APPENDIX B
SAMPLING AND ANALYSIS PLAN

SAP Worksheet #1 -- Title and Approval Page

FINAL
SAMPLING AND ANALYIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
Remedial Action, Installation Restoration Site 25
Former Naval Air Station Moffett Field, California
January 2012

Prepared for:
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Contract Task Order CTO 0003

Prepared by: _____ 1/17/2012
Richard Flynn Date
Program Chemist, ITSI Gilbane

Approved by: _____ 01/18/2012
Joseph Michalowski, Ph.D., C.H.M.M. Date
Acting Quality Assurance Officer
U.S. Navy, NAVFAC SW

EXECUTIVE SUMMARY

INTRODUCTION

ITSI Gilbane Company (ITSI Gilbane), has prepared this Sampling and Analysis Plan (SAP) to describe sampling and analysis activities to be performed during implementation of Remedial Action (RA) at Installation Restoration (IR) Site 25 at the former Naval Air Station Moffett Field, California. The project site is situated 35 miles south of San Francisco, and 10 miles north of San Jose, California.

ITSI Gilbane has prepared this SAP on behalf of the United States Department of the Navy (Navy). ITSI Gilbane is implementing the Remedial Action (RA) under the Naval Facilities Engineering Command Southwest (NAVFAC SW) Environmental Multiple Award Contract (EMAC), Contract Number N62473-10-D-0808, Task Order (TO) 0003.

SUMMARY OF PROJECT OBJECTIVES

The main objective of this project is to conduct a remedial action using excavation of sediment and off-site disposal so that IR Site 25 is available for unlimited use and unrestricted exposure, including potential use as a tidal marsh that supports a wide variety of habitat and species, and use as a managed pond that retains storm water and provides open water habitat. The excavations will progress as necessary to achieve the upper bound (do-not-exceed) remediation goals (RGs). The Navy does not anticipate excavating much deeper than 2 feet below ground surface (bgs) or expanding the excavations horizontally outside the polygons identified for remediation. A record of decision (ROD) was prepared by the Navy to present the remedy selected by the Navy and U.S. EPA with concurrence from the State of California, as represented by the California Regional Quality Control Board (Water Board). The Navy developed two sets of site-specific RGs in the feasibility study and published them in the ROD, as indicated in the table below. By attaining the (upper bound) do-not-exceed RGs at each polygon to be excavated (to be confirmed by confirmation sampling), the (lower bound) site-wide average RGs will be attained.

Remediation Goals for IR Site 25

	Lead	***Zinc***	***Total DDT***	***Total PCBs***
Site-Wide Average (lower bound)	33	180	0.016	0.200
Do-Not-Exceed (upper bound)	93.8	314	0.109	0.210

Notes:
1. RGs are for the tidal marsh scenario.
2. All values are in micrograms per kilogram (parts per million)

It is estimated that 33,000 cubic yards of material will be removed during the RA with options for additional excavation. Backfill of the excavation will only be required in areas if it is necessary to maintain the hydraulic conditions at the site (for example in wetland areas so that re-vegetation efforts are successful). This SAP also includes formal procedures for characterizing the clean fill sediment (on-site borrow material) so that the data are suitable for use in a post-RA calculation of site-wide average concentrations of COECs.

Table of Contents: SAP Worksheets

List of Figures

List of Attachments

Acronyms

ºC	degrees Celsius
ºF	degrees Fahrenheit
µg/L	micrograms per liter
ANSI/ASQ	American National Standards Institute/American Society for Quality
ASTM	American Society for Standards and Materials
BFB	bromofluorobenzene
BRAC	Base Realignment and Closure
BSU	Bay Sediment Unit
CA	corrective action
CAS	Chemical Abstract Service
CCC	Calibration Check Compound
CCR	California Code of Regulations
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
COC	chain of custody
COEC	constituent of ecological concern
COD	Chemical Oxygen Demand
COPC	Chemical of Potential Concern
CSM	Conceptual Site Model
CSO	Caretaker Site Office
CTO	Contract Task Order
CTR	California Toxics Rule
CWA	Clean Water Act
DoD	Department of Defense
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethylene
DDT	dichlorodiphenyltrichloroethane
DFG	Department of Fish and Game (California)
DI	deionized
DQI	Data Quality Indicator
DQO	Data Quality Objective
DRO	Diesel Range Organics
EDD	Electronic Data Deliverable
EDM	Eastern Diked Marsh
ELAP	Environmental Laboratory Accreditation Program
EPA	Environmental Protection Agency
EWI	Environmental Work Instruction
FCR	Field Change Request
FSP	Field Sampling Plan

Acronyms (Continued)

GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
GIS	Geographic Information System
GPS	Global Positioning System
GRO	Gasoline Range Organics
GW	groundwater
HCl	Hydrochloric acid
HPS	Hunters Point Shipyard
HSO	Health and Safety Officer
HSP	Health and Safety Plan
ICAL	initial calibration
ICV	initial calibration verification
IDW	investigation-derived waste
IPO	isolated petroleum occurrence
KCl	potassium chloride
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LIMS	Laboratory Information Management Systems
LNAPL	Light non-aqueous phase liquids
L/min	liters per minute
MB	method blank
MDL	Method Detection Limit
MO	Motor Oil
MS	mass spectrometer
MS/MSD	Matrix Spike/Matrix Spike Duplicate
msl	mean sea level
MROSD	Midpeninsula Regional Open Space District
mV	millivolts
NA	Not Applicable
NAVFAC	Naval Facilities Engineering Command
NCR	Nonconformance Report
NEDD	Navy Electronic Data Deliverable
NMCPHC	Navy and Marine Corps Public Health Center
NEIC	National Enforcement Investigations Center
NIRIS	Naval Installation Restoration Information Solution
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities List

Acronyms (Continued)

PAH	Polycyclic aromatic hydrocarbons
PA/SI	Preliminary Assessment/Site Investigation
PAL	Project Action Limit
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PDF	Portable Document Format
PM	Project Manager
PMO	Project Management Office
POC	Point of Contact
POE	point of exposure
POM	point of measurement
PQCM	Project Quality Control Manager
PQOs	Project Quality Objectives
PTFE	polytetrafluoroethylene (Teflon)
PVI	petroleum vapor intrusion
QA	quality assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	quality control
QCPM	Quality Control Program Manager
QCSR	Quality Control Summary Report
QL	Quantitation Limit
RAC	Remedial Action Contract
RCRA	Resource Conservation and Recovery Act
RL	reporting limit
ROICC	Resident Officer in Charge of Construction
RPD	relative percent difference
RPM	Remedial Project Manager
RWQCB	Regional Water Quality Control Board
SAP	Sampling and Analysis Plan
SD	standard deviation
SDG	sample delivery group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQLs	Sample Quantitation Limits
SRM	Standard Reference Material
SVOC	semivolatile organic compound
SWRP	Storm Water Retention Pond
SWSB	Storm Water Settling Basin
TBD	To Be Determined
TPH	Total Petroleum Hydrocarbons

Acronyms (Continued)

UFP	Uniform Federal Policy
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service
UST	Underground storage tank
VOA	volatile organic analyte
VOC	volatile organic compound

SAP Worksheet #2 -- SAP Identifying Information

Site Name/Number:	**Moffett Field**
Operable Unit:	**IR SITE 25Parcel B**
Contractor Name:	**Innovative Technical Solutions, Inc.**
Contract Number:	**N62473-10-D-0808**
Contract Title:	Naval Facilities Engineering Command Southwest (NAVFAC SW) Environmental Multiple Award Contract (EMAC)
Work Assignment Number (optional):	**CTO 0003**

1. This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP)* (U.S. EPA, 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (U.S. EPA, 2002).*

2. Identify regulatory program: CERCLA

3. This SAP is a: Project-Specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	**Date**
Navy Kick-Off Meeting with Bryce Bartelma, Dave Smith, Maryann Hough, and Scott Anderson (US Navy), Arvind Acharya, Robert Lindfors, Don Marini, Jim Schollard, and Carole Fried (ITSI Gilbane))	05 October 2010

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	**Date**
None	not applicable

6. List organizational partners (stakeholders) and connection with lead organization:

United States Environmental Protection Agency (USEPA) – environmental regulatory agency review for federal government

United States Fish and Wildlife Service (USFWS) – regulatory agency review

California Department of Fish and Game (DFG) – regulatory agency review

NASA Ames Research Center (ARC) – operator of Moffett Field facility

Midpeninsula Regional Open Space District (MROSD) – landowner of western portion of SWRP

California Regional Water Quality Control Board (Water Board) – environmental regulatory agency review for State of California

Remedial Advisory Board (RAB) for Moffett Field (document review)

7. Lead organization: United States Department of the Navy (Navy)

If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below. See following pages, right-hand column.

SAP Worksheet #2 -- SAP Identifying Information (Continued)

SAP elements and required information that are not applicable to the project.

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	
2	Table of Contents SAP Identifying Information	
3	Distribution List	
4	Project Personnel Sign-Off Sheet	
Project Organization		
5	Project Organizational Chart	
6	Communication Pathways	
7	Personnel Responsibilities and Qualifications Table	
8	Special Personnel Training Requirements Table	Omitted: no special training required
Project Planning/ Problem Definition		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	
11	Site-Specific Project Quality Objectives	
12	Measurement Performance Criteria Table	
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	
14	Summary of Project Tasks	
15	Reference Limits and Evaluation Table	
16	Project Schedule/Timeline Table	
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	
18	Sampling Locations and Methods/ SOP Requirements Table Sample Location Map(s)	
19	Analytical Methods/SOP Requirements Table	
20	Field Quality Control Sample Summary Table	
21	Project Sampling SOP References Table Sampling SOPs	Omitted: Information contained on Worksheets #14 and #17
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	
24	Analytical Instrument Calibration Table	
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	

SAP Worksheet #2 -- SAP Identifying Information (Continued)

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	
Data Management Tasks		
29	Project Documents and Records Table	
30	Analytical Services Table Analytical and Data Management SOPs	
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	
32	Assessment Findings and Corrective Action Responses Table	
33	QA Management Reports Table	
D. Data Review		
34	Verification (Step I) Process Table	
35	Validation (Steps IIa and IIb) Process Table	
36	Validation (Steps IIa and IIb) Summary Table	
37	Usability Assessment	

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1

Name of SAP Recipient	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address
Bryce Bartelma	Navy RPM	BRAC PMO West	619-532-0975	Bryce.bartelma.ctr@navy.mil 1455 Frazee Road, Suite 900 San Diego, CA 92108
Maryann Hough	BRAC Contract Specialist	BRAC PMO West	619-532-0791	Maryann.hough@navy.mil 1455 Frazee Road, Suite 900 San Diego, CA 92108
Joseph Michalowski	Acting Navy QAO	NAVFAC SW	619-532-4125	joseph.michalowski@navy.mil 1220 Pacific Highway, Bldg 127 San Diego, CA 92132-5190
Scott Anderson	BRAC Environmental Coordinator	BRAC PMO West	619-532-0938	scott.d.anderson@navy.mil 1455 Frazee Road, Suite 900, San Diego, CA, 92108
Doug DeLong	BRAC Caretaker Site Office (CSO)	Navy CSO	415-743-4713	doug.delong@navy.mil 1 Avenue of the Palms., Suite 161 San Francisco, CA 94130
Gary Munekawa	Resident Officer In Charge of Construction (ROICC)	ROICC San Francisco Bay Area	650-603-9834	Gary.munekawa@navy.mil P.O. Box 68 (Building 107) Moffett Field, CA 94035-0068
David Smith	Resident Officer In Charge of Construction (ROICC)	ROICC San Francisco Bay Area	650-603-9836	David.r.smith2@navy.mil P.O. Box 68 (Building 107) Moffett Field, CA 94035-0068
Melinda Dragone	U.S. EPA - Region 9 Project Manager	U.S. EPA	415-947-4184	Dragone.Melinda@epamail.epa.gov 75 Hawthorne Street San Francisco, CA 94105

SAP Worksheet #3 -- Distribution List (continued)

Name of SAP Recipient	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address
Elizabeth Wells, P.E.	Water Resources Control Engineer	Water Board	510-622-2440	1515 Clay Street, Suite 1400 Oakland, CA 94612
Robert Lindfors, P.E.	Project Manager	ITSI Gilbane	925-946-3173	rlindfors@itsi.com
Scott Lovesy	Site Superintendent	ITSI Gilbane	925-250-5972	slovesy@itsi.com
Richard Flynn	Program Chemist	ITSI Gilbane	925-946-3103	rflynn@itsi.com
Eric Mruz	Refuge Manager	US FWS	510-792-0222	9500 Thornton Ave. Newark, CA. 94560
Ryan Olah	Project Manager	USFWS	916-414-6639	2800 Cottage Way, Rm W-2605 Sacramento, CA 95825
Florence Gardipee	Biologist	USFWS	916-414-6675	2800 Cottage Way, Rm W-2605 Sacramento, CA 95825
Allen C.L. Tsao	Toxicologist	DFG	916-323-4731	1700 K Street, Suite 250 Sacramento, CA 95811
Tami Nakahara	Wildlife Biologist	DFG	916-324-8452	1700 K Street, Suite 250 Sacramento, CA 95811
Don Chuck	Restoration Project Manager	NASA ARC	650-604-0237	Mail Stop 237-14 (Bldg 241, Room 104) Moffett Field, CA 94035
Cameron L. Johnson	Project Manager	USACE San Francisco District	415-503-6773	1455 Market Street San Francisco, CA 94103
Ana Ruiz	Planning Manager	MROSD	650-691-1200	330 Distel Circle Los Altos, CA 94022-1404
Bill Berry	RAB Co-Chair	N/A	650-604-0511	PO Box 7 Moffett Field, CA 94035
Linda Ellis	RAB Member	N/A	408-772-3289	550 Ellis Street Mountain View, CA 94043

Name of SAP Recipient	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address
Bob Moss	RAB Member	N/A	N/A	Bmoss33@att.net 4010 Orme Palo Alto, CA 94306
Lenny Siegel	RAB Member	N/A	650-969-1545	269 Loreto Street Mountain View, CA 94041
Peter Strauss	RAB Technical Advisor	N/A	N/A	petestrauss1@comcast.net 317 Rutledge Street San Francisco, CA 94110
Libby Lucas	RAB Member	N/A	N/A	Jlucas1099@aol.com 174 Yerba Santa Los Altos, CA 94022
Steve Williams	RAB Member	N/A	650-906-7827	200 Park Ave Santa Cruz CA 95062

