80% of the methylene chloride, corrected for water solubility of methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in SW846 Method 3520 (Continuous Liquid-Liquid Extraction). Alternative physical techniques for breaking up emulsions may be acceptable.
- 9.1.1.7 Drain the bottom layer (methylene chloride) into a 250 mL graduated cylinder or other calibrated glassware.
- 9.1.1.8 Repeat the extraction twice more, using a 60 mL aliquot of methylene chloride each time. Collect the solvent in the same graduated cylinder (or equivalent) as described in 9.1.1.7. Record the volume recovered (recommended) and other prep information as an indication of extraction efficiency.
- 9.1.1.9 Put a plug of glass wool in a glass or Teflon funnel and fill about 2/3 full with anhydrous sodium sulfate. Rinse the funnel and sodium sulfate with 30-40 mL of methylene chloride, discard rinsate. Pour the extract through the rinsed sodium sulfate into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. Rinse the graduated cylinder, then the sodium sulfate, with small amounts of methylene chloride. Add these rinses to the K-D.
- 9.1.1.10 Add a few boiling chips (6.2) to the K-D and attach a 3-ball Snyder to the top. Pre-wet the column by adding about 1 mL of methylene chloride to the inverted column before attaching it to the K-D.

Note: The concentration step is critical; losses of target compounds can occur if care is not taken.

- 9.1.1.11 Place the K-D in a heated water bath set at 95°C so that the receiver tube

is immersed in hot water and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume is less than or equal to 10 mL, remove the K-D from the bath and allow it to cool completely.

Note: The extraction and concentration steps must be performed under a hood. Not only is the methylene chloride a potential health hazard (see MSDS), *if the heated water bath is not properly temperature-controlled, the concentration apparatus can explode.*

- 9.1.1.12 After the K-D has cooled, rinse the Snyder column and middle flask with a small amount of methylene chloride. Transfer the extract to a calibrated 15 mL centrifuge tube, rinsing with a small amount of methylene chloride. Rinse all of the ground glass joints well, as compounds collect on the ground glass.
- 9.1.1.13 If further concentration is desired, carefully concentrate the extract to no less than 1.0 mL under a gentle stream of nitrogen using the evaporation apparatus. If the extract is highly colored, forms a precipitate, or stops evaporating, the final volume should be higher (5-10 mL). Transfer to a labeled vial of appropriate size with Teflon-lined cap, mark the meniscus. Extracts should be stored in a non-frost free freezer.
- 9.1.1.14 Record information for the extraction and concentration steps.
- 9.1.1.15 Mount the continuous extractor on appropriate racks.
- 9.1.1.16 Put 250 mL methylene chloride in a round bottom flask, add a few boiling chips. Add 300 mL of methylene chloride to the extractor flask.
- 9.1.1.17 Check and note the initial pH of the sample. Prepare surrogate and laboratory control standards as in 9.1.2.3 and 9.1.2.4.
- 9.1.1.18 When introducing water into the extractor, minimize disturbance of the solvent layer and avoid getting water into either sidearm by carefully pouring the water down the back of the extractor.
- 9.1.1.19 For samples in 1 L or smaller bottles, mark the meniscus on the side of the sample bottle and pour approximately 1 L of the sample into the extractor flask. Measure the exact volume by adding tap water to the bottle to the marked level and measuring the volume with a graduated cylinder. For samples in larger bottles, measure 1 L of the sample in a graduated cylinder. Record the volume of sample used for extraction.
- 9.1.1.20 If necessary, add enough carbon free water to the extraction flask to allow the solvent in the removable side arm to just begin to drip into the

round bottom flask. Record the total volume of carbon free water that was added.

- 9.1.1.21 Remove the condenser from the rack and wipe the lower joint and lip with a tissue soaked with solvent. Place the condenser on top of the extractor. Turn on the cool water supply and check the flow indicators.
- 9.1.1.22 Turn on the heating mantle and record the starting time. Check after 15 minutes to be sure that the solvent in the round bottom flask is boiling, that solvent is dripping from the lip on the condenser, and that the volume of the solvent in the round bottom flask is still about 240 mL.
- 9.1.1.23 Check all extractor joints for leaks with a Kimwipe. Allow the extraction to proceed for 18-24 hours. Surrogate recovery from blanks can be used as an indicator of extraction efficiency, and extraction times can be adjusted to optimize results.
- 9.1.1.24 Turn off the heating mantle and allow the apparatus to cool (30-60 minutes) with water flowing through the condenser.
- 9.1.1.25 The solvent contained in the round bottom flask is the extract. Transfer the extract to a 400 mL glass graduated cylinder, rinsing with a small amount of Methylene chloride. If the volume of solvent is less than about 250 mL, record it.
- 9.1.1.26 Go to 9.1.2.9 and proceed with the prep, through the nitrogen concentration.

9.1.2 Soil Preparation - Soxhlet Extraction

- 9.1.2.1 Decant any water layer that may accompany the solid layer in the sample. Note what percent of the sample the water represents and, if sufficient volume exists, extract and analyze the water for DRO. Also note the apparent condition of the sample (presence of foreign materials, variable particle size, presence of oil sheen, multiple phases, etc).
- 9.1.2.2 Weigh 10 g to 30 g of the original sample into an extraction thimble. Add an equal weight of anhydrous sodium sulfate and stir the mixture well with a wooden tongue depressor. The sample should have a grainy texture - if the sample clumps, add more sodium sulfate until a grainy texture is achieved and note the addition. (Do this for all samples and standards.)
- 9.1.2.3 Dry the sample in a hood or warm (no greater than $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$) oven overnight. If necessary, cool in a desiccator until the sample reaches room temperature and reweigh to the nearest 0.001g for the moisture determination.

$$\% \text{ Moisture} = (A-C)/(A-B) \times 100$$

Where:

A= weight of boat + wet sample

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.

9.1.2.4 Place loaded thimbles in extractors and add surrogate to both field and quality control samples.

9.1.2.5 Add CVS to the duplicate LFBs. These quality control samples should contain 10 g of methylene chloride rinsed Ottawa Sand or alternative standard soil. In addition, prepare a method blank.

9.1.2.6 Add 300 mL of methylene chloride to the 500 mL extraction flask. Less extraction solvent may be used if the quality control criteria specified in Section 10 and Table 1 are met. Also add a few methylene chloride washed carborundum boiling chips to the flask. Connect the extractor to the flask and the condenser to the extractor. Allow samples to extract for 18-24 hours, or as long as necessary to achieve optimum surrogate recovery. Be sure that coolant is flowing around the condensers.

9.1.2.7 Recommendation: After extraction, disassemble extractor and add about 3 g anhydrous sodium sulfate to the extract and allow to incubate for 2 hours. (This assures that the extract is water-free before concentration.)

9.1.2.8 Transfer extract into a clean 500 mL K-D and proceed from 9.1.1.9.

9.1.3 Moisture Determination for Solids

9.1.3.1 Moisture determinations must accompany all soils data (reported in mg/dry kg) so the client can, at will, determine the results in the original soil condition. Reporting in mg/dry kg can best be done if an unpreserved portion of the sample (collected without methanol) is provided. Because of the potential for high petroleum compound concentrations in the soil, all drying should be done under a functioning hood.

9.1.3.2 To determine percentage of moisture, pre-weigh an aluminum weighing boat. Weigh 5-10 g of the sample into the boat and record both weights to the nearest 0.001 g. Dry the sample overnight in a warm (100±5°C)

oven.

- 9.1.3.3 Remove the sample from the oven and cool in a desiccator until the sample reaches room temperature, and weigh to the nearest 0.001g. Record the weight.
- 9.1.3.4 Return the soil sample to the oven for an additional time period (not less than 2 hours), cool again in the desiccator until the sample reaches room temperature, and weigh to the nearest 0.001g.
- 9.1.3.5 If the weight of the sample has remained constant ($\pm 4\%$) from the initial "dry" weight (9.1.3.4), use this number for the moisture determination (see 9.6.2). If the second weighing shows that the sample has lost further weight, continue drying and weighing the sample until the weight becomes constant, then proceed to 9.6.2.
- 9.1.3.6 If a sample contains a high concentration of petroleum product, constant weight may be difficult to attain. If, after several tries, the $\pm 4\%$ criteria cannot be reached an estimated % moisture may be reported with appropriate explanation.