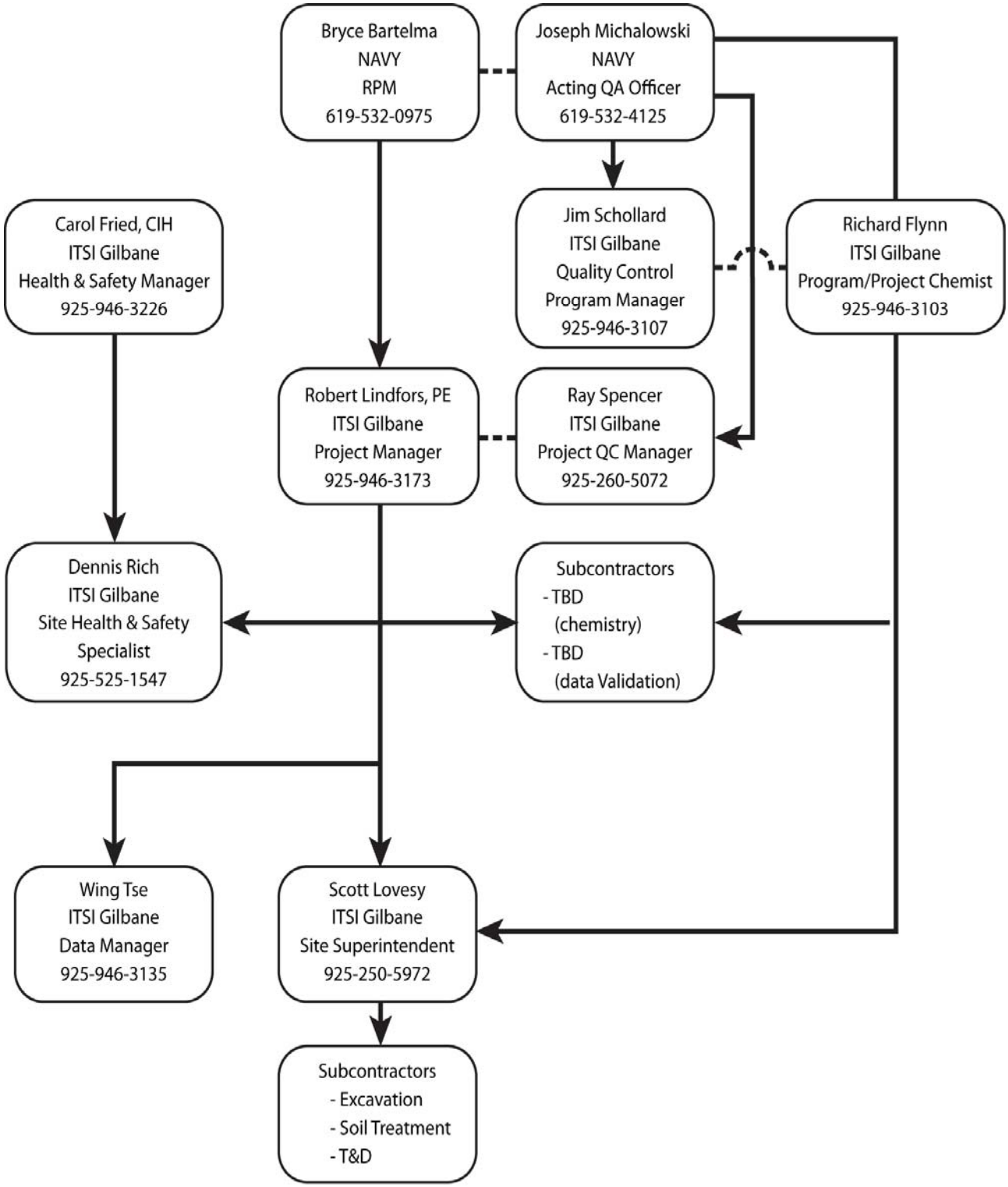
N/A = Not available

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Kimberly Tom	ITSI Gilbane/Data Manager	925-946-3135			
Robert Lindfors	ITSI Gilbane/Project Manager	925-946-3173			
Scott Lovesy	ITSI Gilbane/Site Superintendent	925-250-5972			
Gail Jones	ITSI Gilbane/Site Coordinator	925-946-3291			
Ray Spencer	ITSI Gilbane/Project Quality Control Manager	925-946-3107			
Dennis Rich	ITSI Gilbane/Health and Safety Officer	925-250-1547			
Sue Bell	Accutest/Laboratory Project Manager	813-741-3338			
Dolores Queka	Accutest/Laboratory QAO	408-588-0200			
Evin McKinney	Synectics	916.561.3180			

Note: Completed Project Personnel Sign-Off Sheet will be maintained in project files in Walnut Creek office.

SAP Worksheet #5 -- Project Organizational Chart
(UFP-QAPP Manual Section 2.4.1)



SAP Worksheet #6 -- Communication Pathways
(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Approvals	Acting Navy QAO Navy RPM	Joseph Michalowski Bryce Bartelma	joseph.michalowski@navy.mil Bryce.bartelma.ctr@navy.mil	ITSI Gilbane PM to request approval of SAP from QAO and approval of Work Plan from Navy RPM. PM to acquire approval to initiate field work from Navy RPM and QAO after appropriate agency approval has been obtained.
Corrective Actions	ITSI Gilbane PQCM ITSI Gilbane Program Chemist ITSI Gilbane PTM	Ray Spencer Richard Flynn Gail Jones	rspencer@itsi.com rflynn@itsi.com gjones@itsi.com	Initiator of needed corrective action notifies responsible party, documents need for action, and forwards documentation to PM and PQCM for concurrence or rejection; responsible party documents action taken and forwards to PM for review.
Changes to field methods	ITSI Gilbane Site Superintendent	Scott Lovesy	slovesy@itsi.com	ITSI Site Coordinator notifies PM of the need for changes to field procedure. Pm notifies Navy RPM and QAO. Approval from Navy RPM must be obtained before proceeding. Only significant changes require QAO approval.
Notifications	ITSI Gilbane PM	Robert Lindfors	rlindfors@itsi.com	PM to provide Navy RPM and ROICC with all project-required notifications within 24 hours. All non-conformant laboratory data will be reported to the NAVFAC SW QAO within 24 hours or next business day after discovery.

SAP Worksheet #6 -- Communication Pathways (Continued)

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Stop work issues	ITSI Gilbane QCPM ITSI Gilbane PQCM ITSI Gilbane HSO Acting Navy QAO Navy RPM	Jim Schollard Ray Spencer Robert Guerrero Joseph Michalowski Bryce Bartelma	jschollard@itsi.com rspencer@itsi.com rguerrero@itsi.com joseph.michalowski@navy.mil Bryce.bartelma.ctr@navy.mil	Initiator of stop work order notifies Navy ROICC and ITSI Gilbane PM. For stop work initiated by the Navy, the Contracting Officer must be notified.
Excavation activities and location modifications	ITSI Gilbane Site Superintendent	Scott Lovesy	slovesy@itsi.com	Obtain approval to commence work and to change any locations or field methods from the ITSI Gilbane PM. Work will not proceed without approval by the PM.
Hazardous conditions during excavation that raise question of stopping work (e.g., BTXE + MTBE above action level of 5 ppm in breathing zone)	ITSI Gilbane Site Superintendent ITSI Gilbane SHSS	Scott Lovesy Dennis Rich	slovesy@itsi.com drich@itsi.com	Site superintendent will confer with ITSI Gilbane HSO to determine whether work needs to be stopped; the HSO will report stop-work decision to ITSI Gilbane PM and Navy ROICC

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table
(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Bryce Bartelma	Navy RPM	US Navy	Overall project execution and coordination with site representatives, regulatory agencies, and Navy management. Actively participates in the DQO process, and provides management and technical oversight during data collection. Has authority to approve real-time modifications to the project (in coordination with Navy Contracting staff), is notified of delays in or changes to field work, and has authority to stop work and initiate corrective action at any time.
Joseph Michalowski	Acting Navy QAO	US Navy	Responsible for QA issues for all Navy work; provides government oversight of the QA program for contractors. Reviews and approves the SAP and any significant SAP modifications or amendments; has the authority to suspend project activities if Navy quality requirements are not met.
Robert Lindfors	PM	ITSI Gilbane	Develops and implements all Task Order documents and activities. Assures overall project quality, implementation of three-phase quality control activities, and compliance with project schedule; and performs contract management, technical oversight, and report generation. Responsible for notifying the RPM of significant project information, including (but not limited to) project progress, schedule compliance, modifications to work, delays, analytical data quality issues, and safety-related issues.
Jim Schollard	Quality Control Program Manager	ITSI Gilbane	Reviews QC processes, issues corrective action orders; assures adherence to requirements of the QC program, including the CDQMP, Contractor Quality Control (CQC) Plan, and SAP, as appropriate. Can receive communication from the PM, Program Chemist, PQCM, and field staff. Has the authority to stop work and initiate corrective action.

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table (Continued)

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Richard Flynn	Program Chemist	ITSI Gilbane	Assists the QC Program Manager in assuring that project activities adhere to the requirements of the CDQMP and assesses the propriety of the proposed analytical methodology; assists in the preparation of the SAP and with management of project tasks associated with sampling; reviews preservation requirements; coordinates SAP review/approval and other QA issues with the Navy QAO; conducts general oversight of and communication with Project Chemist and field personnel in relation to sampling activities; coordinates sample collection and analysis with the analytical laboratory; implements appropriate quality control activities and corrective actions; coordinates data validation activities and the uploading of data to appropriate databases.
Scott Lovesy	Site Superintendent	ITSI Gilbane	Conducts oversight of all field activities; ensures implementation of individual elements of project-specific work plans and sampling plans; responsible for overseeing the work of any subcontractors performing field-related tasks.
Gail Jones	Site Coordinator	ITSI Gilbane	Ensures implementation of individual elements of project-specific sampling plans and is responsible for overseeing the collection of samples and coordinating shipments with laboratories; ensures that the sampling protocol is followed per the SAP.
Ray Spencer	Project QC Manager	ITSI Gilbane	Implements field-related quality control activities, issues nonconformance reports (NCRs), initiates necessary rework and/or corrective actions, and communicates with the PM, QC Program Manager, Site Coordinator, and Project Chemist.
Richard Flynn	Project Chemist	ITSI Gilbane	Coordinates sample collection and analysis with the analytical laboratory, reviews analytical data as it is reported, and implements appropriate quality control activities and corrective actions.

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table (Continued)
(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Sue Bell	Laboratory Project Manager	Accutest	Directs the performance chemical analyses; assures compliance with all project requirements regarding performance of analytical procedures; supplies sample containers; handles and preserve samples in accordance with project-specified protocols.
Evin McKinney	Data Validator	Synectics	Performs data validation on all analytical data used for project decisions.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Remedial Action

Site Name: Moffett Field, Site IR 25

Site Location: Moffett Field, California

Projected Date(s) of Sampling: Beginning May 2011

Project Manager: Robert Lindfors, Innovative Technical Solutions, Inc.

Date of Scoping Session: 05 October 2010

Scoping Session Purpose: Discuss objectives, scope and tentative schedule

Scoping Session Participants:

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Bryce Bartelma	Remedial Project Manager	Navy	619- 532-0975	Bryce.bartelma.ctr@navy.mil	Navy RPM
Arvind Acharya	Program Technical Manager	ITSI Gilbane	925-946-3206	aacharya@itsi.com	ITSI Gilbane Program Manager
Robert Lindfors	Project Manager	ITSI Gilbane	925-946-3173	rlindfors@itsi.com	ITSI Gilbane PM
Gail Jones	Site Coordinator	ITSI Gilbane	925-946-3291	gjones@itsi.com	ITSI Gilbane Site Coordinator

Comments/Decisions:

Action Items:

Consensus Decisions:

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SAP Worksheet #10 -- Problem Definition
(UFP-QAPP Manual Section 2.5.2)

Step 1 of the Data Quality Objectives (DQO) process for this project is presented on this worksheet. Steps 2 through 7 are presented on Worksheet #11.

Step 1: State the Problem.
Previous uses at the Site have resulted in contamination of the sediment with lead, zinc, PCBs and DDT at concentrations which pose a risk to ecological receptors under a tidal marsh reuse scenario. A remedy has been selected to address concentrations in sediment above the established RGs. The remediation strategy is described in the final ROD (Navy, 2009). The remedy selected in the ROD includes excavation of contaminated sediment, as well as in-situ or ex-situ treatment of lead and zinc contamination, and focused restoration and ecological monitoring in areas disturbed by the remedial activities. Prior to placement of any fill material, samples will be collected and analyzed for the COECs. If chemical concentrations in individual samples of the borrow material do not exceed site-specific action levels (see Worksheet #15) then the material will be used to backfill excavated areas. Results of chemical tests of borrow will also be used in the calculation to attain the site-wide average (lower bound) RGs.

Establish the Planning Team:
For this project, the DQO planning team members include the Navy remedial project manager (RPM), contractor technical staff, and representatives of the lead Federal and State regulatory agency stakeholders (USEPA and Water Board, respectively). As lead agency, the Navy is the primary decision-maker with ultimate authority for making final decisions based on the recommendations of the planning team.

Site Background:
The principal elements of the conceptual model are as follows:

- **PHYSICAL SETTING:** Moffett Field is located 35 miles south of San Francisco and 10 miles north of San Jose. IR Site 25 is approximately 230 acres in size and is located in the northwest corner of Moffett Field. Levees constructed in the late 19th and early 20th centuries for commercial salt production currently prevent bay water from reaching IR Site 25. IR Site 25 consists of the Eastern Diked Marsh (EDM; approximately 20 acres), which includes a Storm Water Settling Basin (SWSB); and the Storm Water Retention Pond (SWRP; approximately 210 acres).

- **CLIMATE:** The site and the San Francisco Bay Area in general are characterized by a Mediterranean climate, with mild summer and winter temperatures. Mean annual precipitation in San Francisco is 22.1 inches, with the majority of the precipitation occurring between November and March. The mean yearly low and high temperatures are 51 degrees Fahrenheit (^{o}F) and 63 ^{o}F, respectively. The area is often marked by heavy fog, which can impair navigation.

- **GEOLOGY and HYDROGEOLOGY:**. Sediment at IR Site 25 consists of varying combinations of clay, silt, sand and gravel, as a result of the mixing of estuarine and alluvial deposits which occurred during the late Pleistocene and Holocene Epochs.

  Groundwater at the Site occurs in upper and deep aquifers, with continuous and semi-continuous aquitards dividing the two. The upper aquifers are divided into A, B, and C aquifers. Agricultural wells have been historically located in the C aquifer at Moffett Field, and drinking water wells have been located in the C aquifer elsewhere in the Santa Clara Valley. No agricultural or municipal wells are located in the A or B aquifers at the Site.

- **PREVIOUS STUDIES:** From 1994 to 2006, the Navy conducted a series of environmental investigations at IR Site 25 in conjunction with NASA, EPA, and the Water Board. A station-wide Remedial Investigation (RI) Report for Moffett Field was completed in 1996 and included a baseline human health risk assessment (HHRA). Phase I and Phase II Site-wide Ecological Assessments (SWEA) were completed in 1994 and 1997. The RI Report and Phase II SWEA evaluated IR Site 25 under its current land use as a storm water retention pond that provides seasonal wetland habitat. Because tidal marsh restoration is being considered by MROSD and NASA as a future land use, the Navy prepared an addendum to the Station-Wide RI Report in 2005 to evaluate potential risk for the revised land use for IR Site 25. As part of that addendum, the nature and extent of contamination, baseline HHRA, and ecological risk assessment (ERA) were updated to evaluate potential risks to human and wildlife at IR Site 25 if it is restored to a tidal marsh. In addition, the vertical extent of contamination was defined during pre-excavation sampling activities in 2002. As part of these investigations, sediment and surface water samples were collected to investigate the nature and extent of contamination at IR Site 25. Sediment samples were collected from 284 locations throughout all portions of IR Site 25, and 18 surface water samples were collected from the Eastern Diked Marsh and stormwater retention pond. Samples were analyzed for a variety of chemicals of potential concern, including metals, pesticides, polychlorinated biphenyls (PCB), and petroleum hydrocarbons.

- **REGULATORY FRAMEWORK:** Remediation of Moffett Field, Site 25 is being administered under Section 117(a) of CERCLA and Section 300.430(f)(2) of the National Oil and Hazardous Substances Pollution Contingency Plan (NCP). Remedial Investigation (RI) and Feasibility Study (FS) Reports and Addenda were prepared for IR Site 25. These reports and other documents are contained in the administrative record file for Site 25. A public comment period was held from January 9 through February 9, 2009, and a public meeting was held on January 22, 2009. As a result of this regulatory process, site-specific RGs for sediment at IR Site 25 for the tidal marsh scenario were developed based on a comparison of risk-based concentrations with background concentrations for lead, zinc, total DDT, and total PCBs. A range of RGs was developed for each chemical to meet the Remedial Action Objectives for IR Site 25. RGs for each chemical at Site 25 include both a site-wide average (lower bound) and a do-not-exceed (upper bound) value.