9.1.4 Dilution Technique

- 9.1.4.1 This is used for product or waste samples for which extraction is not appropriate and which are soluble in methylene chloride.
- 9.1.4.2 Weigh 1 g of sample into a 10 mL volumetric flask. Dilute to 10 mL with methylene chloride. Transfer to a 12 mL vial with a Teflon lined lid. Mark meniscus and store at $4 (\pm 2) ^\circ\text{C}$, or -10 to $-20 ^\circ\text{C}$.

9.2 Gas Chromatography

9.2.1 Conditions (Recommended):

Set helium column pressure to 20#. Set column temperature to 40°C for 2 minutes, then ramp at a rate of $12^\circ\text{C}/\text{min}$ to 320°C and hold for 15 min. (run time = 36 minutes). Set FID Detector to 320°C and injector to 280°C . The reference book High Resolution Chromatography by Hewlett-Packard is a good source of information on how to optimize flow rates, etc.

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

- 9.2.2.1 Resolution of the methylene chloride solvent front from C_{10} .

- 9.2.2.2 The separation number, TZ, should be greater than 15 for C₂₄ and C₂₅, if RRO is to be analyzed concomitantly.

$$TZ = \frac{\text{retention time } C_{25} - \text{retention time } C_{24}}{W_{1/2} \text{ of } C_{25} + W_{1/2} \text{ of } C_{24}} - 1$$

Where "W_{1/2}" = peak width at half-height

- 9.2.2.3 The column must be capable of separating typical diesel components from the surrogate and internal standards. In particular, there are potential problems with the resolution of n-C₁₉/ortho-terphenyl and n-C₂₁/5 alpha-androstane at varying relative concentrations.

9.3 Calibration

- 9.3.1 Calibrate the GC, set up as in 9.2, with an initial five point (recommended) calibration using DCS (7.4.3). The final calibration curve must be represented by no less than 3 concentrations of DCS.
- 9.3.2 Choose DCS concentrations to cover the DRO range expected in the samples, or the linear range of the instrument, whichever is less. One of the concentrations must be at or near the practical quantitation limit.
- 9.3.3 Tabulate the area response of the calibration standard against mass injected. The ratio of the response to the amount injected, defined as the response factor (RF), can be calculated for the standard at each concentration. If the average percent relative standard deviation (%RSD) is less than 25% over the working range, linearity through the origin can be assumed, and the continuing calibration response factor can be used in place of a calibration curve.

$$\text{Internal Standard Response Factor} = \frac{(Ax)(Cis)}{(Cx)(Ais)}$$

Where: Ax = Peak area of analyte
 Ais = Peak area of internal standard
 Cis = Concentration of internal standard
 Cx = Concentration of analyte

Alternately, external standard calibration may be used (See Method 8000 [1]).

Then,

$$\text{External Standard Response Factor} = \frac{\text{Total peak area of standard}}{\text{Mass injected}}$$

- 9.3.4 The calibration curve must be confirmed using the CVS (7.4.5). This standard verifies the accuracy of the calibration. The concentration of the CVS should be within the expected concentration range of the samples to be analyzed.
- 9.3.5 The working RF or calibration curve must be verified on each working day (24 hours) by the injection of aCCS (7.4.3) at a concentration mid-point on the calibration curve. The CCS is a diluted aliquot of the same standard used to initially calibrate the instrument. If the response for the CCS varies from the predicted response by more than 25%, a new calibration curve must be prepared.

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_1} \times 100$$

where:

R1 = Average RF from the calibration curve

R2 = Response Factor from CCS

9.4 Retention Time Window Definition:

- 9.4.1 Before establishing windows, be certain that the GC system is within optimum operating conditions (6.6). Make three injections of the Retention Time Window Standard (7.4.3) throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.
- 9.4.2 Calculate the standard deviation of the three absolute retention times for decane and pentacosane and the surrogate.
- 9.4.2.1 The retention time window for individual peaks is defined as plus or minus three times the standard deviation of the absolute retention time for each component.
- 9.4.2.2 In those cases where the standard deviation for a particular analyte is zero, the laboratory should use ± 0.05 min. as a retention time window.
- 9.4.3 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed or instrument conditions changed. The data must be retained by the laboratory for at least a year.
- 9.4.4 Retention time windows must be verified regularly and updated no less frequently than once a year.

9.5 Gas Chromatograph Analysis

- 9.5.1 Samples are analyzed by GC/FID. Optimum injection volumes (2 uL using the conditions established in 9.2) must be established for specific instrument

conditions.

- 9.5.2 For internal standard calibration, the internal standard is spiked into each sample and standard at a concentration of 200 ug/mL of sample extract. Twenty uL of 5-alpha androstane stock at 1000 ug/mL may be spiked into the 1 mL final volume or a corresponding amount may be added to an aliquot of the final extract. (Note: DRO values >2000 ug/mL may lead to measurement bias due to coelution with the internal standard.)
- 9.5.3 If initial calibration (9.3) has been performed, verify the calibration by analysis of a mid-point CCS. With each day's run, open a 24 hour analysis window. This is done by running the Retention Time Window Standard (7.4.4).
- 9.5.4 Calculate the percent difference of the response factor from the mean response factor as in 9.3.2. This is done for DRO as a group from the CCS. If the response factor has a percent difference greater than 25%, corrective action must be taken.
- 9.5.5 A methylene chloride blank must be 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 methylene chloride blank is integrated over the DRO area in the same manner as for the field samples and is reported as the solvent blank. (Refer to reference 4.) **Do not baseline subtract. This information is for data interpretation purposes only.**
- 9.5.6 Blanks should also be run after samples suspected of being highly concentrated to prevent carryover. If the blank analysis shows contamination above the practical quantitation limit, the column must be baked out and subsequent blanks analyzed until the system is shown to retain contaminant at concentrations less than the PQL.
- 9.5.7 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 response of the major peaks should be kept in the upper half of the linear range of the calibration curve. Due to potential measurement bias, internal standard calibration should not be used when DRO exceeds 5000 ug/mL in the final extract (ref. 9.5.2). The sample should be diluted or external standard calibration should be used.

9.6 Calculations:

- 9.6.1 Internal Standard Calibration: The concentration of DRO in the sample must be determined by calculating the absolute weight of analyte chromatographed from a

summation of peak response for all chromatographic peaks eluting between the peak start of n-decane and the peak start of n-pentacosane, using the calibration curve or the response factor determined in section 9.3. Also refer to Section 9.4 (Retention Time Window Definition).

The concentration of DRO is calculated as follows:

Aqueous/Soil samples:

$$C_s = \frac{(A_x)(C_{is})(D)(V_t)}{(A_{is})(RF)(V_s)}$$

Where:

C_s = Concentration of DRO (mg/L or mg/kg).

A_x = Response for the DRO in the sample, units in area.

RF = Response Factor from CCS (see 9.3.1).

A_{is} = Response for the internal standard, units same as for A_x .

C_{is} = Internal standard concentration (mg/mL).

V_t = Volume of final extract in mL.

D = Dilution factor, if dilution was performed on the sample prior to analysis. If no dilution was made, $D = 1$, dimensionless.

V_s = Amount of sample extracted in L or kg.

9.6.2 To calculate mg/dry kg for soil samples,

$$\text{mg/dry kg DRO} = \frac{C_s}{1 - (\% \text{ moisture}/100)}$$

The % moisture calculation must be included in the data package (see 9.1.4).

9.6.3 External Standard Calibration:

Aqueous/Soil samples:

$$C_s = \frac{(A_x)(A)(V_t)(D)}{(A_s)(V_s)}$$

Where:

C_s = Concentration of DRO (mg/L or mg/kg).

A_x = Response for the DRO in the sample, units in area.

A_s = Response for the external standard, units same as for A_x .

A = External standard concentration (mg/mL).