SAP Worksheet #10 -- Problem Definition (Continued)

Conceptual Site Model:

Constituents of Ecological Concern (COECs):

Results of the previous investigations have found lead, zinc, dichlorodiphenyltrichloroethane (DDT) and its breakdown products, and PCBs in sediment at concentrations that pose unacceptable risk to the environment.

Sources:

The likely sources of this contamination are from historical discharge of storm water to IR Site 25, and routine application of DDT before it was banned in the 1970s. Storm water discharge to IR Site 25 is currently monitored and controlled as part of NASA's storm water permit. In 1997, Aroclor-1268 was detected in sediment samples collected from the SWSB. The source of the Aroclor-1268 was traced to Hangar 1 by sampling the manholes in the storm water collection system upstream from the settling basin. The source of the Aroclor-1268 was identified as the building materials of Hangar 1. In 2004, NASA conducted a source identification study to identify potential sources of PCBs to the storm water system and ultimately to IR Site 25. Sediment samples were collected from a swale south of the Site 8 fence (the Waste Oil Transfer Area) and around Buildings 26, 45, 525, 583C, and 951. Aroclor-1260 was detected in sediment samples at these locations. These potential sources of contamination to IR Site 25 will be addressed by separate remedies. Two areas close to the two storm water discharge locations in the EDM were identified as containing the most elevated levels of lead, zinc, total DDT (i.e. DDT and its breakdown products DDD and DDE), and PCBs in the sediment. The first area is located near the discharge from the SWSB, and the second area is the site of the historical outfall for the Lindbergh Avenue ditch. In general, concentrations of chemicals are higher near these locations and lower farther from these locations. In 2005, NASA removed PCB-contaminated sediments from a limited area directly adjacent to the SWSB discharge.

Fate and Transport:

The COECs are expected to be relatively immobile at the Site. These COECs tend to bind with sediments rather than existing is soluble forms. In a typical marsh environment (a reducing environment), lead and zinc tend to form complexes with organic material, to sorb to oxides and clay minerals, and to form insoluble sulfides. DDT and PCBs have a strong affinity for sediments and organic matter, and do not easily partition into water. Because of this immobility, the primary transport mechanisms are re-suspension and physical transport. The sediments at IR25 are unlikely to experience re-suspension or transportation because there is little circulation within the marsh; there are no tidal currents, and the shallow depth of the retention pond limits the effect of waves.

A major chemical fate of DDT and PCBs in sediment is degradation and transformation by microbial metabolism; however, degradation by any means occurs very slowly. For PCBs, fate is affected by the degree of chlorination. The more highly chlorinated PCB congeners may persist in the environment for decades.

SAP Worksheet #10 -- Problem Definition (Continued)

Potential Risk Drivers:

Chemical data were used to assess potential risks to both human and ecological receptors (plants and animals that inhabit or visit the site) under the future use of IR Site 25 as a tidal marsh. The Environmental Risk Evaluation (ERA) (PRC, 1994) concluded that risks to ecological receptors are driving the risk at IR Site 25; therefore, ecological receptors are the focus of the remedial action for IR Site 25. Risks to human receptors were found to be at an acceptable level at Site 25, however, it is noted that remediation to protect ecological receptors will further reduce potential risk to human health. The ERA concluded that lead, zinc, total DDT, and PCBs in sediment pose an unacceptable risk to invertebrates, birds, and mammals, and a potential risk to amphibians and reptiles that would be present at the site if it were restored to tidal marsh. Two-tiered RGs were developed for each chemical to meet the Remedial Action Objectives for IR Site 25. The RGs (as developed by Navy in the ROD) for each chemical at IR Site 25 consist of a site-wide average and a do-not-exceed value.

Backfill of the excavation will only be required in areas if it is necessary to maintain the hydraulic conditions at the site (for example in wetland areas so that re-vegetation efforts are successful). Prior to placement of any fill material, samples will be collected and analyzed for the COECs. If chemical concentrations in individual samples of the borrow material do not exceed the do-not- exceed (upper bound) RGs (see Worksheet #15) then the material will be used to backfill excavated areas. Results of chemical tests of borrow material will also be used in the calculation to attain the site-wide average (lower bound) RGs.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

Steps 2 through 7 of the DQO process for this project are presented below.

Step 2: Identify the Goal of the Study

Specify the principal study questions:
The goal of this project is to conduct a remedial action using excavation of sediment and off-site disposal, in order to make IR Site 25 available for unlimited use and unrestricted exposure. The purpose of sampling is to determine when a sufficient amount of contaminated sediment has been removed to achieve the do-not-exceed RGs shown below. By attaining the do-not-exceed (upper bound) RGs at each polygon to be excavated (to be confirmed by confirmation sampling), the (lower bound) site-wide average RGs will be attained.

	Lead	***Zinc***	***Total DDT***	***Total PCBs***
Site-Wide Average RG (lower bound)	33	180	0.016	0.200
Do-Not-Exceed RG (upper bound)	93.8	314	0.109	0.210

Notes:
1. RGs are for the tidal marsh scenario.
2. All values are in micrograms per kilogram (parts per million)

Principal study question:
1. Are COECs present in the post-excavation sediment samples at levels which exceed the do-not-exceed RGs?
2. Are COECs present in the material proposed for backfilling of the excavated areas at levels exceeding the site-specific action limits (see Worksheet #15)?

Step 3: Identify Information Inputs

Identify the types of information that are needed.
Data on the concentration of site COECs in the remaining sediment after contaminated sediment has been removed is needed to determine whether or not further excavation is necessary.

Identify the sources of information.
The needed information will be obtained by collecting and analyzing sediment confirmation samples from excavations, and from samples of the borrow material, as described on Worksheets #14, #17, and #18.

Identify appropriate sampling and analysis methods.
Collection of excavation confirmation sediment samples will be performed in accordance with standard field methods as described on Worksheets #14, #17 and #18. Sediment samples will be analyzed for the following COECs :

- Lead and zinc by EPA Method 6010B;
- Six isomers of DDT by EPA Method 8081A;
- Total PCBs by EPA Method 8082.

SAP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements (Continued)

Borrow material will be analyzed for the COECs, as follows:

- Lead and zinc by EPA Method 6010B;
- Six isomers of DDT by EPA Method 8081A;
- Total PCBs by EPA Method 8082.

The project quantitation limit goals associated with the test methods are identified on Worksheet #15.

Step 4: Define the Boundaries of the Study

Specify the target population of interest:
Target population includes all possible sets of sediment samples remaining from excavated areas within the Site.

Specify the spatial and temporal boundaries and other practical constraints:
The lateral extent of excavation is limited to the boundaries identified on Figures SP-1 through SP-10. The excavations will progress as necessary to achieve the do-not-exceed RGs; however, Navy does not anticipate excavating deeper than 2 feet below ground surface (bgs) or expanding the excavations horizontally outside the polygons identified for remediation.

Temporal considerations: Sediment removal (excavation) activities and T&D activities are scheduled to start in summer 2012. This start date will allow for sediment handling and sampling to occur during dry-weather months to provide clean, efficient site operations. The precise sequence of excavation will depend on presence or absence of surface water and wildlife in the areas to be excavated, and will be decided upon during the pre-construction phase just before major site activities are initiated.

The current period of performance for this task order, including reporting, extends to 17 September 2013.

Specify the scale of estimates to be made:
The analytical methods selected for analysis should provide sufficient accuracy to estimate concentrations within the range of applicable action levels for this project. Therefore, the selected methods should be able to adequately evaluate whether or not COECs are present at concentrations above the project action levels, and thus provide data usable for project decisions.

Step 5: Develop the Analytical Approach
Determine the key study parameter and a specification of the estimator:
The key parameters to be estimated are the concentrations of lead, zinc, total DDT, and total PCBs in site sediment. The data will be used to evaluate whether or not COECs are present at concentrations below the project action limits, and if additional excavation is necessary to reduce concentrations to acceptable levels.

Specify the Action Level
The results of the investigation will be compared to the project action limits specified on Worksheet #15.

SAP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements (continued)

Specify the Decision Rules

1. After sediment has been excavated at a given location, **if** concentrations of COECs in sediment samples from the excavation floor are less than the project action limits (see Worksheet #15), **then** conclude that potential adverse impacts to ecological receptors are not present in the sediment and recommend no further excavation; **else** conclude that potential risks to ecological receptors are present, excavate an additional 6 inches of sediment, and take another sediment confirmation sample. **If** the concentrations of COECs in sediment are not less than the project action limits after two additional excavations of 6 inches, **then** ITSI Gilbane will consult with the Navy to plan an appropriate course of action; **else** conclude that potential adverse impacts to ecological receptors are not present in the sediment.

2. **If** the concentrations of COECs in the fill material are lower than the do-not-exceed RGs, **then** the material will be considered suitable for use as backfill material in areas where backfill is required (see Salt Marsh Habitat Restoration Plan [Appendix H to the RD/RAWP] for areas to be backfilled); **else** conclude that potential risks to ecological receptors are present in the borrow material; reject this material and locate a suitable source. Analytical results of the borrow material will also be used in the calculation to prove attainment of the site-wide average (lower bound) RGs.

Step 6: Specify Performance or Acceptance Criteria

Specify how uncertainty will be accounted for in the estimate:
A biased sampling scheme has been designed to identify appropriate sampling locations to evaluate site subsurface conditions. The locations were selected on the basis of previous sampling results (ROD, Navy, 2009). Because inputs are subjective, uncertainty in decision errors cannot be rigorously quantified using statistical evaluation.

To limit uncertainty in the data obtained, criteria for the precision, accuracy, representativeness, completeness, and comparability parameters and reporting limits for the chemicals of concern have been developed (Worksheet #37). The data that meet these criteria will be of definitive quality. Data that do not meet these criteria are not considered definitive, and may be used only if qualified as estimated data.

Specify performance or acceptance criteria:
Samples must be collected and analyzed in accordance with the QAPP, and project quantitation limits (QLs) for samples must be equal to, or less than, the project action levels, as specified on Worksheet #15.

SAP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements (Continued)

Step 7: Develop the Detailed Plan for Obtaining Data

Select the sampling design:

The sampling approach and methods, sampling design, and rationale are described in Worksheet #17. The number and frequency of samples to be collected are listed in Worksheet #18. For developing the sampling design for confirmation sampling, the polygons to be excavated were separated into three groups, corresponding to small, medium, and large areas, i.e., Group 1--less than 10,000 square feet (SF); Group 2--10,000 SF to less than 40,000 SF; and Group 3--40,000 SF and greater. Group 1 areas will have one confirmation sample collected from the excavation footprint; Group 2 areas will have two; and Group 3 areas will have four. For most areas, this approach correlates to a frequency of about 1 sample per 100 ft by 100 ft area. A frequency of one sample per 10,000 ft2, correlating to an area of 100' by 100', is more conservative than the original sample grid used in characterizing the site, i.e., 160 foot spacings in the north and central portions of the SWRP and 80-foot spacings for the southern portion of the SWRP and for the EDM.

SAP Worksheet #12 -- Measurement Performance Criteria Table
(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – Field QC Samples

Matrix: Sediment

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	**QC Sample** (Assesses Error for Sampling [S], Analytical [A] or both [S&A])
Field Duplicates	All	NA[1]	NA	NA	NA
Source Water Blanks[2]	All	one per batch of source water[2]	Accuracy/Bias – Contamination	<RL	(S)
Equipment Rinsate Blanks[2]	All	one per day of sampling[2]	Accuracy/Bias – Contamination	<RL	(S)
Temperature Blanks	All	one per shipping container (cooler)	Accuracy/Bias	$4 \pm 2\ ^{o}C$	(S)

Notes:
RL = reporting limit
NA = not applicable

[1] Due to the heterogeneous distribution of contaminants typically found in soils/sediments, field duplicate samples are not considered reliable for determining analytical precision, and will not be collected for this project.

[2] The exclusive use of disposable equipment is intended to obviate the need for these blanks. However, should circumstances necessitate the need for re-usable equipment, then these blanks will be collected at the specified frequency.

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
NA	NA	NA	NA	NA

Notes:

NA = not applicable

SAP Worksheet #14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

Major tasks associated with the sampling effort will include the following:

Pre-sampling Tasks:

Before beginning field work, Navy RPM, Navy ROICC, and appropriate agencies/personnel will be notified. Field personnel will read the relevant section of the SAP, read the Site-Specific Health and Safety Plan, and sign the Project Personnel Sign-off Sheet (work sheet #4 of this SAP). ITSI Gilbane will secure all pertinent permits and conduct biological surveys and utility clearance activities as described in Sections 4.0 and 5.0 of the Work Plan.

Sampling Tasks:

Confirmation samples will be collected from the base of each excavation. Sediment samples from the surface of the sediment remaining in place after excavation will be taken from each polygon using a hand auger or disposable scoop. Samples will be submitted to a fixed laboratory for analysis as described on Worksheet #15. If non-disposable sampling equipment is used, equipment rinsate blanks will be collected per Worksheet #20.

Post-Sampling Field Tasks:

Equipment decontamination, waste characterization and disposal, and surveying will be performed as detailed in SAP Worksheets #17 and #21, and Section 6.0 of the Work Plan.

Analytical Tasks:

Sediment samples collected during sampling activities will be analyzed at a subcontracted laboratory. Samples will be analyzed for the following COECs :

- lead and zinc by EPA Method 6010B;
- six isomers of DDT by EPA Method 8081A;
- Total PCBs by EPA Method 8082.

The project quantitation limit goals associated with the test methods are identified on Worksheet #15.

Quality Control Tasks:

Analytical methods will require the applicable QC tasks described in the respective methods, including initial calibrations, continuing calibrations, tuning, reagent blanks, surrogates, replicates, control spikes, and others, as necessary. Media-specific field quality control samples (as described on Worksheet #12) including source water blanks, equipment rinsate samples, and field duplicates will be collected as appropriate, and used to measure process performance.

SAP Worksheet #14 -- Summary of Project Tasks (Continued)

Data Management and Review Tasks:

Analytical data generated by the fixed analytical laboratory will be reviewed by the laboratory using three levels of document review and reporting. Review processes will be documented using appropriate checklists, forms, or logbooks, which will be signed and dated by the reviewers. Analytical and field data will be uploaded to the Naval Installation Restoration Information Solution (NIRIS) database via the Navy Electronic Data Deliverables (NEDD) format.

Third Party Data Validation:

Data review and validation will be performed by an independent third-party data validation subcontractor. Moffett Naval Air Station is on the EPA NPL Site List, thus the data will be validated at 80% EPA Level III and 20% EPA Level IV, as described on Worksheet #36.

Procedures for data recording, management, auditing, and correction will include the following:

Field Documentation

Complete and accurate documentation is essential to demonstrate that field sampling procedures are carried out as described in this SAP. Field activities and sample collection will be documented using standard forms including, but not limited to: boring logs, well completion logs, well development forms, groundwater sampling forms, sample labels, chain of custody forms, waste management labels, and hazardous waste labels. The purpose of standardized field documentation and sampling procedures is to maintain the integrity and defensibility of field documentation and field samples throughout the project. Examples of field forms are presented in Attachment 1.

Each field sample will be labeled and sealed immediately after it is collected. Sample labels and other sample-identification documents will be carefully prepared to maintain control of sample disposition, as described in SAP Worksheet #27.