V_t = Volume of Final extract in mL.

D = Dilution factor, if dilution was performed on the sample prior to analysis. If no dilution was made, $D = 1$, dimensionless.

V_s = Amount of sample extracted in L or kg.

9.6.4 Some software programs are capable of performing 9.6.1 and 9.6.3 with minimal analyst intervention. Additionally, some software programs can "update" a calibration curve based on the response of the CCS. If a calibration curve is updated in this manner, a valid CVS must be analyzed and results must fall within the Quality Control Criteria specified in Section 10 and Table 1 before field samples can be analyzed.

10. Quality Control (See Table 1 of this method).

10.1 The laboratory must establish and maintain the ability to generate acceptable accuracy and precision and to demonstrate through the analysis of quality control check standards that the operation of the measurement system is in control. This must include the analysis of QC check samples plus the calculation of average recovery and the standard deviation of recovery as outlined in method 8000, section 8.0 [1], and in this method.

10.2 After successful calibration (Section 9.8), analyze the reagent blank. The reagent blank must be analyzed with every extraction batch. The surrogate recovery must be within established limits (see Method AK 102.0, Table 1), or within the limits established by the project plan (whichever is more stringent) and the control sample must not have DROs above the practical quantitation limit.

10.3 With every batch or 20 samples, duplicate LFBs must be analyzed (reagent water or Ottawa sand matrix, as is appropriate to the samples being analyzed). The accuracy and precision of the duplicate standards must be within established limits (Table 1). Also, the mid-point CCS must be analyzed at the end of each sequence and once per 20 samples, and compared to the successful calibration as described in 9.8.6, and fall within established limits (Table 1).

10.4 Every batch of samples extracted must be accompanied by a method blank to demonstrate that samples are free from method interference. The method blank must have DROs less than the PQL.

10.5 Each laboratory should generate control limits based on the average recovery, with ± 2 standard deviations as a warning limit and ± 3 standard deviations the action limit.

10.6 If any of the criteria in 9.3, 10.2, 10.3 and 10.4 are not met, corrective action must be taken before samples are analyzed.

10.7 Calculate the surrogate standard recovery in each sample. If recoveries are outside established limits, (Table 1), verify calculations, dilutions, and standard solutions. Verify instrument performance.

10.7.1 High recoveries may be due to a coeluting matrix interference or the presence of high molecular weight contaminants; examine the sample chromatogram.

- 10.7.2 High recoveries may also be due to memory effects caused by poor sample volatility, backflash, or carryover; check instrument conditions, injection volume, and injector temperature.
- 10.7.3 Low recoveries may be due to adsorption by the sample matrix (muskeg, tundra, forest loam, etc).
- 10.7.4 Low recoveries may also be caused by incorrect integration. The chromatographic profile of DROs may not give baseline resolution of all components, resulting in a characteristic rise in baseline underneath the resolved hydrocarbon components. Do not use peak-to-peak integration, use forced baseline integration.
- 10.7.5 If internal calibration has been used, DRO results must be normalized using the internal standard response. If surrogate recovery is still outside of established limits, corrective action must be taken.
- 10.7.6 If external calibration has been used, and surrogate recovery is outside of established limits, offered corrective action must be taken.
- 10.8 If field samples show low surrogate recovery due to suspected matrix effects, DRO results must be flagged. If the surrogate recovery is <50% and the calculated DRO concentration falls within a factor of 2 of the action level, the laboratory should recommend that the client resubmit the sample for matrix spike/matrix spike duplicate analysis. (To perform matrix spike analyses, follow 9.1, except use a field sample instead of a standard matrix.) This is a recommendation, not a requirement of the method, and therefore, the onus is not on the analytical laboratory to absorb the cost of the additional analyses.
- 10.9 Matrix spikes are recommended for specific sampling programs. Field blanks, trip blanks, field duplicates are required as stated in Chapter 2, section 9 of the UST Procedures Manual.
- 10.10 Minimum quality control acceptance criteria are set forth in this section. More stringent quality control criteria may be required by specific project plans.
- 10.11 Corrective Action
 - 10.11.1 Calibration
 - 10.11.1.1 If the initial calibration does not meet the criteria set forth in 9.3.3 and 9.3.4, the instrument must be recalibrated.
 - 10.11.1.2 If the continuing calibration does not meet the criteria set forth in 9.3.5 and Table 1, the instrument must be recalibrated.

10.11.2 Surrogates

10.9.2.1 If surrogates are outside established control limits (Table 1), the following assessments and/or correction actions must occur:

A) Check to be sure there are no errors in calculations and that the concentration of the surrogate and internal standard solution are correct.

B) Check instrument performance to determine if it is within acceptable guidelines.

C) Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

D) Reprepare and reanalyze the sample if none of the above resolves the problem.

10.11.2.2 If the surrogate recoveries that are outside the control limits cannot be attributed to lab error, the decision to reanalyze or flag the data should be made in consultation with the client. Provided all other QC acceptance criteria are met (section 10), it is only necessary to reprepare/reanalyze a sample one time to demonstrate that a poor surrogate recovery is due to matrix effects. A relationship can be established between surrogate recovery and moisture content of organic soils, which may help in diagnosing the cause of poor surrogate recoveries.

10.11.3 Blanks: Additional blanks may be necessary for certain projects to meet the goals of Chapter 2, section 9 of the UST Procedures Manual.

10.11.3.1 Instrument Blanks:

Instruments must be evaluated with each batch (or daily, whichever is more frequent) and must demonstrate that the analytical system is free from contamination. This is best accomplished by analyzing an Instrument Blank.

10.11.3.2 Method Blank:

Method Blanks must be analyzed with each extraction batch IF the results of the field samples show contamination above the MCL. The Method Blank for AK102 can also serve as the Reagent Blank if DRO is less than the PQL.

10.11.3.3 Field Blank:

If the field samples yield DROs above the MCL, and contamination is

found below the PQL in the Reagent Blank, a Field Blank should be analyzed to identify whether the source of contamination originated in the field sample collection procedure, during trip or during storage in the laboratory.

(Note: Blanks are reported by value. DO NOT BLANK SUBTRACT. This information is for data quality assessment purposes only.)

10.11.4 Laboratory Fortified Blanks

10.11.4.1 If the analyte recovery from the LFBs is outside the established recovery limits (Table 1), the following assessments and/or corrective actions must occur:

A) Check to be sure there are no errors in calculations and that the concentration of the analyte solution is correct.

B) Check instrument performance to determine if it is within acceptable guidelines.

C) Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

D) Reprep and reanalyze the samples if none of the above resolves the problem.

10.11.4.2 If the relative percent difference between the LFB results exceeds the control limits, but meets the percent recovery criteria (Table 1), the following assessments and/or corrective actions must occur:

A) Check to be sure that there are no errors in calculations, and that the same amount and source of analyte solution, solvent and water were used for both samples in the set.

B) Check to determine if instrument performance is still within acceptable guidelines, and that conditions did not change during the course of the batch analysis.

C) Recalculate the data if calculation error is suspected.

D) Repeat the LFB duplicate extraction and analysis, along with a representative number of samples (10% of the samples from the batch OR 1 sample, whichever is more) from the analytical batch with the failed LFB RPD. The re-analysis of the field samples is to demonstrate comparability of the extraction/analysis conditions at the time of re-extraction and analysis to those at the time of the failed QC.

11. Method Performance

11.1 Single lab method performance data for the DROs method in Ottawa sand and other soil types is presented below.

11.2 Results for diesel spikes (methylene chloride extraction direct injection, soils)

<u>Matrix</u>	<u>Diesel Spike Amount</u> <u>mg/kg</u>	<u>Percent</u> <u>Recovery</u>
Ottawa Sand	70	97
Ottawa Sand	70	98
Glacial Blue Clay	70	70
Glacial Blue Clay	70	76
Forest Loam	70	136
Forest Loam	70	163
River Sediment	70	142
River Sediment	70	167
Marine Sand	70	95
Marine Sand	70	88

Notes: Analyses performed by State of Alaska, DEC Laboratory. Diesel used = DCS.
All highly organic soil matrices showed high analyte recovery due to naturally occurring DROs.

11.3 The method detection limit for soil calculated according to 40 C.F.R., Part 136, Appendix B (1994) was 1.6 mg/kg (external standard calibration, Ottawa sand).

12. References

1. USEPA "SW-846 Test Methods for Evaluating Solid Waste", 3rd Edition; Methods 8000, 8100, 3510, 3520, 3540, and 3550.

2. "Method OA-2: Extractable Petroleum in Products," Revision January 10, 1990", University Hygienic Laboratory, Iowa City, Iowa.

3. "Method for Determination of Extractable Petroleum Hydrocarbons (EPH) in Soil and Water," Draft-February 28, 1990, prepared for Total Petroleum Hydrocarbons Method Committee by Midwest Research Institute.

4. Zilis, K., M. McDevitt, and J. Parr, "A Reliable Technique for Measuring Petroleum Hydrocarbons in the Environment," presented at the conference on Petroleum Hydrocarbons and Organic Chemicals in Groundwater, NWWA, Houston, Texas, November 1988.

5. American Petroleum Institute "Method for the Determination of DROs," Draft Revision 2-February 5, 1992, prepared for Total Petroleum Hydrocarbons Method Committee.

6. "Leaking Underground Fuel Tank (LUFT) Field Manual," State Water Resources Control

Board, State of California, Sacramento, CA, May 1988.

7. Fitzgerald, John, "Onsite Analytical Screening of Gasoline Contaminated Media Using a Jar Headspace Procedure" in Petroleum Contaminated Soils, Vol. 2, 1989.
8. Senn, R.B., and M.S. Johnson, "Interpretation of Gas Chromatographic Data in Subsurface Hydrocarbon Investigations," Ground Water Monitoring Review, 1987.
9. Hughes, B.M., and D.E. McKenzie, C.K. Trang, L.S.R. Minor, "Examples of the Use of an Advanced Mass Spectrometric Data Processing Environment for the Determination of Sources of Wastes" presented at 5th Annual Waste Testing and Quality Assurance Symposium, July 1989.
10. ASTM "Standard Methods for Comparison of Waterborne Petroleum Oils by Gas Chromatography," pp. 3328-78.
11. API consensus "Method for the Determination of Diesel Range Organics," Revision 2, 2/5/92.
12. Research done by the State of Alaska, Department of Environmental Conservation, Division of Environmental Quality, Juneau Environmental Analysis Laboratory.

Method AK 102.0, Table 1
ACCEPTANCE CRITERIA FOR QUALITY CONTROL

<u>ANALYTE</u>	<u>SPIKE CONCENTRATION</u>		<u>CONTROL LIMITS</u>	
	<u>Water (mg/L)</u>	<u>Soil (mg/kg)</u>	<u>% Recovery</u>	<u>Relative % Difference</u>
Lab Fortified Blanks				
Diesel Range Organics	0.5	20	60 - 120	20
Continuing Calibration/ Calibration Verification				
Diesel Range Organics			75 - 125	
Laboratory Sample Surrogate Recovery				
Ortho-terphenyl	0.02	0.8	60 - 120	
Field Sample Surrogate Recovery				
Ortho-terphenyl	0.02	0.8	50 - 150	

Method AK 103.0
For Determination of Residual Range Organics
Revision 2.0, 1/31/96

1. Scope and Application

1.1 Objectives

- 1.1.1 This method is designed to measure the concentration of Residual Range Organics (RRO) in soil. This corresponds to an n-alkane range from the beginning of C_{25} to the end of C_{36} , and a boiling point range of approximately 400° C to 500° C.
- 1.1.2 The method is primarily designed to measure lubricating or motor oils or other heavy petroleum products. Components greater than C_{36} present in products such as asphalts, and mid-range petroleum products such as diesel and bunker C, are also detectable under the conditions of the method.
- 1.1.3 This method can be an extension of the Method for Determination of Diesel Range Organics as specified in AK102. All quality assurance requirements of both methods (Section 10) must be met. Reasonable modification in order to accommodate the concurrent analysis of DRO and RRO is within the scope of this method.

1.2 Quantitation Limits: The practical quantitation limit (PQL) for this method of analysis of RROs is based on studies done by laboratories other than the State of Alaska, Department of Environmental Conservation, State Chemistry Laboratory and is approximately 100 mg/kg for soils using motor oil as a standard.

1.3 Dynamic Range: Dilutions should be performed as necessary to put the chromatographic envelope within the linear range of the method. Linear range is dependent in part upon column type, detector sensitivity, and injection volume. Typically, the approximate range is 10 mg/L to 200 mg/L.

1.4 Experience: This method is based on a solvent extraction, gas chromatography (GC) procedure. This method should be used by, or under the supervision of, analysts experienced in the use of solvent extractions and gas chromatographs and skilled in interpreting gas chromatograms and their use as a quantitative tool.

2. Method Summary

2.1 This method provides gas chromatographic conditions for the detection of high molecular weight semi-volatile petroleum products such as motor oils. Other non-petroleum

products, with similar characteristics and boiling points, may also be detected with this method. The sample is spiked with a surrogate compound and extracted with methylene chloride. The extract is dried and concentrated to a known volume. A portion of the dried, concentrated extract is injected into a capillary column gas chromatograph equipped with a flame ionization detector (FID), which has been temperature programmed to facilitate separation of organic compounds. Quantitation must be performed by comparing the total chromatographic area between the peak start of C_{25} and the peak end of C_{36} , both resolved and unresolved components, based on FID response compared to a blended commercial standard called the Residuals Calibration Standard (see paragraph 3.2).

- 2.2 This version of the method was developed by Dr. Mary Jane Pilgrim, and is based in part on US EPA Methods 8000 and 8100, SW-846, "Test Methods for Evaluating Solid Waste," 3rd Edition [1], Method OA-2 [2], the API consensus method "Method for the Determination of Petroleum Hydrocarbons", Original version, 2/3/92 [3] and work by the EPA Total Petroleum Hydrocarbons Method Committee [4], the State of Oregon, "Total Petroleum Hydrocarbon Methods" QAR 340-122-350 dated December 11, 1990 and the State of Washington, "Hydrocarbon Identification Method" WTPH-HCID from Guidance for Remediation of Releases from Underground Storage Tanks, document 91-30 dated July 1991, and data from Alaska's State Chemistry Laboratory, with support from the Storage Tank Program .

3. Definitions

- 3.1 Residual Range Organics (RRO): All chromatographic peaks, 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 of the motor oil standard within the same (C_{25} - C_{36}) range as determined from FID response using baseline-baseline integration.
- 3.2 Residuals Calibration Standard (RCS): A blend of equal weights of 30 weight and 40 weight motor oils (1:1) and diluted to appropriate concentrations in methylene chloride. This standard serves as a calibration standard for RRO. It is recommended that the RCS components be combined with the DCS components if DRO (AK102) is to be done simultaneously.
- 3.3 Surrogate Control Standard (SCS): n-Triacontane-d62 or equivalent, used as a laboratory data quality control. Any variance from this surrogate must be approved by the ADEC Approval Authority, and a demonstration of suitability must be performed. This surrogate may be combined with the surrogate for DRO (AK102) if the methods are to be done simultaneously.
- 3.4 Surrogate Control Sample : A method blank sample spiked with surrogate . The surrogate recovery is used as a laboratory control (see Method AK 103.0, Table 1.

- 3.5 Calibration Verification Standard (CVS): A commercial motor oil blend, prepared as in 3.2 but with products from a source other than those used to prepare the RCS. It is used by the laboratory as a quality control check to verify the accuracy of the calibration.
- 3.6 Laboratory Fortified Blank (LFB): A method blank sample spiked with CVS (3.5). The spike recovery is used to evaluate method control (see Method AK 103.0, Table 2).
- 3.7 Retention Time Window Standard: A mixture of the normal alkanes n-pentacosane (C_{25}) and n-hextriacontane (C_{36}) which is analyzed once every 24 hour "day" or with each batch of samples, whichever is less frequent, not to exceed 20 samples per batch. This standard serves to define the retention time window for RRO.
- 3.8 Internal Standard: No internal standard has been used in development of this method. Any internal standard which mimics the chemical characteristics of heavy petroleum products may be used, with prior ADEC approval.
- 3.9 Standard Soil: Ottawa sand or other standard soil with characteristics which match the field samples as closely as possible, used in quality control standards.
- 3.10 Continuing Calibration Standard (CCS): A mid-range working standard diluted from the RCS (3.2), used to verify that the analytical system is operating in a manner comparable to that at the time of calibration.
- 3.11 Other terms are as defined in SW-846 [1].