Field personnel will record and document field activities in a logbook and/or other media. All entries must be legible, in ink, and primarily factual in content. The logbook will list the project name and number, the site name and location, and the names of subcontractors, the service client, and the project manager. Hypothetical information may be entered but will be noted as such. Logbook corrections will be made by striking out the incorrect entry with a single line and entering the correct information. Both entries and corrections will be initialed by the person making the entry. If a correction is made at a later time or date, the correction date is also entered. Unused partial logbook pages will be crossed out and signed and dated at the end of each workday.

The following information will be recorded in the logbook, as applicable:

- Author name and date
- Field instrument calibration methods, and identification number
- Chronology and locations of activities
- Names and affiliations of all on-site personnel or visitors

SAP Worksheet #14 -- Summary of Project Tasks (Continued)

- Weather conditions during the field activity
- Summary of daily activities and significant events
- Notes of conversations with coordinating officials
- Dates and times of sample collection, and name(s) of sampler(s)
- Sample identification numbers, volume collected, sampling method, and container (size/type) for each sample, including QC samples.
- References to other field logbooks or forms that contain specific information
- Discussions of problems encountered and their resolution
- Discussions of deviations from the SAP or other governing documents.

Sample processing techniques such as filtration, compositing, and preservation techniques should be noted in the logbook. Alternatively, this information may be contained on the chain-of-custody form, groundwater sampling form, or other field form. The logbook will then contain a unique identifier (e.g., chain of custody serial number) linking the field logbook entry to the field form.

Field logbooks must be permanently bound with pre-printed page numbers. Field logbooks must be signed and dated each day of field work. If logbook duties are transferred, the individuals relinquishing and receiving the logbook will both sign and date the logbook and record the transfer time.

Data Transfer and Transmittal

For the preparation of the Closure Report described in this SAP, ITSI Gilbane will summarize and evaluate the analytical results in laboratory reports, report results of field QA samples, and provide a Quality Control Summary Report (QCSR). Field data, including field notes copied from bound field logbooks or other media, field forms, boring logs, well completion forms, and field analysis results, will be presented in the report.

For samples collected and analyzed as part of this project, analytical results are required to be delivered in both hardcopy and electronic data deliverable (EDD) formats. An automated laboratory information management system (LIMS) must be used to produce the electronic copy. Manual generation of the electronic file (data entry by hand) is unacceptable. The laboratory will verify the electronic data files internally before they are issued. The electronic data will correspond exactly to the hard-copy data. No duplicate data will be submitted. Data will be delivered in a format compatible with NEDD and Geotracker, or applicable standards, as requested.

Assessment/Audit Tasks:

During project activities, ongoing assessments will include peer review, quality control reviews, audits of field operations, checks to see that project personnel have read appropriate planning documents and are following documented procedures, and reviews to ensure that clearance activities and preliminary work have been satisfactorily completed. Laboratory audits are not scheduled to occur in conjunction with this project; however, the Navy QAO may audit any part of the task, including the laboratory, at any time at its discretion.

SAP Worksheet #14 -- Summary of Project Tasks (Continued)

Data Review Tasks:
All analytical data generated by subcontract laboratories in support of this project will be reviewed internally by the laboratory prior to reporting, to assure the validity of reported data. This internal laboratory process will consist of data reduction and three levels of document review. As the analytical data are received by ITSI Gilbane, the Project Chemist will compare the generated data with project goals and objectives to ensure project DQOs can be met by the data. Data review and validation will also be performed by a third-party data validation service. The data will be validated as noted above.

Data storage, archiving, retrieval, and security will be managed as follows:

In conformance with Navy Environmental Work Instruction (EWI) #6, [Navy, 2005] data deliverables will be submitted electronically to the NIRIS system in NEDD format within 30 days after final data validation has been completed.

ITSI Gilbane will maintain electronic copies of all monitoring well purge and sampling forms, chain-of-custody forms, and all NEDDs. All data, field notes, raw analytical information, etc., will be stored in hardcopy and electronic format by ITSI Gilbane in a central project file for the period specified in the contract. ITSI Gilbane will also store the data electronically in project files on the company's main server, which is backed up externally on a secondary hard-drive system, which is also backed up on magnetic tape for long-term storage.

All relevant raw data and documentation, including (but not limited to) logbooks, data sheets, electronic files, and final reports, will be maintained by the fixed laboratory for at least five years. In conformance with specifications in Navy EWI #4, the hard copy of the analytical data will be delivered to the Navy's Administrative Record Department upon submittal of the final report. Data will be archived at ITSI Gilbane's office until this delivery to the Navy.

SAP Worksheet #15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment (confirmation sampling)
Analytical Groups: Metals, Pesticides, PCBs

Analyte	CAS Number	Project Action Limit[1] (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal[2] (mg/kg)	Laboratory-specific	
					QL (mg/kg)	MDL (mg/kg)
Zinc	7439-92-1	180	IR Site 25 ROD	80	2	0.21
Lead	7440-66-6	33	IR Site 25 ROD	18	2	0.27
Total DDT[3]	-	0.016	IR Site 25 ROD			
4,4'-DDT	50-29-3	-	-	0.0027	0.0013	0.00049
2,4'-DDT	789-02-6	-	-	0.0027	0.00067	0.000026
4,4'-DDE	72-55-9	-	-	0.0027	0.0013	0.0004
2,4'-DDE	3424-82-6	-	-	0.0027	0.00067	0.00006
4,4'-DDD	72-54-8	-	-	0.0027	0.0013	0.00034
2,4'-DDD	53-19-0	-	-	0.0027	0.00067	0.000036
Total PCBs[4]	1336-36-3	0.200	IR Site 25 ROD	0.100	0.034	0.017

Notes

[1] The Project Action Limit (PAL) is the site-wide average RG (lower bound) listed on Table 11 of the Record of Decision (Navy, 2009).

[2] Project quantitation limit s for analytes other than DDT were set to approximately one half the PAL. Because there are no RGs for the six individual DDTs that constitute the Total DDT, each individual PQL was set to the lower bound RG for Total DDT (0.016 mg/kg) divided by six = 0.0027 mg/kg.

[3] Total DDT is defined as the sum of detected concentrations of 2,4'-DDT, 4,4'-DDT, 2,4'-DDE, 4,4'-DDE, 2,4'-DDD and 4,4'-DDD. The sum of all DDT isomers cannot exceed the listed PAL.

[4] Total PCBs is defined as the sum of detected concentrations of Arochlors.

Abbreviations

MDL = method detection limit
mg/kg = milligrams per kilogram
QL = quantitation limit

SAP Worksheet #15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Fill material (on-site borrow characterization)
Analytical Groups: Metals, Pesticides, PCBs

Analyte	CAS Number	Project Action Limit[1] (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal[2] (mg/kg)	Laboratory-specific	
					QL (mg/kg)	MDL (mg/kg)
Zinc	7439-92-1	180	IR Site 25 ROD	90	2	0.21
Lead	7440-66-6	33	IR Site 25 ROD	16	2	0.27
Total DDT[3]	-	0.016	IR Site 25 ROD	-	-	-
4,4'-DDT	50-29-3	-	-	0.0027	0.0013	0.00049
2,4'-DDT	789-02-6	-	-	0.0027	0.00067	0.000026
4,4'-DDE	72-55-9	-	-	0.0027	0.0013	0.0004
2,4'-DDE	3424-82-6	-	-	0.0027	0.00067	0.00006
4,4'-DDD	72-54-8	-	-	0.0027	0.0013	0.00034
2,4'-DDD	53-19-0	-	-	0.0027	0.00067	0.000036
Total PCBs[4]	1336-36-3	0.200	IR Site 25 ROD	0.100	0.034	0.017

Notes

[1] The Project Action Limit (PAL) is the site-wide average RG (lower bound) listed on Table 11 of the Record of Decision (Navy, 2009).

[2] Project quantitation limit s for analytes other than DDT were set to approximately one half the PAL. Because there are no RGs for the six individual DDTs that constitute the Total DDT, each individual PQL was set to the lower bound RG for Total DDT (0.016 mg/kg) divided by six = 0.0027 mg/kg.

[3] Total DDT is defined as the sum of detected concentrations of 2,4'-DDT, 4,4'-DDT, 2,4'-DDE, 4,4'-DDE, 2,4'-DDD and 4,4'-DDD. The sum of all DDT isomers cannot exceed the listed PAL.

[4] Total PCBs is defined as the sum of detected concentrations of Arochlors.

Abbreviations

MDL = method detection limit
mg/kg = milligrams per kilogram
QL = quantitation limit

SAP Worksheet #16 -- Project Schedule /Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Anticipated Date(s) of Initiation	Anticipated Date of Completion	Deliverable	Deliverable Due Date
Prepare Internal Draft SAP	ITSI Gilbane	9 Sep 2010	11 March 2011	Internal Draft Work Plan/SAP	11 March 2011
Navy Review /Comment	Navy	15 March 2011	28 March 2011	Internal Draft Work Plan/SAP	none
Draft Work Plan (w/ SAP) to Regulators	ITSI Gilbane	28 March 2011	31 March 2011	Draft Work Plan	none
Regulator Review/Comment	RWQCB	31 March 2011	29 April 2011	Draft Work Plan	6 January 2011
Prepare Final SAP	ITSI Gilbane	29 April 2011	15 May 2011	Final Work Plan/SAP	17 January 2011
Field Work	ITSI Gilbane	01 Sept 2011	01 Nov 2011	Analytical Data	none
Preparation of Draft RA Completion Report	ITSI Gilbane	01 November 2011	15 January 2012	Draft RA Completion Report for IR Site 25	none
Preparation of Final RA Completion Report	ITSI Gilbane	01 March 2012	01 June 2012	Draft RA Completion Report for IR Site 25	12 June 2012

SAP Worksheet #17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

Sampling locations are shown on Figures SP-1 through SP-10. The number and frequency of samples to be collected are listed in Worksheet #18.

Through previous work at the site, Navy has identified Site areas in need of remediation to achieve the RGs (ROD, Navy 2009). During this previous work (performed in years past, prior to ITSI Gilbane's involvement at the Site), IR Site 25 was divided into polygon areas based on previous sampling locations. The size of each area was calculated using the Thiessen polygon method of interpolation.

(The Thiessen polygons used in the IR Site 25 sampling design were originally designed by the Navy nearly 10 years ago during the remedial investigation phase, and augmented recently during the pre-design investigation. The approach was documented in the ROD (Navy, 2009). This method is a recognized area-weighted method by which each polygon contains all the area that is closer to a given sample point than to any other sample point. The approach is commonly applied to characterize sediment sites with complex interactions at site boundaries, e.g., levees, and with patchy areas of elevated chemical concentrations (San Diego Region Water Board, 2001; USEPA, 1991, 2000).)

Thiessen polygons were then mapped around individual sampling locations so that the sides of each polygon were equidistant from adjacent sampling locations. Concentrations of COECs detected in sediment from a sampling location were assumed to represent all sediment with the polygon. If concentrations of one or more COECs in a given polygon were above the do-not-exceed remediation goal, then that polygon was identified for remediation (ROD, Navy, 2009).

Confirmation sediment samples for regulatory verification will be collected from each excavation bottom (discrete) and sent to the laboratory to be analyzed for total DDT, lead, total PCBs, and zinc. Samples will be collected in the excavator bucket after the excavator operator safely stages the bucket on ground surface for the field technician’s safety and convenience. Samples will be taken from sediments that have not come into contact with the blade or sides of the bucket. Samples will be immediately labeled and packaged for transport to the selected off-site DoD ELAP-certified analytical laboratory. In excavations that are dry and are easily and safely accessed by our Field Sampling Technicians, ITSI will collect samples with a stainless steel spoon, stainless steel push tube, disposable wooden or plastic scoop, or hand auger.

Excavation Sediment Sampling Procedure

Sediment confirmation samples will be collected from the open excavations resulting from the removal action from the excavation bottoms. The polygons to be excavated have been segregated into three groups, corresponding to small, medium, and large areas thusly:

Group 1--less than 10,000 square feet (SF);
Group 2--10,000 SF to less than 40,000 SF; and
Group 3--40,000 SF and greater.

SAP Worksheet #17 -- Sampling Design and Rationale (cont'd)

(UFP-QAPP Manual Section 3.1.1)

Group 1 areas will have one confirmation sample collected from the excavation footprint; Group 2 areas will have two; and Group 3 areas will have four or more. For most areas, this approach correlates to a frequency of about 1 sample per 100 foot X 100 foot area. Sampling locations are shown on Figures SP-1 through SP-10. Sample containers will be appropriately labeled and stored in an insulated container with ice. See SOP PR-TC-02040101 for Sample Handling Procedures.

Clean Fill Material Sampling Procedure

Sample collection to confirm that clean fill (on-site "borrow" sediment to be used for backfill) will conform to COECs will be performed at a frequency of 1 sample per 500 cubic yards (CY). The volume of borrow material to be characterized is 20,000 CY; therefore 40 borrow area samples will be collected. A rectangular sample grid will be established; each sample will be collected at a depth interval of 3-9 inches and submitted for analysis as described in Work Sheet #15.

Sample Preservation Procedures

To preserve sample quality after sample collection up to the time of sample analysis, all samples with be properly preserved in accordance with the applicable preservation technique specified in the relevant analytical method. Pre-cleaned sample containers from the analytical laboratory containing the appropriate preservative will be used. Preservation requirements are listed on Worksheet #19. All analytical methods for the project also require samples to be stored at 4±2 °C; this requirement will be met by shipping samples in a cooler with sufficient ice to keep samples cool until the samples are received at the laboratory.

Equipment Cleaning and Decontamination Procedures

To prevent cross-contamination, sampling technicians will decontaminate all reusable sampling equipment that may come into contact with sediment before its initial use on the site and before each subsequent use. Water to be used for rinsing will be analyzed for all target analytes at the beginning of the field program to verify that target analytes are not present above the practical quantitation limit.