4. Interferences

- 4.1 Other organic compounds including, but not limited to, animal and vegetable oil and grease, chlorinated hydrocarbons, phenols, phthalate esters, and biogenic terpenes are measurable under the conditions of this method. Some lighter petroleum products such as bunker C and diesels, as well as crude oils, may produce a response within the retention time range for RRO. As defined in the method, the RRO results include these compounds.
- 4.2 Method interferences may be reduced by washing all glassware with hot soapy water and then rinsing it with tap water, methanol, and methylene chloride. Heating the glassware to reduce contaminants should not be necessary if this cleaning method is followed. At least one blank must be analyzed with each extraction batch to demonstrate that the samples are free from method interferences.
- 4.3 High purity reagents such as Burdick and Jackson GC² methylene chloride or Baker capillary grade methylene chloride must be used to minimize interference problems.
- 4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. When an unusually concentrated sample is encountered, it should

be followed by a solvent blank to check for instrument contamination.

5. Safety Issues

- 5.1 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. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety should be available and should be identified for use by the analyst.
- 5.2 A hearing protection device should be used when performing sonication.

6. Apparatus and Materials

(Unless otherwise indicated, all apparatus and materials are suggested.)

6.1 Glassware

- 6.1.1 4 oz. amber glass wide mouth jars with Teflon-lined screw caps
- 6.1.2 250 mL glass centrifuge tubes (if using sonication extraction).
- 6.1.3 Two mL glass vials with Teflon-lined cap (autosampler vials).
- 6.1.4 Disposable pipettes: Pasteur.
- 6.1.5 Graduated cylinders: 250 mL.
- 6.1.6 Glass or Teflon funnels.
- 6.2 Boiling chips - Approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.
- 6.3 Micro syringes: One ul, 5 ul, 10 ul, 25 ul, and 100 ul or as needed.
- 6.4 Water bath - Heated with concentric ring cover, capable of temperature control (+/- 2°C). The bath should be used in a hood.

6.5 An analytical balance capable of accurately weighing 0.0001 g should be used for preparing standards and % moisture determinations. A top-loading balance capable of weighing to the nearest 0.1 g should be used for sample preparation.

6.6 Stainless steel spatula.

6.7 Gas Chromatography

6.7.1 Gas Chromatograph: Analytical system including appropriate gas supply and all required accessories, including a Flame Ionization Detector (FID), column supplies, gases, and syringes. A data system capable of determining peak areas using a forced baseline - baseline projection is required. A data system capable of storing and reintegrating chromatographic data is recommended.

6.7.2 Columns

6.7.2.1 Column 1: 5 M x 0.53 mm SGE HT-5, 0.1 micron film thickness.

6.7.2.2

Alternate columns: 30 M x 0.32 mm ID J&W DB-1 or DB-5, 0.25 micron film thickness.

6.7.2.3

Other Columns may be used - capillary columns may be required to achieve the necessary resolution. The column must resolve C_{25} from C_{24} in a midrange RCS or CVS if AK102 is to be done simultaneously. See 9.2.2 for additional column performance criteria.

6.8 Sonication

6.8.1 Ultrasonic cell disrupter: A horn-type sonicator equipped with a titanium tip should be used. A Heat Systems-Ultrasonics, Inc. Model W-385 (475 watt) sonicator or equivalent (power wattage must be a minimum of 375 with pulsing capability and No. 200 1/2 inch Tapped Disrupter Horn) plus No. 207 3/4 inch Tapped Disrupter Horn, and No. 419 1/8 inch Standard tapered Microtip probe.

6.8.2 A Sonabox or equivalent is recommended with the above disrupter for decreasing sound (Heat Systems-Ultrasonics, Inc. Model 432 13 or equivalent).

6.9 Soxhlet extraction apparatus as described in SW-846 Method 3540 [1].

6.10 Nitrogen evaporator with high purity (grade 4.5 or equivalent) nitrogen gas source.

7. Reagents and Standards

7.1 Reagent Water: Water that has been shown to be free from RRO compounds - a

Millipore system or equivalent is recommended.

- 7.2 Methylene Chloride, Hexane, Acetone - pesticide grade or equivalent. At a minimum, the solvents must be shown to be free from RRO.
- 7.3 Sodium Sulfate - (American Chemical Society (ACS) grade) granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray, or by extracting three times with methylene chloride and drying at 100±5° C. Incomplete cleaning of sodium sulfate can result in RRO contamination.
- 7.4 Stock Standard Solutions - Prepare the following stock standards. Unless noted, all are prepared in the methylene chloride listed in 7.2 above. Standards preparation should follow guidelines in SW846 [1]. All standards prepared by the laboratory must be stored without headspace at -10 to -20° C and protected from light. Marking of the meniscus is helpful in maintaining stock standard integrity. Standards must be replaced within 6 months of preparation. Standards should be checked regularly to assure their integrity. Standards which are purchased pre-made from commercial suppliers may be kept for the life, and under the conditions, specified by the manufacturer if different than described in this paragraph.
 - 7.4.1 Recommended Surrogate: 5000 ug/mL n-Triacontane-d62 (dTC). A working solution is made at 500 ug/mL (recommended concentration) in acetone.
 - 7.4.2 Residuals Calibration Standard (RCS): A blend of equal weights of motor oil, mixed together to form a composite motor oil (1:1, 30 weight: 40 weight :) is used to prepare stock calibration standards in methylene chloride. No fewer than 3 concentrations of this Residuals Calibration Standard are used for instrument calibration. A five point calibration curve is recommended. Other than one standard concentration near the practical quantitation limit, the expected range of concentrations found in project samples should define the working range of the calibration.
 - 7.4.3 Retention Time Window Standard : A stock solution of C₂₅ and C₃₆ n-alkanes with each component at a level of at least 10,000 ug/mL (recommended). This blend of alkanes serves as a retention time window defining mix for RRO.
 - 7.4.4 Stock CVS: From a blend of commercial motor oils other than those used to prepare the RCS, make an equal weight mixture as described above (7.4.2). A working solution is made at a recommended concentration of 5,000 ug/mL in acetone.

8. Sample Collection, Preservation, Containers, and Holding Times

- 8.1 Soils are collected in a core tube or 4 or 8 oz amber glass jar with Teflon lined lid. The samples are stored at $4 \pm 2^{\circ}$ C from the time of collection until extraction. Extraction must be performed on soils within 14 days.[1]. All analyses of extracts must take place within 40 days.
- 8.2 Soil samples to be analyzed for volatiles, DRO and RRO may be collected in the same, methanol preserved container and stored as for GRO (AK101). If this option is selected, the mechanics of the collection, preservation and container should be discussed with the client before sampling kit preparation. RRO extraction and analysis must still meet the requirements of 8.1, above.

9. Procedure

- 9.1 Sample Preparation: The preferred procedure for extraction is Method 3540 (Soxhlet Extraction). However, any sample extraction technique which meets the quality assurance requirements specified in Section 10 and Table 1 of this method may be used.

9.1.1 Soil Preparation - Soxhlet Extraction

- 9.1.1.1 Decant any water layered on the sample. Refer to method AK102, section 9.1.2.1 if DRO is to be done simultaneously. Mix the sample well and note any foreign objects or anomalies (variably particle size, presence of oil sheen, multiple phases, etc.).
- 9.1.1.2 Weigh 10 g to 30 g of the original sample into an extraction thimble. Add an equal weight of anhydrous sodium sulfate and stir the mixture well with a wooden tongue depressor, taking care to not rupture the thimble. The sample should have a grainy texture - if the sample clumps, add more sodium sulfate until a grainy texture is achieved and note the addition. (Do this for all samples and standards.)
- 9.1.1.3 Place loaded thimbles in extractors and add surrogate to all samples, both field and quality control.
- 9.1.1.4 Add CVS to the duplicate LFBs. These standards should contain 10 g of methylene chloride rinsed standard soil. In addition, prepare a method blank
- 9.1.1.5 Add 300 mL of methylene chloride to the 500 mL extraction flask. Less extraction solvent may be used if the quality control criteria specified in Section 10 and Table 1 are met. Also add a few methylene chloride washed carborundum boiling chips to the flask. Connect the extractor to the flask and the condenser to the extractor. Allow samples to extract for 18-24 hours, or as long as necessary to achieve optimum surrogate

recovery. Be sure that coolant is flowing around the condensers.

9.1.1.6 Recommendation: After extraction, disassemble extractor and add about 3 g anhydrous sodium sulfate to the extract and allow to incubate for 2 hours. (This assures that the extract is water-free before concentration.)

9.1.1.7 Put a plug of glass wool in a glass or Teflon funnel and fill about 2/3 full with anhydrous sodium sulfate. Rinse the funnel and sodium sulfate with 30-40 mL of methylene chloride, discard rinsate. Pour the extract through the rinsed sodium sulfate into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. Rinse the graduated cylinder, then the sodium sulfate, with small amounts of methylene chloride. Add these rinses to the K-D.

9.1.1.8 Add a few boiling chips (6.2) to the K-D and attach a 3-ball Snyder to the top. Pre-wet the column by adding about 1 mL of methylene chloride to the inverted column before attaching it to the K-D.

Note: The concentration step is critical; losses of target compounds can occur if care is not taken.

9.1.1.9 Place the K-D in a heated water bath set at 95°C so that the receiver tube is immersed in hot water and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume is less than or equal to 10 mL, remove the K-D from the bath and allow it to cool completely.

Note: The extraction and concentration steps must be performed under a hood. Not only is the methylene chloride a potential health hazard (see MSDS), *if the heated water bath is not properly temperature-controlled, the concentration apparatus can explode.*

9.1.1.10 After the K-D has cooled, rinse the Snyder column and middle flask with a small amount of methylene chloride. Transfer the extract to a calibrated 15 mL centrifuge tube, rinsing with a small amount of methylene chloride. Rinse all of the ground glass joints well, as compounds collect on the ground glass.

9.1.1.11 If further concentration is desired, carefully concentrate the extract to no less than 1.0 mL under a gentle stream of nitrogen using the evaporation apparatus. If the extract is highly colored, forms a precipitate, or stops evaporating, the final volume should be higher (5-10 mL). Transfer to a labeled vial of appropriate size with Teflon-lined cap, mark the meniscus. Extracts should be stored in a non-frost free freezer.

9.1.1.12 Record information for the extraction and concentration steps.

9.1.2 Moisture Determination for Solids

9.1.2.1 Moisture determinations must accompany all soils data (reported in mg/dry kg) so the client can, at will, determine the results in the original soil condition. Reporting in mg/dry kg can best be done if an unpreserved portion of the sample (collected without methanol) is provided. Because of the potential for high petroleum compound concentrations in the soil, all drying should be done under a functioning hood.

9.1.2.2 To determine percentage of moisture, pre-weigh an aluminum weighing boat. Weigh 5-10 g of the sample into the boat and record both weights to the nearest 0.001 g. Dry the sample overnight in a warm ($100 \pm 5^\circ\text{C}$) oven.

9.1.2.3 Remove the sample from the oven and cool in a desiccator until the sample reaches room temperature, and weigh to the nearest 0.001g. Record the weight.

9.1.2.4 Return the soil sample to the oven for an additional time period (not less than 2 hours), cool again in the desiccator until the sample reaches room temperature, and weigh to the nearest 0.001g.

9.1.2.5 If the weight of the sample has remained constant ($\pm 4\%$) from the initial "dry" weight (9.1.3.4), use this number for the moisture determination (see 9.6.2). If the second weighing shows that the sample has lost further weight, continue drying and weighing the sample until the weight becomes constant, then proceed to 9.6.2.

9.1.2.6 If a sample contains a high concentration of petroleum product, constant weight may be difficult to attain. If, after several tries, the $\pm 4\%$ criteria cannot be reached an estimated % moisture may be reported with

appropriate explanation.

9.1.3 Dilution Technique

- 9.1.3.1 This is used for product or waste samples for which extraction is not appropriate and which are soluble in methylene chloride.
- 9.1.3.2 Weigh 1 g of sample into a 10 mL volumetric flask. Dilute to 10 mL with methylene chloride. Transfer to a 12 mL vial with a Teflon-lined lid. Mark meniscus and store at 4° ($\pm 2^\circ$) C, or -10 to -20 °C.

9.2 Gas Chromatography

- 9.2.1 Conditions (Recommended): Set helium column pressure to 20#. Set column temperature to 40° C for 2 minutes, then ramp at a rate of 12° C/min to 380° C and hold for 15 min. (run time = 49 minutes). Set FID Detector to 380° C and injector to 280° C. The reference book High Resolution Chromatography by Hewlett-Packard is a good source of information on how to optimize flow rates, etc.
- 9.2.2 Performance Criteria: GC run conditions and columns must be chosen to meet the following criteria:
- 9.2.2.1 Resolution of the methylene chloride solvent front from C₁₀, if DRO (AK102) is to be done simultaneously.
- 9.2.2.3 The separation number, TZ, should be greater than 15 for C₂₄ and C₂₅ if DRO is to be analyzed concomitantly.

$$TZ = \frac{\text{retention time } C_{25} - \text{retention time } C_{24}}{W_{1/2} \text{ of } C_{25} + W_{1/2} \text{ of } C_{24}} - 1$$

Where "W _{1/2}" = peak width at half-height

- 9.2.2.4 The column must be capable of separating typical motor oil components from surrogate and internal standards.

9.3 Calibration

- 9.3.1 Calibrate the GC, set up as in 9.2, with an initial five point (recommended) calibration using RCS (7.4.2). The final calibration curve must be represented by no less than 3 concentrations of RCS.
- 9.3.2 Choose Residual Calibration Standard concentrations to cover the RRO range expected in the samples, or the liner range of the instrument, whichever is less. One of the concentrations must be at or near the practical quantitation limit.
- 9.3.3 Tabulate the area response of the calibration standard against mass injected. The ratio of the response to the amount injected, defined as the response factor (RF), can be calculated for the standard at each concentration. If the average percent relative standard deviation (%RSD) is less than 25% over the working range, linearity through the origin can be assumed, and the continuing calibration response factor can be used in place of a calibration curve.

$$\text{Internal Standard Response Factor} = \frac{(Ax) (Cis)}{(Cx) (Ais)}$$

Where: Ax = Peak area of analyte

Ais = Peak area of internal standard

Cis = Concentration of internal standard

Cx = Concentration of analyte

Alternately, external standard calibration may be used (See Method 8000 [1]). Then,

$$\text{External Standard Response Factor} = \frac{\text{Total peak area of standard}}{\text{Mass injected}}$$

- 9.3.2 The calibration curve must be confirmed using the CVS (7.4.5). This standard verifies the accuracy of the calibration. The concentration of the CVS should be within the expected concentration range of the samples to be analyzed.
- 9.3.3 The working response factor or calibration curve must be verified on each working day (24 hours) by the injection of a CCS (7.4.3) at a concentration mid-point on the calibration curve. If the response for this standard varies from the predicted response by more than 25%, a new calibration curve must be prepared.

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_1} \times 100$$

where:

R_1 = Average RF from the calibration curve

R_2 = Response Factor from CCS

9.4 Retention Time Window Definition

9.4.1 Before establishing windows, be certain that the GC system is within optimum operating conditions (9.2). Make three injections of the Retention Time Window Standard (7.4.3) throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

9.4.2 Calculate the standard deviation of the three absolute retention times for each NAS component (C_{25} and C_{36}) and the surrogate.