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling location/ID number	*Matrix*	*Depth*[1] (inches)	*Analytical group*[2]	*Number of samples*	*Sampling SOP reference*
Confirmation Sampling, IR Site 25, Moffett Field					
A1.1-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A2.1-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A2.1-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A3.1-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A3.1-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN05	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN06	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN07	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN08	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN09	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.1-CN010	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.2-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.2-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.3-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.3-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.4-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.5-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A4.5-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A5.1-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A5.1-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A5.1-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A5.1-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A5.2-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A5.2-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A5.2-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A5.2-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling location/ID number	*Matrix*	*Depth*[1] (inches)	*Analytical group*[2]	*Number of samples*	*Sampling SOP reference*
A5.2-CN05	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A5.2-CN06	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.1-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.1-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.1-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.1-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.2-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.2-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.2-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.2-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.3-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.3-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.4-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A6.5-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.1-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.1-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.1-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.1-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.2-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.2-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.3-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.3-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.3-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.3-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.4-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.4-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.5-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.5-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.6-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.6-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.7-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.8-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.8-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.9-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.9-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling location/ID number	*Matrix*	*Depth*[1] (inches)	*Analytical group*[2]	*Number of samples*	*Sampling SOP reference*
A8.10-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.11-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.12-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.12-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.13-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.14-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.15-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.15-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.6-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.17-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.18-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.19-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.20-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.21-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.22-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.23-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.24-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.25-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A8.25-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.1-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.1-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.1-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.1-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.2-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.3-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.3-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.4-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.5-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.5-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.6-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.6-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.7-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.8-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling location/ID number	*Matrix*	*Depth*[1] (inches)	*Analytical group*[2]	*Number of samples*	*Sampling SOP reference*
A9.8-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.10-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.11-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.12-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A9.12-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.1-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.1-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.2-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.2-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.3-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.4-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.5-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.6-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.7-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.8-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.9-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.10-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.11-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.12-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.13-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.14-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.15-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.16-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.17-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.18-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.19-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.20-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.21-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.22-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.23-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.24-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.25-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.26-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.27-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.28-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling location/ID number	*Matrix*	*Depth*[1] (inches)	*Analytical group*[2]	*Number of samples*	*Sampling SOP reference*
A10.29-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.30-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.31-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.32-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.33-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.34-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.35-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.36-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.37-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.38-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.39-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.40-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.41-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.42-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.42-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.42-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.42-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.42-CN05	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.43-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.44-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.45-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.45-CN02	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.45-CN03	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.45-CN04	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.46-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.47-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.48-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.49-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.50-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.51-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.52-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.53-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.54-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.55-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.56-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling location/ID number	*Matrix*	*Depth*[1] (inches)	*Analytical group*[2]	*Number of samples*	*Sampling SOP reference*
A10.57-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.58-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.59-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.60-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
A10.61-CN01	S	0-6	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
Characterization of On-Site Borrow Material, IR Site 25, Moffett Field					
BA-1	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-2	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-3	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-4	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-5	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-6	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-7	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-8	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-9	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-10	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-11	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-12	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-13	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-14	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-15	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-16	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-17	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-18	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-19	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-20	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-21	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-22	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-23	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-24	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-25	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling location/ID number	*Matrix*	*Depth*[1] (inches)	*Analytical group*[2]	*Number of samples*	*Sampling SOP reference*
BA-26	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-27	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-28	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-29	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-30	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-31	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-32	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-33	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-34	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-35	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-36	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-37	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-38	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-39	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17
BA-40	S	3-9	Lead, Zinc, DDTs, PCBs	1	See Worksheet #17

Notes:

[1] Sediment confirmation samples (CN designation) to be taken at the surface remaining after excavation.

[2] DDTs and PCBs are defined in Worksheet #15.

S = sediment

SAP Worksheet #19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method // SOP Reference	Containers (number, size, and type)	Sample volume[1] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[2] (preparation / analysis)
Sediment	Metals (Pb, Zn)	EPA 6010B/6020/ prep EPA 3050B // met003_5_ICPAES	4-oz Jar with Teflon-lined lid	5g	None	Analysis –180 days
Sediment	Polychlorinated biphenyls (PCBs)	EPA 8082; prep EPA 3550B // EGC8082-12	8-oz Jar with Teflon-lined lid	50 g	Cool at 4±2 ^{o}C	Extraction – none Analysis – 40 days
Sediment	Six DDT isomers	EPA 8081A; prep EPA 3550B // EGC8081-15	8-oz Jar with Teflon-lined lid	50 g	Cool at 4±2 ^{o}C	Extraction – None Analysis – 40 days

[1] Minimum sample volume or mass requirement if different from the container volume.

[2] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

SAP Worksheet #20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of PT Samples	Total No. of Samples to Lab
Sediment	(ALL)	170	0	18 (9 pairs)	0	0*	0	0	188

Notes:

MS/MSD = matrix spike/matrix spike duplicate (in this context, a "pair" means 2 samples, consisting of one MS and one MSD.)

* The exclusive use of disposable equipment is intended to obviate the need for these blanks. However, should circumstances necessitate the need for re-usable equipment, then these blanks will be collected at a frequency of one per day of sampling.

SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
None	NA	NA	NA	NA	NA	NA	NA

NA= not applicable

SAP Worksheet #23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
met003_5	Metals by ICP-AES, 10-8-2010	Definitive	Sediment/metals	ICP/AES	Accutest	N
EGC8082-12	Determination of PCBs Using GC System, 5/24/2010	Definitive	Sediment/Total PCBs	Gas chromatograph	Accutest	N
EGC8081-15	Determination of Organochlorine Pesticides Using GC System, 8/4/2010	Definitive	Sediment/DDT isomers	Gas chromatograph	Accutest	N

SAP Worksheet #24 -- Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Inductively coupled plasma (ICP), ICP-AES	Tune (ICPMS only)	Daily prior to analysis and every 12 hours (ICPMS only)	Refer to criteria listed in the method	Retune instrument and verify	Laboratory technician	MET100-11 AES226-04
	Initial Calibration, 1-point plus a blank minimum (ICAL)	Daily, prior to analyses; re-analyze upon failure of ICV or CCV	$r \geq 0.995$	(1) Evaluate system (2) Recalibrate	Laboratory technician	
	Initial Calibration Verification (ICV)	After calibration, prior to analysis	90-110% recovery for ICV	(1) Evaluate system (2) Reanalyze ICV (3) Recalibrate (4) Reanalyze affected samples	Laboratory technician	
	Continuing Calibration Verification (CCV)	Every ten samples, and at end of run	90-110% recovery for CCV	(1) Evaluate system (2) Reanalyze CCV (3) Recalibrate (4) Reanalyze affected samples	Laboratory technician	
	Calibration Blank	After every calibration verification	No analytes detected ≥ RL	Perform maintenance as described on WS #25, then re-analyze calibration blank and previous 10 samples	Laboratory technician	

SAP Worksheet #24 -- Analytical Instrument Calibration Table (Cont'd)

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Gas Chromatograph	Initial Calibration, 5-point minimum (ICAL)	Initially and as required	%RSD $\leq$ 20% or r $\geq$ 0.995	(1) Evaluate system (2) Recalibrate	Laboratory technician	SV001_5 LA-2/LA-9
	Method blanks and instrument blanks	After initial calibration	No target analytes present above ½ RL	(1) Reanalyze blank (2) Clean system (3) Reanalyze affected samples	Laboratory technician	
	Continuing Calibration Verification (CCV)	Every 10 injections and at beginning and end of sequence	85-115% recovery	(1) Evaluate system (2) Reanalyze standard (3) Recalibrate (4) Reanalyze affected samples	Laboratory technician	

Notes:

CCC = Calibration Check Compounds

SPCC = System Performance Check Compounds

GC/MS = Gas Chromatography / Mass Spectrometry

m/z = mass-to-charge ratio

RL = Reporting Limit

RF = Response Factor

%RSD = Percent Relative Standard Deviation

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Activity (Maintenance / Testing / Inspection)	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Gas Chromatograph (GC)	**Maintenance:** Check/replace any applicable: liner insert, column, septa, syringe, glass wool plug, thermal traps	As needed	Method QA/QC requirements are met	Replace parts and retune or rerun standards	Laboratory technician	SV001_5
	Maintenance: Check/replace gas drying and purifying cartridges	When indicated to be necessary	No color change visible in indicating traps or non-indicating traps less than 6 to 12 mo. old	Replace non-indicating traps every six to 12 mo. or when indicating traps start to change color	Laboratory technician	
	Inspection: Oven performance	Daily, as part of retention time check of standards	Properly functioning oven	Repair oven and retune / rerun standards	Laboratory technician	
	Testing: Surrogate Standards	Every sample spiked sample, standard, and method blank	Advisory QC acceptance criteria per method specification or laboratory statistically established limits	Troubleshoot & remedy problem, then re-extract, and re-analyze all affected samples	Laboratory technician	
	Testing: MDL Study	For each analytical system; once per 12-month period and after every major repair	Detection limits established will be at least two times below the RLs	Perform Instrument Maintenance (if necessary) / Re-extract MDL Study (if necessary) , re-run the MDL study	Laboratory technician	

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table (continued)
(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Activity (Maintenance / Testing / Inspection)	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICP	**Inspection:** Pumps and tubing inspection	Daily	Properly functioning pumps and tubing	Repair or replace pumps and tubing	Laboratory technician	MET100-11
	Inspection: Torch and injector	Daily	Intensity within 20% of expected value	Clean or replace	Laboratory technician	
	Inspection: Nebulizer	Daily	Acceptable Response	Clean or replace	Laboratory technician	
	Maintenance: Autosampler tracks	Every six months	None	Wipe the tracks with a Kim-Wipe saturated with 1-in-3 or clear oil.	Laboratory technician	
	Testing: QC Check - Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	At initiation of analysis on each instrument and for each analyst	Average recovery and standard deviation must be within limits specified in the analytical method for each compound	Troubleshoot analytical system, repeat test.	Laboratory technician	
	Testing: QC Check - IDL study	Once per 3 month period	No official criteria. IDLs will be below the MDLs.	Perform inspection and maintenance, repeat IDL study.	Laboratory technician	
	Testing: QC Check - MDL study (water only)	Once per 12 month period	No official criteria. MDLs will be below the RLs.	Perform inspection and maintenance, repeat IDL study	Laboratory technician	

SAP Worksheet #26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): sample technician / ITSI Gilbane
Sample Packaging (Personnel/Organization): Project Task Manager / ITSI Gilbane
Coordination of Shipment (Personnel/Organization): Project Task Manager / ITSI Gilbane
Type of Shipment/Carrier: Overnight shipping service such as FedEx or Laboratory Courier
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Laboratory receipt clerk / Accutest
Sample Custody and Storage (Personnel/Organization): Laboratory technician or custodian /Accutest
Sample Preparation (Personnel/Organization): Laboratory technician / Accutest
Sample Determinative Analysis (Personnel/Organization): Laboratory manager / Accutest
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 30 Days
Sample Extract/Digestate Storage (Number of days from extraction/digestion): 30 Days
Biological Sample Storage (Number of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Laboratory technician / Accutest
Number of Days from Analysis: 30 Days, or as requested by client

Notes:
NA = Not applicable
TBD: To be determined. The laboratory has not yet been selected. These fields will be completed prior to submission of the Final SAP, and submitted to the Navy for approval prior to implementation of field activities.

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SAP Worksheet #27 -- Sample Custody Requirements Table
(UFP-QAPP Manual Section 3.3.3)

Sample Labeling System:
Samples will be labeled systematically, using the following sample IDs as examples:

- A1.1-CN01, where
  - A1.1 is the identifier for the polygon area in which the sample is located (see Figure SP-1).
  - CN indicates a confirmation sample.
  - 01 the sequential sample number for a given polygon and will serve as a unique identifier for the sample.

Unique sample numbers will be used to designate samples and sample locations, and these identifiers will be used for coding, tracking, and reporting chemical data. Each sample number will be unique within the project, will be traceable to a specific sampling event, and will not obviously indicate the type of sample (e.g., field sample, field blank). Chemical data produced by the contract laboratory will be reported using the sample numbers as identified on the chain-of-custody forms.

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):
Standardized sample custody procedures will be followed from sample collection, through transfer, storage, and analysis, to ultimate disposal. Sample custody begins with shipment of the empty sample containers from the laboratory to the office or site. Sample containers will be shipped from the laboratory in sealed containers with appropriate seals and custody information. All sample containers will be properly labeled, and collected samples will be monitored for temperature control in the field and during laboratory transport and storage. Temperature blanks will be used in all coolers containing samples requiring preservation at reduced temperature. Samples will always be accompanied by a chain-of-custody record. When samples are transferred, both the individual relinquishing and the individual receiving the samples will sign, date, and note the transfer time on the chain of custody record. Samples will be packaged for shipment with completed sample labels for each sample container, sample containers carefully packed upright and on ice, and with a chain of custody record in a Ziploc™ bag.

Custody seals will be used when samples are shipped via commercial courier service, and must be placed on the cooler so that the seals have to be broken before the cooler can be opened. The seals must be signed and dated by the field personnel. Samples may be hand-delivered to the laboratory, transported by commercial or laboratory couriers, or shipped to the laboratory using an overnight shipper.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):
A designated laboratory sample custodian will accept custody of the samples and verify that the information on the sample labels matches that on the chain-of-custody form(s). Pertinent information as to sample condition, shipment, pickup, and courier will also be checked on the chain-of-custody form. The temperature inside the cooler and of the temperature blank will be measured immediately after the cooler is opened, and the results will be recorded. Information on the date and time of receipt, method of shipment, and sample condition will also be recorded. The custodian will then

SAP Worksheet #27 -- Sample Custody Requirements Table (Continued)

enter the appropriate data into the laboratory sample tracking system. The sample custodian will use the sample numbers on the sample labels for tracking and also assign a unique laboratory number to each sample. The custodian will then transfer the samples to the proper analyst(s) or store the samples in the appropriate secure area. Data sheets and laboratory records will be retained by the laboratory as part of the permanent documentation for a period of at least 3 years. Samples and extracts will be retained by the analytical laboratory for a minimum of 30 days after the laboratory reports the data. Unless notified otherwise by the site managers, excess or unused samples may be disposed of by the laboratory in a manner consistent with local government regulations.

Chain-of-Custody Procedures:

A chain-of-custody form will be completed for every group of samples sent to the analytical laboratory, to document sample possession from the time of collection to sample receipt by the laboratory; and a copy of the form will accompany the shipment. Each completed chain-of-custody form will contain the following information: sample identification number(s); name(s) and signature(s) of collectors, samplers, or recorders; ITSI Gilbane project number, project name, and location of project; the project manager's name and contact information; the date and time of collection; sample type(s) and analyses requested; and signatures of persons relinquishing and receiving the samples. When samples are transferred, the individuals relinquishing and receiving the samples will sign, date, and note the transfer time on the chain-of-custody form.

NOTE: Example field forms including a chain-of-custody form and sample collection log are included in Attachment 1.