9.4.2.1 The retention time window for individual peaks is defined as plus or minus three times the standard deviation of the absolute retention time for each component.

9.4.2.2 In those cases where the standard deviation for a particular analyte is zero, the laboratory should use ± 0.05 min. as a retention time window.

9.4.3 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed or instrument conditions changed. The data must be retained by the laboratory.

9.4.4 Retention time windows must be verified regularly and updated no less frequently than once a year.

9.5 Gas Chromatograph Analysis

9.5.1 Samples are analyzed by GC/FID. Optimum injection volumes (2 μ L using the conditions established in 9.2) must be established for specific instrument conditions.

9.5.2 For internal standard calibration, the internal standard is spiked into each sample and standard at a specified concentration. (Note: High RRO values may lead to measurement bias due to coelution with the internal standard.)

9.5.3 If initial calibration (9.3) has been performed, verify the calibration by analysis of a mid-point CCS (9.3.2). With each day's run, open a 24 hour analysis window. This is done by running the Retention Time Window Standard (7.4.3).

9.5.4 Calculate the percent difference of the response factor from the mean response factor as in 9.3.3. This is done for RRO as a group from the CCS. If the average response factor has a difference greater than 25%, corrective action must be taken.

- 9.5.5 A methylene chloride blank must be analyzed each day to determine the area generated on 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 C₂₅ and the peak end of C₃₆. This blank is integrated over the RRO area in the same manner as for the field samples and is reported as the solvent blank [Refer to reference 4]. **Do not baseline subtract. This information is for data interpretation purposes only.**
- 9.5.6 Blanks should also be run after samples suspected of being highly concentrated, to prevent carryover. If the blank analysis shows contamination above the practical quantitation limit, the column must be baked out and subsequent blanks analyzed until the system is shown to retain contaminants at concentrations less than the PQL.
- 9.5.7 If the RRO 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 response of the major peaks should be kept in the upper half of the linear range of the calibration curve. Due to potential measurement bias, internal standard calibration should not be used when RRO exceeds 5000 ug/mL in the final extract. The sample should be diluted or external standard calibration should be used.
- 9.5.9.1 Identification of motor oil or other products is achieved by direct comparisons of sample chromatograms to retention times and peak patterns of standard product chromatograms. Regardless of whether a product identification can be made on this basis, an alkane range or boiling point range should be reported to encompass approximately 95% of the product envelope, along with the midrange boiling point of the area of greatest response.
- 9.5.9.2 The analyst should generate a value for RROs and qualitatively identify motor oil or other products when reporting data. The experience of the analyst weighs heavily in the interpretation of the chromatogram. References 7, 8, and 9 contain some background information on hydrocarbon pattern recognition. Environmental samples may contain more than one type of product, and loss of light end components may mean the product has been in the subsurface a longer period of time.
- 9.5.9.3 Qualitative information must be reported on the final data transmittal. At a minimum, this information must include an alkane range or boiling point range of the product, and the boiling point of the area of maximum response. If possible, product identification should be included.

9.6 Calculations:

9.6.1 Internal Standard Calibration: The concentration of RROs in the sample must be determined by calculating the absolute weight of analyte chromatographed from a summation of peak response for all chromatographic peaks eluting between the peak start of n-pentacosane and the peak start of n-pentetracontane, using the calibration curve or the response factor determined in section 9.3. Also refer to Section 9.4 (Retention Time Window Definition).

The concentration of RRO is calculated as follows:

Aqueous/Soil samples:

$$C_s = \frac{(A_x)(C_{is})(D)(V_t)}{(A_{is})(RF)(V_s)}$$

Where:

C_s = Concentration of RROs (mg/L or mg/kg).

A_x = Response for the RROs in the sample, units in area.

RF = Response Factor from CCS (see 9.3.1).

A_{is} = Response for the internal standard, units same as for A_x .

C_{is} = Internal standard concentration (mg/mL).

V_t = Volume of final extract in mL.

D = Dilution factor, if dilution was performed on the sample prior to

V_s = Amount of sample extracted in L or kg.

9.6.2 To calculate mg/dry kg for soil samples,

$$\text{mg/dry kg RRO} = \frac{C_s}{1 - (\% \text{ moisture}/100)}$$

The % moisture calculation must be included in the data package (see 9.1.1).

9.6.3 External Standard Calibration:

Aqueous/Soil samples:

$$C_s = \frac{(A_x)(A)(V_t)(D)}{(A_s)(V_s)}$$

Where:

C_s = Concentration of RROs (mg/L or mg/kg).

A_x = Response for the RROs in the sample, units in area.

A_s = Response for the external standard, units same as for A_x .

A = External standard concentration (mg/mL).

V_t = Volume of Final extract in mL.

D = Dilution factor, if dilution was performed on the sample prior to analysis. If

Vs = Amount of sample extracted in L or kg.

- 9.6.4 Some software programs are capable of performing 9.6.1 and 9.6.3 with minimal analyst intervention. Additionally, some software programs can "update" a calibration curve based on the response of the CCS. If a calibration curve is updated in this manner, a valid CVS must be analyzed and results must fall within the Quality Control Criteria specified in Section 10 and Table 1 before samples can be analyzed.

10. Quality Control (See Table 1)

- 10.1 The laboratory must establish and maintain the ability to generate acceptable accuracy and precision and to demonstrate through the analysis of quality control check standards that the operation of the measurement system is in control. This must include the analysis of QC check samples plus the calculation of average recovery and the standard deviation of recovery as outlined in method 8000, section 8.0 [1], and this method.
- 10.2 After successful calibration (Section 9.3), analyze the reagent blank sample. The reagent blank must be analyzed with every extraction batch. The surrogate recovery must be within established limits (see Method AK 103.0, Table 1), or within the limits established by the project plan (whichever is more stringent) and the control sample should not have R R O above the practical quantitation limit.
- 10.3 With every batch, duplicate LFBs must be analyzed (employing the standard soil matrix appropriate to the samples being analyzed). The accuracy and precision of the duplicate standards must be within established limits. Also, the mid-point CCS must be analyzed at the end of each sequence and once per 20 samples, and compared to the successful calibration as described in 9.8.6, and fall within established limits (Table 1).
- 10.4 Every batch of samples extracted must be accompanied by a method blank to demonstrate that samples are free from method interference. The method blank must have RRO less than the Practical Quantitation Limit.
- 10.5 Each laboratory should generate control limits based on the average recovery, with ± 2 standard deviations as a warning limit and ± 3 standard deviations the action limit.
- 10.6 If any of the criteria in 9.3, 10.2, 10.3 and 10.4 are not met, corrective action must be taken.
- 10.7 Calculate the surrogate standard recovery in each sample. If recoveries are outside established limits (Table 1) verify calculations, dilutions, and standard solutions. Verify instrument performance.
- 10.7.1 High recoveries may be due to a coeluting matrix interference or the presence of

high molecular weight contaminants; examine the sample chromatogram.

- 10.7.2 High recoveries may also be due to memory effects caused by poor sample volatility, backflash, or carryover. Check instrument conditions, injection volume, and injector temperature.
- 10.7.3 Low recoveries may be due to adsorption by the sample matrix (muskeg, tundra, forest loam, etc).
- 10.7.4 Low recoveries may also be caused by incorrect integration. The chromatographic profile of RRO may not give baseline resolution of all components, resulting in a characteristic rise in baseline underneath the resolved hydrocarbon components known as the "unresolved complex mixture", or UCM. Do not use peak to peak integration; use forced baseline integration.
- 10.7.5 If internal calibration has been used, RRO results must be normalized using the internal standard response. If surrogate recovery is still outside of established limits, corrective action must be taken.
- 10.7.6 If external calibration has been used and surrogate recovery is outside of established limits, corrective action must be taken.
- 10.8 When field samples show surrogate recovery outside of acceptable limits due to suspected matrix effects and the calculated RROs concentration falls within a factor of 2 of the action level, the laboratory should recommend that the client resubmit the sample for matrix spike/matrix spike duplicate analysis. (To perform matrix spike analyses, follow 9.1, except use a field sample instead of a standard matrix.) This is a recommendation, not a requirement of the method, and therefore the onus is not on the analytical laboratory to absorb the cost of the additional analyses.
- 10.9 Matrix spikes are recommended for specific sampling programs. Field blanks, trip blanks, field duplicates are required as stated in Chapter 2, section 9 of the UST Procedures Manual.
- 10.10 Minimum quality control acceptance criteria are set forth in section 10 of this method. More stringent quality control criteria may be required by specific project plans.
- 10.11 Corrective Action
 - 10.11.1 Calibration
 - 10.11.1.1 If the initial calibration does not meet the criteria set forth in 9.3.3 and 9.3.4, the instrument must be recalibrated.
 - 10.11.1.2 If the continuing calibration does not meet the criteria set forth in

9.3.5 and Table 1, the instrument must be recalibrated.