SAP Worksheet #28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Sediment
Analytical Group	Metals
Analytical Method / SOP Reference	6010B / met003_5

QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	1/20 of samples analyzed	All analytes < reporting limit (RL)	Evaluate system. Flag data as specified in EPA National Functional Guidelines	Laboratory technician	Accuracy/Bias of lab system	All analytes < RL
Matrix Duplicate (*)	1/20 of samples analyzed (*)	Relative percent difference (RPD) < 20 %	Perform additional QC tests as stated in Method	Laboratory technician	Accuracy/Bias (matrix interference); Precision of lab system	RPD < 20 %
Laboratory Control Sample (LCS)	1/20 of samples analyzed	Percent Recovery 80 – 120%	Reanalyze LCS once. If acceptable, report acceptable data only. If unacceptable, all samples analyzed after prior acceptable LCS will be reanalyzed	Laboratory technician	Accuracy/Bias of lab system	Percent Recovery 80 – 120%
Matrix Spike/ (MS) Matrix Spike Duplicate (MSD) (*)	1/20 of samples analyzed (*)	Percent Recovery 75 – 125% RPD < 20 %	Perform additional QC tests as stated in Method	Laboratory technician	Accuracy/Bias (matrix interference); Precision of lab system	Percent Recovery 75 – 125% RPD < 20 %

(*) Matrix Duplicate and MS/MSD pairs are to be prepared for each analytical batch (up to 20 samples)

SAP Worksheet #28 -- Laboratory QC Samples Table (continued)
(UFP-QAPP Manual Section 3.4)

Matrix	Sediment
Analytical Group	Polychlorinated Biphenyls
Analytical Method / SOP Reference	8082 / EGC8082-12

QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	1/20 of samples analyzed	All analytes < reporting limit (RL)	Evaluate system. Flag data as specified in EPA National Functional Guidelines	Laboratory technician	Accuracy/Bias of lab system	All analytes < RL
Laboratory Control Sample (LCS)	1/20 of samples analyzed	Percent Recovery 55-115 %	Reanalyze LCS once. If acceptable, report acceptable data only. If unacceptable, all samples analyzed after prior acceptable LCS will be reanalyzed	Laboratory technician	Accuracy/Bias of lab system	Percent Recovery 55-115 %
Matrix Spike/ (MS) Matrix Spike Duplicate (MSD) (*)	1/20 of samples analyzed (*)	Percent Recovery 50-115% RPD < 20 %	Evaluate system. Rerun MS/MSD if warranted.	Laboratory technician	Accuracy/Bias (matrix interference); Precision of lab system	Percent Recovery 50-115% RPD < 20 %
Surrogates/ Internal Standards	Every standard and sample	Percent Recovery 50-150%	Reanalyze sample.	Laboratory technician	Accuracy/Bias of lab system	Percent Recovery 50-150%

(*) Matrix Duplicate and MS/MSD pairs are to be prepared for each analytical batch (up to 20 samples)

SAP Worksheet #28 -- Laboratory QC Samples Table (continued)
(UFP-QAPP Manual Section 3.4)

Matrix	Sediment
Analytical Group	total DDTs
Analytical Method / SOP Reference	8081A / EGC8081-15

QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	1/20 of samples analyzed	All analytes < reporting limit (RL)	Evaluate system. Flag data as specified in EPA National Functional Guidelines	Laboratory technician	Accuracy/Bias of lab system	All analytes < RL
Laboratory Control Sample (LCS)	1/20 of samples analyzed	Percent Recovery 55-115 %	Reanalyze LCS once. If acceptable, report acceptable data only. If unacceptable, all samples analyzed after prior acceptable LCS will be reanalyzed	Laboratory technician	Accuracy/Bias of lab system	Percent Recovery 55-115 %
Matrix Spike/ (MS) Matrix Spike Duplicate (MSD) (*)	1/20 of samples analyzed (*)	Percent Recovery 50-115% RPD < 20 %	Evaluate system. Rerun MS/MSD if warranted.	Laboratory technician	Accuracy/Bias (matrix interference); Precision of lab system	Percent Recovery 50-115% RPD < 20 %
Surrogates/ Internal Standards	Every standard and sample	Percent Recovery 50-150%	Reanalyze sample.	Laboratory technician	Accuracy/Bias of lab system	Percent Recovery 50-150%

(*) Matrix Duplicate and MS/MSD pairs are to be prepared for each analytical batch (up to 20 samples)

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Field notes/logbook	Project file
Chain-of-custody forms	Project file (copy follows samples)
Laboratory raw analytical data	NAVFAC SW Administrative Record; Copy on CD in ITSI Gilbane Project file; laboratory electronic database
Paper copy of all project agency-deliverable documents (e.g., work plans, SAPs, remediation reports, Health and Safety Plans, etc.) submitted to regulatory agencies	NAVFAC SW Administrative Record
Audit/assessment checklists/reports	Project file and laboratory (if applicable)
Corrective action forms/reports	Project file and laboratory (if applicable)
Laboratory equipment calibration logs	Laboratory
Sample preparation logs	Laboratory
Run logs	Laboratory
Sample disposal records	Laboratory
Validated data	Project file; electronic deliverable to Navy database (NIRIS); NAVFAC SW Administrative Record

SAP Worksheet #30 -- Analytical Services Table
(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Sediment	Metals (Pb, Zn)	As Listed on WS #18	6010B	10 Working Days	Accutest N. California 2105 Lundy Avenue San Jose, CA 95131 Sue Bell (813) 741-3338	Curtis and Tompkins, LTD 2323 Fifth Street Berkeley, CA 94710 Mike Dahlquist (510)204-2225
Sediment	Polychlorinated biphenyls (PCBs)	As Listed on WS#18	8082	10 Working Days	Accutest N. California 2105 Lundy Avenue San Jose, CA 95131 Sue Bell (813) 741-3338	Curtis and Tompkins, LTD 2323 Fifth Street Berkeley, CA 94710 Mike Dahlquist (510)204-2225
Sediment	total DDTs	As Listed on WS#18	8081A	15 Working Days	Accutest N. California 2105 Lundy Avenue San Jose, CA 95131 Sue Bell (813) 741-3338	Curtis and Tompkins, LTD 2323 Fifth Street Berkeley, CA 94710 Mike Dahlquist (510)204-2225

Note:

All samples will be sent to Accutest, who will be responsible for any subcontracting of specialty analyses. The selected laboratories are certified by the State of California and are DoD ELAP accredited.

SAP Worksheet #31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organization)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Audits	Annually; at least once per project; with discretionary follow-ups	Internal	ITSI Gilbane	Ray Spencer, PQCM (ITSI Gilbane) or Rich Flynn, Program Chemist (ITSI Gilbane)	Gail Jones, Site Coordinator (ITSI Gilbane)	Ray Spencer, PQCM (ITSI Gilbane) Gail Jones, Site Coordinator (ITSI Gilbane)	Ray Spencer, PQCM (ITSI Gilbane) Jim Schollard, QCPM (ITSI Gilbane)
Field Documen-tation Review	Quarterly	Internal	ITSI Gilbane	Ray Spencer, PQCM (ITSI Gilbane)	Gail Jones, Site Coordinator (ITSI Gilbane)	Ray Spencer, PQCM (ITSI Gilbane) Gail Jones, Site Coordinator (ITSI Gilbane)	Ray Spencer, PQCM (ITSI Gilbane) Jim Schollard, QCPM (ITSI Gilbane)
Field Sampling Technical Systems Audit	At start of field activities	Internal	ITSI Gilbane	Ray Spencer, PQCM (ITSI Gilbane) or Jim Schollard, QCPM (ITSI Gilbane)	Gail Jones, Site Coordinator (ITSI Gilbane) or Robert Lindfors, PM (ITSI Gilbane)	Ray Spencer, PQCM (ITSI Gilbane); Jim Schollard, QCPM (ITSI Gilbane) Gail Jones, Site Coordinator (ITSI Gilbane) and Robert Lindfors, PM (ITSI Gilbane)	Ray Spencer, PQCM (ITSI Gilbane) Jim Schollard, QCPM (ITSI Gilbane)
Field Readiness	At start of field activities	Internal	ITSI Gilbane	Ray Spencer, PQCM (ITSI Gilbane) or Jim Schollard, QCPM (ITSI Gilbane)	Robert Lindfors, PM (ITSI Gilbane)	Ray Spencer, PQCM (ITSI Gilbane); Jim Schollard, QCPM (ITSI Gilbane) Robert Lindfors, PM (ITSI Gilbane)	Ray Spencer, PQCM (ITSI Gilbane) Jim Schollard, QCPM (ITSI Gilbane)

SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Time-frame of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Time-frame for Response
SAP implementation (field issues)	Non-Routine Occurrence Report; Nonconformance Report; Corrective Action Report	Robert Lindfors, PM (ITSI Gilbane); Ray Spencer, PQCM (ITSI Gilbane); Rich Flynn, Program Chemist (ITSI Gilbane); Bryce Bartelma, RPM, (Navy [if issue is major]); Joseph Michalowski, Acting QAO, (Navy)	48 hours to 5 days, depending upon nature of deficiency	Corrective Action Report Form	Ray Spencer, PQCM (ITSI Gilbane); Jim Schollard, QCPM (ITSI Gilbane), or Bryce Bartelma, RPM, (Navy), as required	48 hours to 5 days, depending upon nature of deficiency
SAP implementation (laboratory issues)	Laboratory case narrative; Laboratory variance request; Non-Routine Occurrence Report; Nonconformance Report; Corrective Action Report	Robert Lindfors, PM (ITSI Gilbane); Ray Spencer, PQCM (ITSI Gilbane); Kristen Carlyon, Project Chemist (ITSI Gilbane); Bryce Bartelma, RPM, (Navy [if issue is major]) Joseph Michalowski, Acting QAO, (Navy)	48 hours to 5 days, depending upon nature of deficiency	Corrective Action Report Form (major deficiencies); otherwise, corrective action is documented on the original deficiency document	Ray Spencer, PQCM (ITSI Gilbane); Jim Schollard, QCPM (ITSI Gilbane); Richard Flynn, Program Chemist (ITSI Gilbane); Robert Lindfors, PM (ITSI Gilbane); Bryce Bartelma, RPM, (Navy), as required	48 hours to 5 days, depending upon nature of deficiency
Field Sampling Technical Systems Audit	Field Change Request; Non-Routine Occurrence Report; Nonconformance Report; Corrective Action Report	Robert Lindfors, PM (ITSI Gilbane); Ray Spencer, PQCM (ITSI Gilbane); Jim Schollard, QCPM (ITSI Gilbane); Bryce Bartelma, RPM, (Navy [if issue is major])	24 hours to 5 days, depending upon nature of deficiency	Corrective Action Report form (major deficiencies); otherwise, corrective action is documented on the original deficiency document	Jim Schollard, QCPM (ITSI Gilbane); Robert Lindfors, PM (ITSI Gilbane); Bryce Bartelma, RPM, (Navy), as required	Critical deficiencies-response within 5 days; all other findings-15 days from report

SAP Worksheet #33 -- QA Management Reports Table
(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Field Sampling Technical Systems Audit Report	Once, at startup of sampling	30 days after systems audit	Ray Spencer, PQCM (ITSI Gilbane)	Robert Lindfors, Project Manager (ITSI Gilbane); Jim Schollard, QCPM (ITSI Gilbane); Bryce Bartelma, RPM (Navy)
Data Review Reports	One per data sample delivery group (SDG)	90 days after sampling is completed (component of remediation report)	Evin McKinney, Synectics	Richard Flynn, Program Chemist (ITSI Gilbane); Bryce Bartelma, RPM (Navy)
Quality Control Summary Report (QCSR)	Once	90 days after sampling is completed (component of remediation report)	Kristen Carlyon, Project Chemist (ITSI Gilbane)	Richard Flynn, Program Chemist (ITSI Gilbane); Bryce Bartelma, RPM (Navy)
Monthly Status Report	Monthly throughout project	Monthly throughout project	Robert Lindfors, Project Manager (ITSI Gilbane)	Bryce Bartelma, RPM (Navy); Jim Schollard, QCPM (ITSI Gilbane)
Final Project Report	Once at completion of project	60 days after completion of project field activities	Robert Lindfors, Project Manager (ITSI Gilbane)	Bryce Bartelma, RPM (Navy); Jim Schollard, QCPM (ITSI Gilbane)

SAP Worksheet #34 -- Verification (Step I) Process Table
(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-custody forms	Chain-of-custody forms will be reviewed internally in the field upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the chain-of-custody will be initialed by the reviewer. A copy of the chain-of-custody will be retained in the project file, and the original and remaining copies will be taped inside the cooler for shipment.	I	Ray Spencer, PQCM (ITSI Gilbane) Second-level review by Richard Flynn, Project Chemist (ITSI Gilbane)
Audit reports	A copy of each audit report will be placed in the project file when the report is completed. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the project file. At the beginning of each week, and at the completion of the site work, project file audit reports will be reviewed internally to ensure that all appropriate corrective actions have been taken and that corrective action reports are attached. If corrective actions have not been taken, the project manager will be notified to ensure that action is taken.	I	Ray Spencer, PQCM (ITSI Gilbane) Scott Lovesy, Site Superintendent (ITSI Gilbane) Robert Lindfors, PM (ITSI Gilbane)
Field notes/logbook	Field notes will be reviewed internally, at intervals as needed during the project and at the completion of the work, and placed in the project file. A copy of the field notes will be attached to the final report.	I	Gail Jones, Site Coordinator (ITSI Gilbane) Ray Spencer, PQCM (ITSI Gilbane)
Laboratory data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All data packages will be verified externally (according to the data validation procedures detailed in Worksheet #35).	I, E	Richard Flynn, Project Chemist (ITSI Gilbane) Evin McKinney, Synectics
Electronic Data Deliverables	EDDs are to be internally verified by the laboratory performing the analytical work prior to submittal. EDDs will be verified externally with respect to the hardcopy of the data.	I, E	Dolores Queka, Accutest Evin McKinney, Synectics

SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table

(UFP-QAPP Manual Section 5.2.2)

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Methods	Verify lab is approved by State of California/DoD ELAP and is using analytical procedures which comply with EPA approved standard methods.	Rich Flynn, Project Chemist (ITSI Gilbane)
IIa	Performance requirements	Verify Lab method SOPs are sufficient to satisfy PQOs.	Rich Flynn, Project Chemist (ITSI Gilbane)
IIa	Sampling locations, number of samples	Verify that sample locations and quantities will be sufficient to satisfy PQOs.	Robert Lindfors, Project Manager (ITSI Gilbane)
IIa	List of project-specific analytes	Verify Lab compound list includes all compounds of concern	Rich Flynn, Project Chemist (ITSI Gilbane)
IIa	Chain of custody	Examine chain-of-custody forms for completeness and accuracy.	Rich Flynn, Project Chemist (ITSI Gilbane)
IIa	Sample integrity	Verify samples were properly handled, stored, and processed within method parameters, and within EPA recommended hold times.	Rich Flynn, Project Chemist (ITSI Gilbane)
IIa	SAP	Ensure SAP is compliant with the Contract.	Rich Flynn, Program Chemist (ITSI Gilbane)
IIb	Deviations/ variances	Determine impacts of any deviations or variances from methods to ensure PQOs are met.	Rich Flynn, Project Chemist (ITSI Gilbane)
IIb	Project Quantitation Limit	Ensure that PQLs are achieved as outlined in the QAPP and that the laboratory successfully analyzed a standard at the QL.	Rich Flynn, Project Chemist (ITSI Gilbane)
IIb	QC samples	Ensure that a sufficient number of QC samples are analyzed as outlined in the QAPP to meet PQOs.	Rich Flynn, Project Chemist (ITSI Gilbane)
IIb	Electronic Data Deliverables	Verify that acceptable EDDs have been uploaded to NIRIS and Geotracker if appropriate.	Kimberly Tom, Data Manager (ITSI Gilbane)
IIb	Data usability	Summarize data quality and usability in QCSR	Rich Flynn, Project Chemist (ITSI Gilbane)

SAP Worksheet #36 –Analytical Data Validation (Steps IIa and IIb) Summary Table

(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	**Data Validator** (title and organizational affiliation)
IIa	Sediment	(All)	In accordance with Lab SOP; EPA National Functional Guidelines; DoD QSM	Dolores Queka, Accutest Laboratory QC Officer; Evin McKinney, Synectics
IIb	Sediment	(All)	In accordance with PQOs, and with 3rd party data validators; EWI #1	Rich Flynn, Project Chemist (ITSI Gilbane); Evin McKinney, Synectics

Notes:

Third-party data validation will be conducted in compliance with EWI #1. Moffett Naval Air Station is on the EPA NPL Site List, thus the data will be validated at 80% EPA Level III and 20% EPA Level IV. For Level III data validation, the following items are reviewed:

- Completeness
- Chain of custodies and case narrative
- Holding times and preservation
- Blanks
- Lab QC
- Field QC
- Surrogates and internal standards, where applicable
- Initial and continuing calibrations
- Instrument performance checks

Level IV review will include all of the above, in addition to the following:

- Review of raw data
- Calculation checks of quantified analytical data and QC samples

SAP Worksheet #37 -- Usability Assessment
(UFP-QAPP Manual Section 5.2.3)

A variety of analytical and statistical control parameters will be used during analysis of samples to assess data usability. Analytical results will be evaluated by the project team in accordance with precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters to ensure the attainment of the project-specific DQOs. Of these PARCC parameters, precision and accuracy will be evaluated through the collection of the QC samples listed in Worksheet #20. Precision and accuracy goals for these QC samples are listed in Worksheets #12 and #28.

Contract Laboratory QC Check Samples
Laboratory QC samples consist of method blanks, LCS, MS/MSD samples, surrogates, and laboratory duplicates. All samples will be spiked with surrogate compounds where recommended or required by the method. A method blank, LCS, and laboratory MS/MSD will be analyzed for each analytical batch.

Detection and Quantitation Limits
The method detection limit (MDL) is the minimum quantity of an analyte that can be reliably distinguished from background noise for a specific analytical method. The MDL represents the smallest quantity of an analyte that can be accurately and reproducibly quantified in a given sample matrix. The RL is determined by project objectives (e.g., cleanup goals) or technical limitations (e.g., three to five times the MDL). Worksheet #15 compares the RLs for the selected analytical method(s) to the project RAOs. The RLs and analytical results shall be reported with the same number of significant figures as the do-not-exceed cleanup goals. If the RL determined by the project objectives is not technically attainable (e.g., due to matrix interferences), then the RL will be set to the lowest technically attainable value or an alternative analytical method will be recommended.

Precision
Precision is defined as the degree of mutual agreement between individual measurements of the same property under similar conditions and provides a measurement of the reproducibility of an analytical result. Precision will be evaluated through the analysis of field duplicate samples, LCS and LCSD (if LCSD is run), and MS/MSD samples (see Worksheet #20). Field duplicate samples typically will be collected at a frequency of one duplicate per 10 samples of a given non-soil matrix (Worksheet #20). The identity of field duplicate samples will not be provided to the laboratory, and these samples will not be re-analyzed when field duplicate criteria are not met. Relative percent difference (RPD) criteria are specified in Worksheet #12. QC criteria failures will be documented in the case narrative and included in the Comprehensive Analytical Report. The affected data will be qualified as described in the EPA National Functional Guidelines, and the impact of the QC failures on the DQOs will be assessed in the QCSR.

Combined field and laboratory precision is evaluated by collecting and analyzing field duplicates and then calculating the variance between the samples, typically in terms of RPD, according to the following equation:

$$RPD = \frac{|A-B|}{(A+B)/2} \quad x \quad 100\%$$

SAP Worksheet #37 -- Usability Assessment (Continued)

where: A = First duplicate concentration

B = Second duplicate concentration

The precision data obtained from the results of QA/QC samples allow an approximation of the uncertainty of the analytical results.

Laboratory analytical precision is evaluated by analyzing matrix spikes and matrix spike duplicates (MS/MSD). The laboratory will have experimentally derived acceptance limits for RPDs established for each analytical method and sample matrix. The laboratory will ensure that internal QC sample results lie within acceptance limits; suspect trends will be evaluated and corrective actions taken.

Accuracy

Accuracy is the degree of agreement between an analytical measurement and a reference accepted as a true value. The accuracy of a measurement system can be affected by errors introduced by field contamination, sample preservation, sample handling, sample preparation, or analytical techniques. A program of sample spiking will be conducted to evaluate laboratory accuracy. Accuracy will be evaluated by the percent recovery of the spiked compounds in the LCS, LCS duplicate, and MS/MSD samples. LCS and MS samples will be spiked prior to extraction with the method target compounds indicated in this SAP. MS/MSD and LCS or blank spike samples will be analyzed at a frequency of 5 percent or one per sample delivery group/analytical batch (sample sets are about 10 samples). The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy, using the following equation:

$$\text{Percent Recovery} = \frac{S - C}{T} \times 100$$

where:

S = Measured spike sample concentration

C = Sample concentration

T = True or actual concentration of the spike

SAP Worksheet #37 -- Usability Assessment (Continued)

Worksheet #28 presents accuracy goals for this investigation based on the percent recovery of matrix and surrogate spikes. Results that fall outside the accuracy goals will be further evaluated on the basis of other QC samples.

For MS and MSD, sample heterogeneity and the presence of interfering compounds often negatively affect the accuracy and precision of the analysis. Also, the presence of high levels of target compounds in the sample chosen for spiking may necessitate a dilution of the sample, or may otherwise result in errors in spiked compound recovery. For these reasons, MS/MSD samples may not be truly representative of the accuracy and/or precision of the analytical process.

If MS/MSD analyses do not meet the specified recovery criteria, the recoveries from the LCS will be evaluated. If the LCS accuracy criteria are met, the failure of the MS/MSD will be attributed to interference from the sample matrix, and no corrective action will be required. If the LCS accuracy criteria are not met, the associated primary and QC samples will be re-prepared and re-analyzed.

In cases where re-preparation and re-analysis of the samples is not possible, the QC criteria failures will be documented in the case narrative and included in the Comprehensive Analytical Report. The affected data will be qualified as described in the EPA National Functional Guidelines, and the impact of the QC failures on the DQOs for the project will be assessed in the final report.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition that they are intended to represent. For this project, representative data will be obtained through careful selection of sampling locations and analytical parameters. Representative data will also be obtained through proper collection and handling of samples to avoid interference and minimize cross-contamination.

Representativeness of data will also be ensured through consistent application of the appropriate established field and laboratory procedures. To aid in evaluating the representativeness of the sample results, field and laboratory blank samples will be evaluated for the presence of contaminants. Laboratory procedures will be reviewed to verify that standard operating procedures were followed and method requirements were met during the analysis of project samples. Laboratory sample storage practices, holding times, sub-sampling procedures, method blanks, and evidence of matrix interference will be assessed for potential impacts on the representativeness of the data. Data determined to be non-representative will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

Representativeness as it relates to field procedures refers to the collection of samples that allow accurate conclusions to be made regarding the composition of the sample media at the entire site. Representativeness will be qualitatively assessed by evaluating whether the procedures described in this SAP were followed. The site-sampling layout, including sampling locations, frequency of sampling, and timing of sampling activities, will be reviewed.

SAP Worksheet #37 -- Usability Assessment (Continued)

Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with the QC procedures outlined in this SAP and when none of the QC criteria that affect data usability is exceeded. When data validation is completed, the percent completeness value will be calculated by dividing the number of useable sample results by the total number of sample results planned for this investigation. The evaluation of completeness will help determine whether any limitations are associated with the decisions to be made based on the data collected.

Completeness will be evaluated by reviewing the tasks that contribute to the sampling event, such as chain-of-custody procedures, adherence to the Work Plan, and adherence to this SAP. The QC parameters to be evaluated in determining completeness include: holding times, initial calibrations, continuing calibrations, surrogate recoveries, LCS recoveries, MS/MSD recoveries and RPDs, and laboratory duplicate RPDs. The completeness goal for this project is 95%.

Comparability

Comparability expresses the confidence with which one data set can be compared with another. Comparability of data will be achieved by consistently following standard field and laboratory procedures and by using standard measurement units in reporting analytical data. Analytical methods selected for this field investigation are consistent with the methods used during previous investigations of this type.

To ensure the comparability of laboratory data, the contract laboratory will use standard test methods and means of sample preservation; standard units, detection limits, calculation procedures, and reporting formats; and standard measures of accuracy and precision. Only laboratories that have been approved by the DoD ELAP will perform chemical analyses of environmental samples in support of this CTO.

References

PRC Environmental Management, Inc., 1994. *Final Operable Unit 6, Remedial Investigation, Moffett Federal Airfield, Ca.*

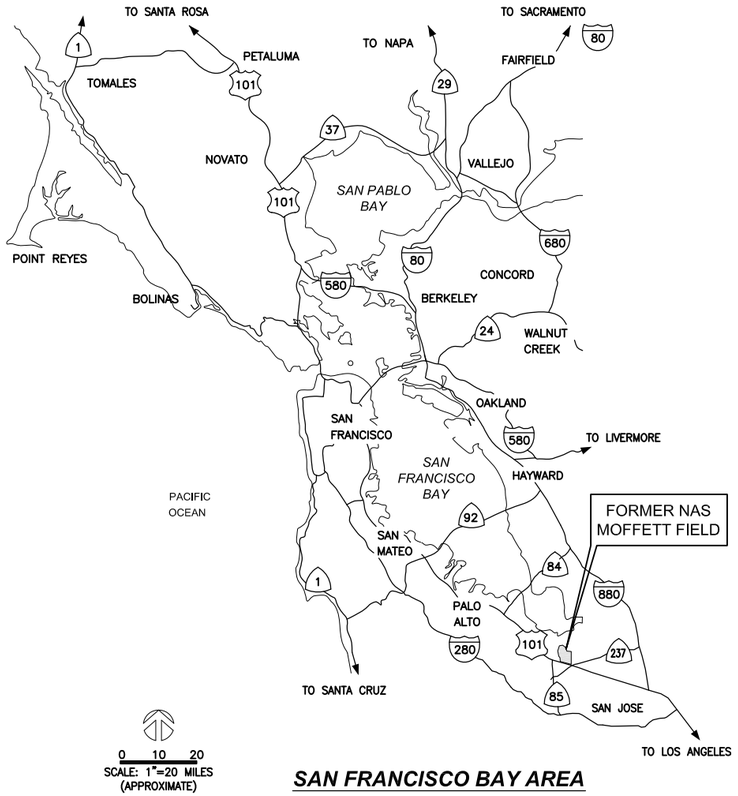
Sultech, 2007. *Final Addendum to the Revised Final Station-Wide Feasibility Study Site 25, Former Naval Air Station Moffett Field, Santa Clara County, California.* 29 June.

United States Department of the Navy (Navy), 2009. *Final Record of Decision, Former Naval Air Station Moffett Field, California.* November.

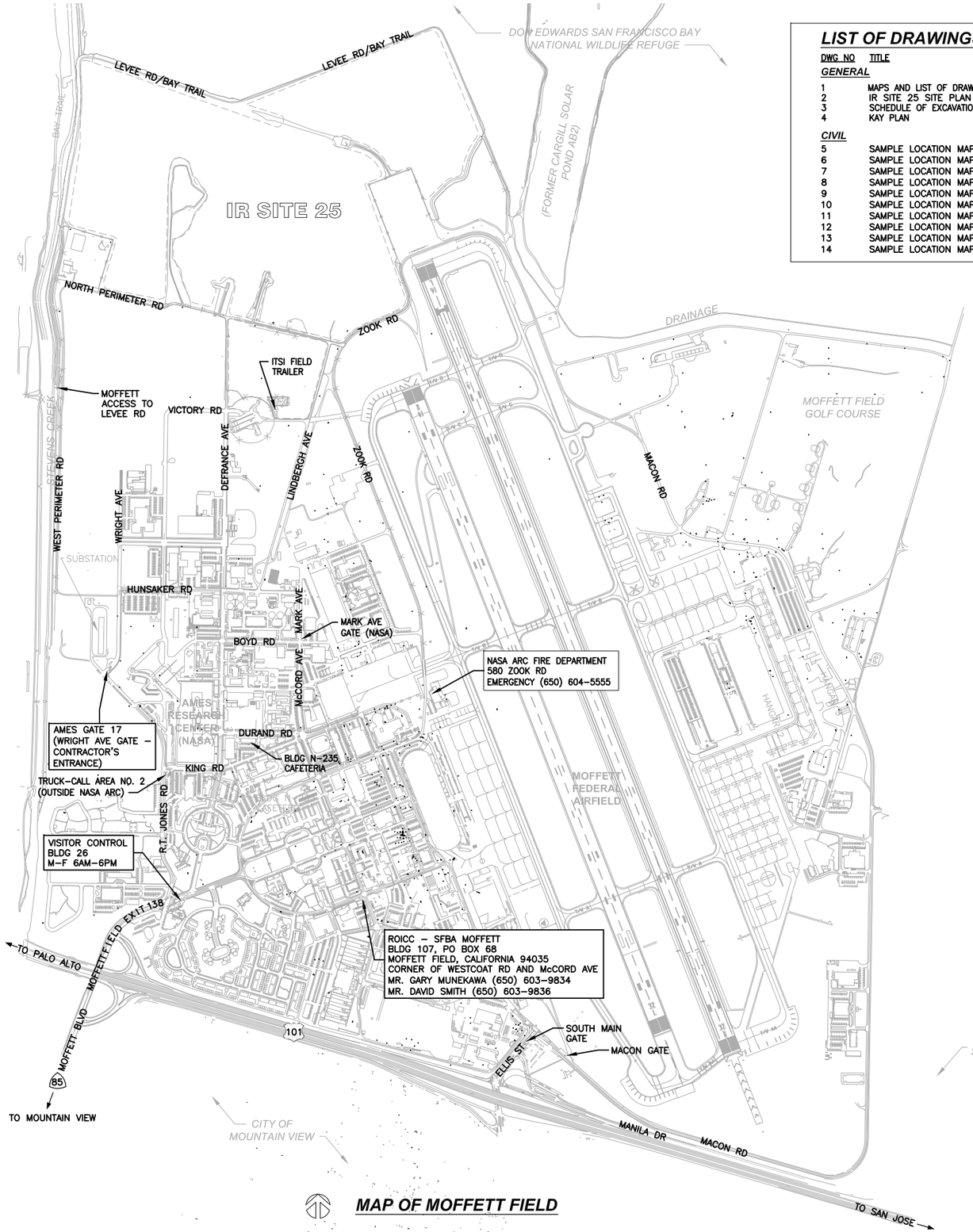
United States Army Corps of Engineers, 2008. Safety and Health Requirements, EM 385-1-1. 15 September.

SAP FIGURES

CALIFORNIA



SAN FRANCISCO BAY AREA



MAP OF MOFFETT FIELD

LIST OF DRAWINGS

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA. 94598

Maps and List of Drawings

Sampling & Analysis Plan January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET ADJUST SCALE ACCORDINGLY

FIGURE 1

IR SITE 25

LOCKED GATE
ALVISO POND A2E (INUNDATED)
INUNDATED
IR SITE 25 BOUNDARY
LEVEE RD/BAY TRAIL
BORROW AREA BA1
BORROW AREA BA2
BORROW AREA BA3
PARTIALLY INUNDATED
STORMWATER RETENTION POND (NE BASIN)
STORMWATER RETENTION POND (CENTRAL BASIN)
STEVENS CREEK SHORELINE STUDY AREA (MROSD)
AOI 14 (NASA)
LANDFILL RD
IR SITE 1 (FORMER LANDFILL)
303
267
217A
GATE 16C
217
GATE 16B
NORTH PERIMETER RD
SOIL LAYDOWN AREA
ZOOK RD
ARC 21
CULVERTS
WESTERN DIKED MARSH
POINT P1
GATE 16A
GATE H
GATE 15B
GATE 16
GATE 16E
LOCKED GATE
EASTERN DIKED MARSH
NA289 (SETTLING BASIN)
GATE 16F
VICTORY RD
ARC 17
TRUCK-CALL AREA NO. 1
FIELD TRAILER
T/W B
T/W C
FLIGHTLINE
STEVENS CREEK
WEST PERIMETER RD
PARSONS AVE
DEFRANCE AVE
LINDBERGH AVE
NORTH WAREHOUSE RD
WRIGHT AVE
ALLEN RD
HUNSAKER RD
AMES GATE 17 (WRIGHT'S AVE GATE – CONTRACTORS' ENTRANCE)
TO R.T. JONES RD (PUBLIC THOROUGHFARE)

N

250 125 0 250 500
GRAPHIC SCALE IN FEET

NOTES:
1. AREAS TO BE EXCAVATED ARE SHADED.