10.11.2 Surrogates

10.11.2.1 If surrogates are outside established control limits (Table 1), the following assessments and/or correction actions must occur:

A) Check to be sure there are no errors in calculations and that the concentration of the surrogate and internal standard solution are correct.

B) Check instrument performance to determine if it is within acceptable guidelines.

C) Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

D) Reprepate and reanalyze the sample if none of the above resolves the problem.

10.11.2.2 If the surrogate recoveries that are outside the control limits cannot be attributed to lab error, the decision to reanalyze or flag the data should be made in consultation with the client. Provided all other QC acceptance criteria are met (section 10), it is only necessary to reprepare/reanalyze a sample one time to demonstrate that a poor surrogate recovery is due to matrix effects. A relationship can be established between surrogate recovery and moisture content of organic soils, which may help in diagnosing the cause of poor surrogate recoveries.

10.11.3 Blanks: Additional laboratory or field blanks may be necessary for certain projects to meet the goals of Chapter 2, section 9 of the UST Procedures Manual.

10.11.3.1 Instrument Blanks: Instruments must be evaluated with each batch (or daily, whichever is more frequent) and must demonstrate that the analytical system is free from contamination. This is best accomplished by analyzing an Instrument Blank.

10.11.3.2 Method Blank: Method Blanks must be analyzed with each extraction batch IF the results of the field samples show contamination above the MCL. The Method Blank for AK102 can also serve as the Reagent Blank if RRO is less than the PQL.

10.11.3.3 Field Blank: If the field samples yield DRO above the MCL, and

contamination is found below the PQL in the Reagent Blank, a Field Blank should be analyzed to identify whether the source of contamination originated in the field sample collection procedure, during trip or during storage in the laboratory. This applies to AK102 and AK103 analyzed in concomitantly.

(Note: Blanks are reported by value. DO NOT BLANK SUBTRACT. This information is for data quality assessment purposes only.)

10.11.4 Laboratory Fortified Blanks

10.11.4.1 If the analyte recovery from the LFBs is outside the established recovery limits (Table 1), the following assessments and/or corrective actions must occur:

A) Check to be sure there are no errors in calculations and that the concentration of the analyte solution is correct.

B) Check instrument performance to determine if it is within acceptable guidelines.

C) Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

D) Reprepare and reanalyze the samples if none of the above resolves the problem.

10.11.4.2 If the relative percent difference between the LFB results exceeds the control limits, but meets the percent recovery criteria (Table 1), the following assessments and/or corrective actions must occur:

A) Check to be sure that there are no errors in calculations, and that the same amount and source of analyte solution, solvent and water were used for both samples in the set.

B) Check to determine if instrument performance is still within acceptable guidelines, and that conditions did not change during the course of the batch analysis.

C) Recalculate the data if calculation error is suspected.

D) Repeat the LFB duplicate extraction and analysis, along with a representative number of samples (10% of the samples from the batch OR 1 sample, whichever is more) from the analytical batch with the failed LFB RPD. The re-analysis of the field samples is to demonstrate comparability of the extraction/analysis conditions at the time of re-extraction and analysis to those at the time of the failed QC.

11. Method Performance

11.1 Specific method performance data for Revision 3.0 of AK103, Residual Range Organics, is not available at this time. Information on method performance for the C25 - C44 range (Revision 2.1) follows.

11.2 The method performance data presented is based on single lab work (State of Alaska, Department of Environmental Conservation, State Chemistry Laboratory). Performance data for the RROs method in Ottawa sand and other soil types is presented below.

11.3 Results for motor oil spikes (methylene chloride extraction direct injection, soils) is from duplicate analyses of matrix spikes on field projects. Biases due to naturally occurring materials and existence of mixed products in the samples may exist.

<u>Matrix</u>	<u>RCS Spike Amount mg/kg</u>	<u>Percent Recovery</u>
Ottawa Sand (1993-1995)	500	91 ± 12
1993 Composite	250	77 ± 13
(S.E. Alaska Soils)	500	107 ± 15
1994 Composite	250	103 ± 10
(S.E. Alaska Soils)	500	103 ± 9
1995 Single Project	500	116 ± 9
(S. E. Alaska Soils)		

- 11.4 The method detection limit for soil calculated according to 40 C.F.R., Part 136, Appendix B (1994) was 51 mg/kg (external standard calibration).

12. References

1. USEPA "SW-846 Test Methods for Evaluating Solid Waste", 3rd Edition; Methods 8000, 8100, 3510, 3520, 3540, and 3550.
2. "Method OA-2: Extractable Petroleum in Products," Revision January 10, 1990", University Hygienic Laboratory, Iowa City, Iowa.
3. "Method for Determination of Extractable Petroleum Hydrocarbons (EPH) in Soil and Water," Draft-February 28, 1990, prepared for Total Petroleum Hydrocarbons Method Committee by Midwest Research Institute.
4. Zilis, K., M. McDevitt, and J. Parr, "A Reliable Technique for Measuring Petroleum Hydrocarbons in the Environment," presented at the conference on Petroleum Hydrocarbons and Organic Chemicals in Groundwater, NWWA, Houston, Texas, November 1988.
5. American Petroleum Institute "Method for the Determination of Diesel Range Organics," Draft Revision 2-February 5, 1992, prepared for Total Petroleum Hydrocarbons Method Committee.
6. "Leaking Underground Fuel Tank (LUFT) Field Manual," State Water Resources Control Board, State of California, Sacramento, CA, May 1988.
7. Fitzgerald, John, "Onsite Analytical Screening of Gasoline Contaminated Media Using a Jar Headspace Procedure" in Petroleum Contaminated Soils, Vol. 2, 1989.
8. Senn, R.B., and M.S. Johnson, "Interpretation of Gas Chromatographic Data in Subsurface Hydrocarbon Investigations," Ground Water Monitoring Review, 1987.
9. Hughes, B.M., and D.E. McKenzie, C.K. Trang, L.S.R. Minor, "Examples of the Use of an Advanced Mass Spectrometric Data Processing Environment for the Determination of Sources of Wastes" presented at 5th Annual Waste Testing and Quality Assurance Symposium, July 1989.
10. American Petroleum Institute, "Method for Determination of Petroleum Hydrocarbons," Draft Revision Original, 3 February 1992, prepared for the Total Petroleum Hydrocarbons Methods Committee.
11. State of Washington, Department of Ecology, "Total Petroleum Hydrocarbons Analytical Method WTPH-HCID."
12. Research done by the State of Alaska, Department of Environmental Conservation, Division of Environmental Quality, State Chemistry Laboratory.

Method AK 103.0, Table 1
ACCEPTANCE CRITERIA FOR QUALITY CONTROL

<u>ANALYTE</u>	SPIKE CONCENTRATION	CONTROL LIMITS	
	<u>Soil (mg/kg)</u>	<u>% Recovery</u>	Relative <u>% Difference</u>
Lab Control			
Samples			
Residual Range Organics	500 mg/kg	60 - 100	20
LCS/CCS			
Residual Range Organics	2000 mg/L	75 - 105	
Surrogate Control			
Samples			
n-Triacontane-d62	50 mg/kg	60 - 100	
Surrogate Recovery (field samples)			
n-Triacontane-d62	50 mg/kg	50 - 150	