FIGURE 2	VERIFY SCALE BAR IS ONE INCH ON ORIGINAL DRAWING IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY	**Remedial Action at IR Site 25** Former NAS Moffett Field Moffett Field, California	Sampling & Analysis Plan January 2012	IR Site 25 Site Plan	ITSI Gilbane 2730 Shadelands Drive Walnut Creek, CA. 94598

EXCAVATIONS AT STORMWATER RETENTION POND

AREA DESIGNATION	SHEET REFERENCE NUMBER	DEPTH (FT)	AREA (SF)	BANK VOLUME (CY)	POSSIBLE TREATMENT
A1.1	CSP-1, -2	0.5	41,273	764	X
A2.1	CSP-2	1.0	23,557	872	-
A3.1	CSP-3	0.5	18,408	341	X
A4.1	CSP-4	0.5	98,938	1,832	X
A4.2	CSP-4	0.5	29,336	543	-
A4.3	CSP-4	0.5	15,024	278	-
A4.4	CSP-4	0.5	14,814	274	-
A4.5	CSP-4, -5	0.5	23,433	434	-
A5.1	CSP-5	0.5	35,696	661	-
A5.2	CSP-5, -6	0.5	59,690	1,105	X
A6.1	CSP-5, -6	1.0	39,614	1,467	-
A6.2	CSP-6, -9	1.0	31,893	1,181	-
A6.3	CSP-6	0.5	18,590	344	X
A6.4	CSP-6	0.5	788	15	-
A6.5	CSP-6	0.5	3,152	58	-
A8.1	CSP-7, -8	0.5	40,244	745	X
A8.2	CSP-8	0.5	29,563	547	-
A8.3	CSP-5, -9	1.5	38,670	2,148	-
A8.4	CSP-8, -9	1.5	23,923	1,329	-
A8.5	CSP-9	0.5	20,556	381	-
A8.6	CSP-9	0.5	21,969	407	-
A8.7	CSP-8	1.5	13,739	763	-
A8.8	CSP-8	0.5	16,028	297	-
A8.9	CSP-8	0.5	16,231	301	X
A8.10	CSP-8	0.5	13,508	250	X
A8.11	CSP-8	1.0	12,805	474	-
A8.12	CSP-8	0.5	30,711	569	X
A8.13	CSP-8	0.5	14,959	277	-
A8.14	CSP-8	0.5	9,139	169	-
A8.15	CSP-8	0.5	19,476	361	-
A8.16	CSP-8	0.5	8,150	151	-
A8.17	CSP-8	1.0	6,676	247	X
A8.18	CSP-8, -10	0.5	5,975	111	-
A8.19	CSP-8	0.5	4,305	80	X
A8.20	CSP-8	0.5	2,591	48	X
A8.21	CSP-8	1.0	2,993	111	X
A8.22	CSP-8	0.5	3,977	74	X
A8.23	CSP-8	0.5	5,988	111	-
A8.24	CSP-8	1.0	10,349	383	-
A8.25	CSP-8	0.5	19,310	358	-
A9.1	CSP-5, -6, -8, -9	0.5	34,249	634	-
A9.2	CSP-9	0.5	5,323	99	-
A9.3	CSP-9	0.5	15,438	286	-
A9.4	CSP-9	1.0	13,911	515	X
A9.5	CSP-9	0.5	17,638	327	-
A9.6	CSP-9	1.0	23,062	854	X
A9.7	CSP-9	0.5	7,899	146	X
A9.8	CSP-9	0.5	23,701	439	-
A9.9	CSP-9	0.5	7,474	138	-
A9.10	CSP-9	1.0	7,222	267	-
A9.11	CSP-9	1.0	9,294	344	-
A9.12	CSP-9	0.5	19,040	353	-
A10.1	CSP-8, -10	0.5	23,379	433	-
A10.2	CSP-8, -10	0.5	15,206	282	-
A10.3	CSP-10	1.0	11,704	433	X
A10.4	CSP-10	0.5	8,689	161	-
A10.5	CSP-10	1.0	7,675	284	X
A10.6	CSP-10	0.5	8,312	154	-
A10.7	CSP-10	0.5	9,278	172	-
TOTALS	-	-	1,114,535	27,184	-

EXCAVATIONS AT EASTERN DIKED MARSH

AREA DESIGNATION	SHEET REFERENCE NUMBER	DEPTH (FT)	AREA (SF)	BANK VOLUME (CY)	POSSIBLE TREATMENT
A10.8	CSP-10	0.5	5,311	98	X
A10.9	CSP-10	0.5	9,154	170	X
A10.10	CSP-10	0.5	6,620	123	X
A10.11	CSP-10	0.5	5,034	93	X
A10.12	CSP-10	0.5	4,498	83	X
A10.13	CSP-10	0.5	1,010	19	X
A10.14	CSP-10	0.5	252	5	X
A10.15	CSP-10	0.5	1,591	29	X
A10.16	CSP-10	0.5	6,985	129	X
A10.17	CSP-10	0.5	2,849	53	X
A10.18	CSP-10	0.5	1,064	20	X
A10.19	CSP-10	0.5	6,260	116	X
A10.20	CSP-10	0.5	6,315	117	X
A10.21	CSP-10	0.5	2,848	53	X
A10.22	CSP-10	0.5	4,156	77	-
A10.23	CSP-10	0.5	3,470	64	X
A10.24	CSP-10	0.5	9,396	174	-
A10.25	CSP-10	0.5	4,266	79	-
A10.26	CSP-10	0.5	10,320	191	-
A10.27	CSP-10	0.5	3,953	73	-
A10.28	CSP-10	0.5	4,808	89	X
A10.29	CSP-10	0.5	4,612	85	X
A10.30	CSP-10	0.5	6,539	121	-
A10.31	CSP-10	0.5	3,574	66	-
A10.32	CSP-10	0.5	5,852	108	-
A10.33	CSP-10	0.5	3,483	65	X
A10.34	CSP-10	0.5	8,301	154	-
A10.35	CSP-10	0.5	8,972	166	X
A10.36	CSP-10	0.5	8,408	156	X
A10.37	CSP-10	0.5	7,243	134	-
A10.38	CSP-10	0.5	12,025	223	X
A10.39	CSP-10	1.0	4,354	161	X
A10.40	CSP-10	0.5	12,500	231	-
A10.41	CSP-10	0.5	10,437	193	-
A10.42	CSP-10	0.5	54,344	1,006	-
A10.43	CSP-10	1.0	1,369	51	-
A10.44	CSP-10	0.5	624	12	-
A10.45	CSP-10	0.5	1,140	21	X
A10.46	CSP-10	1.0	694	26	X
A10.47	CSP-10	1.0	705	26	X
A10.48	CSP-10	0.5	1,177	22	X
A10.49	CSP-10	0.5	4,895	91	-
A10.50	CSP-10	0.5	1,379	26	X
A10.51	CSP-10	0.5	1,888	35	X
A10.52	CSP-10	0.5	1,344	25	X
A10.53	CSP-10	1.0	474	18	X
A10.54	CSP-10	0.5	933	17	X
A10.55	CSP-10	1.0	1,127	42	X
A10.56	CSP-10	0.5	877	16	-
A10.57	CSP-10	0.5	1,347	25	-
A10.58	CSP-10	1.0	1,094	41	X
A10.59	CSP-10	0.5	916	17	-
A10.60	CSP-10	1.0	697	26	-
A10.61	CSP-10	1.0	1,976	73	-
TOTALS	-	-	275,460	5,332	-

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA, 94598

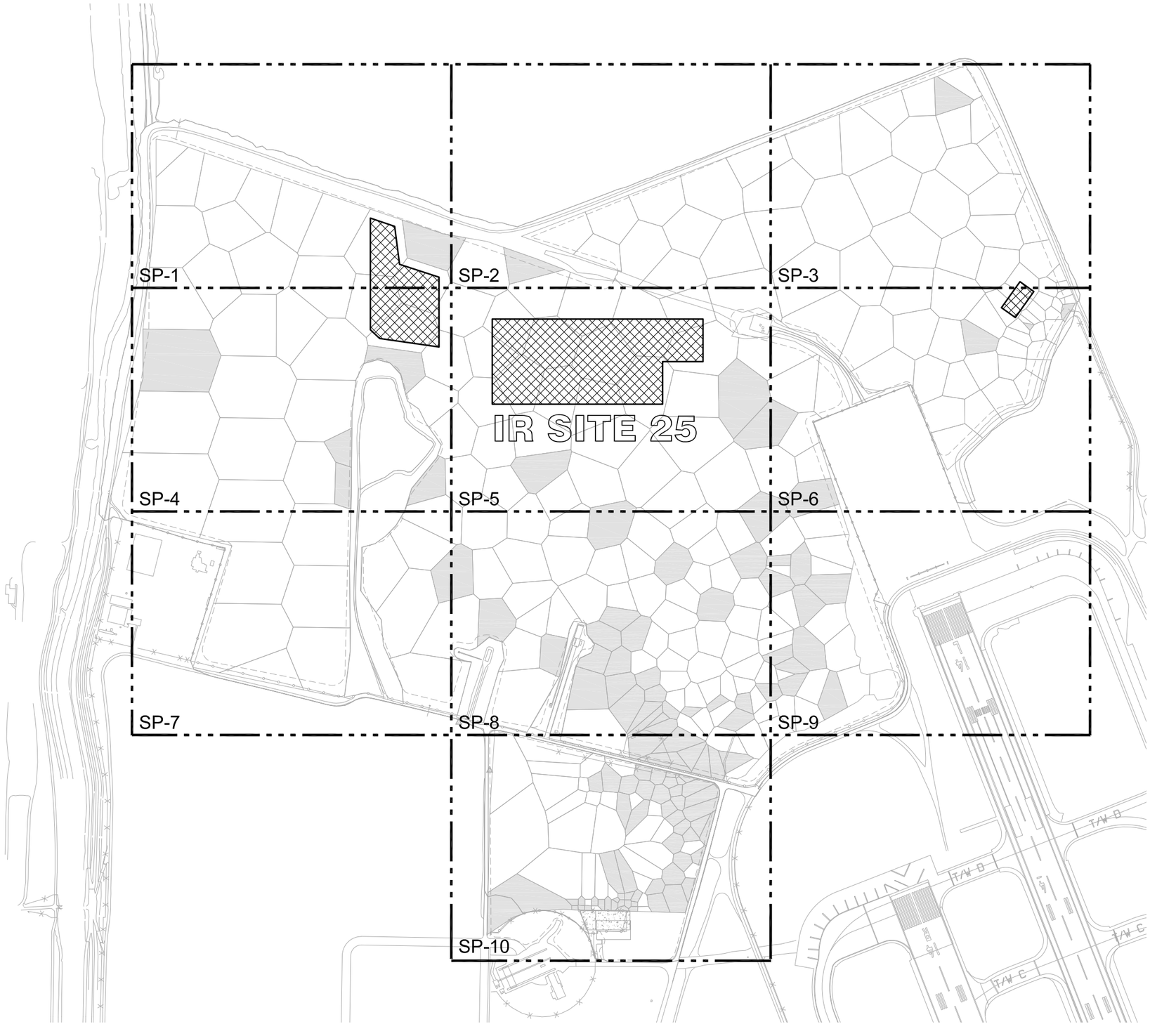
Schedules of Excavations

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE 3



WARNING: BURIED GAS TRANSMISSION LINE

TAKE EVERY PRECAUTION REGARDING THE EXISTING NATURAL GAS AND OTHER POTENTIAL LINES. BEFORE ANY EARTH–DISTURBING OR INVASIVE ACTIVITY, DO THE FOLLOWING AT A MINIMUM:

1. AT LEAST 2 WORKING DAYS BUT NOT MORE THAN 14 CALENDAR DAYS PRIOR TO COMMENCING EXCAVATION, CONTACT USA IN CONFORMANCE WITH CALIFORNIA STATE LAW (CODE SECTION 4216.1) TO MARK UNDERGROUND INSTALLATIONS.
2. CONTACT PG&E DIRECTLY TO CONFIRM THE DEPTH AND LOCATION OF THE NATURAL GAS AND OTHER POTENTIAL LINES.
3. CONTACT THE FACILITY OPERATOR (NASA) TO LOCATE UNDERGROUND PRIVATE LINES AND INSTALLATIONS.
4. WHERE THE GAS LINES CROSS A PLANNED EXCAVATION AREA, MARK THE LINES THROUGH THE EXCAVATION AREA AND A MINIMUM OF 50 FT IN EACH DIRECTION OUTSIDE THE EXCAVATION AREA.
5. DETERMINE THE EXACT LOCATION OF EXISTING NATURAL GAS AND OTHER POTENTIAL LINES IN CONFLICT WITH THE EXCAVATION AREA BY DIGGING WITH HAND TOOLS, OR USING SOME OTHER ADEQUATE MEANS TO SAFELY IDENTIFY THEM.
6. HAND DIGGING SHALL BE PERFORMED TO 3 FT ON EITHER SIDE OF THE MARKED UNDERGROUND INSTALLATION AND TO A DEPTH OF 1 FT BELOW THE PLANNED EXCAVATION DEPTH. CONSULT WITH ITSI AFTER HAND DIGGING TO VERIFY LOCATIONS OF THE EXISTING UNDERGROUND INSTALLATIONS.
7. ONLY AFTER DOCUMENTED CONSULTATION WITH THE FACILITY AND UNDERGROUND INSTALLATION OWNERS AND ITSI WILL EXCAVATION USING MECHANICAL MEANS BE ALLOWED. SUBMIT DOCUMENTATION TO ITSI PRIOR TO COMMENCING MECHANICAL EXCAVATION. PROVIDE A SPOTTER TO WATCH FOR UNDERGROUND INSTALLATIONS DURING EXCAVATION.
8. ON EACH DAY THAT EXCAVATION IS CONDUCTED OVER A GAS LINE OR OTHER IDENTIFIED LINE, ADVISE ALL SITE WORKERS OF THE SITUATION.

LEGEND

AREAS TO BE EXCAVATED AND CONFIRMED AS CLEAN

AREAS TO BE CHARACTERIZED AS BORROW MATERIAL

N

250 125 0 250 500
GRAPHIC SCALE IN FEET

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA, 94598

Key Plan

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE 4

PLOT BY: PERMANEX — Jan 12, 2012 — 12:41:34pm

DRAWING: P:\07204.0000 MAFAC SW R&D\07204.0003 RA at IR 25, Moffett Field\10.0 CAD\1400_CAD\ Current Drawings\04 SAP Sampling & Analysis P\07204.0003-MOFFETT-IR25-SAP Figures.dwg

LEGEND

- ⊠ BORROW AREA SEDIMENT SAMPLE
- ⊗ CONFIRMATION SEDIMENT SAMPLE
- IR SITE 25 BOUNDARY

LOCKED GATE

ALVISO POND A2E
(INUNDATED)

LEVEE RD (BAY TRAIL)

LEVEE RD (BAY TRAIL)

BA1–01
BA1–02
BA1–03
BA1–04
BA1–05

A1.1–CN01
A1.1–CN02
A1.1–CN03
A1.1–CN04

A1.1

SP-1 SP-2 SP-3
SP-4 SP-5 SP-6
SP-7 SP-8 SP-9
SP-10

KEY PLAN

N

50 25 0 50 100
GRAPHIC SCALE IN FEET

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA, 94598

Sample
Location Map (1)

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE SP-1

PLOT BY: PFERNANDEZ — Jan 12, 2012 - 9:45:08am

DRAWING: P:\07204.0000 NAVFAC SW RAC\07204.0003 RA at IR 25, Moffett Field\100 CADD\400_CADD Current Drawings\04 SAP Sampling & Analysis 07204.0003–MOFFETT–R25–SAP Figures.dwg

LEGEND

⊗ CONFIRMATION SEDIMENT SAMPLE

– – – – – IR SITE 25 BOUNDARY

ALVISO POND A2E
(INUNDATED)

LEVEE RD (BAY TRAIL)

A1.1

A2.1

A2.1–CN01

A2.1–CN02

KEY PLAN

SP-1	SP-2	SP-3
SP-4	SP-5	SP-6
SP-7	SP-8	SP-9
	SP-10	

N

50 25 0 50 100
GRAPHIC SCALE IN FEET

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA. 94598

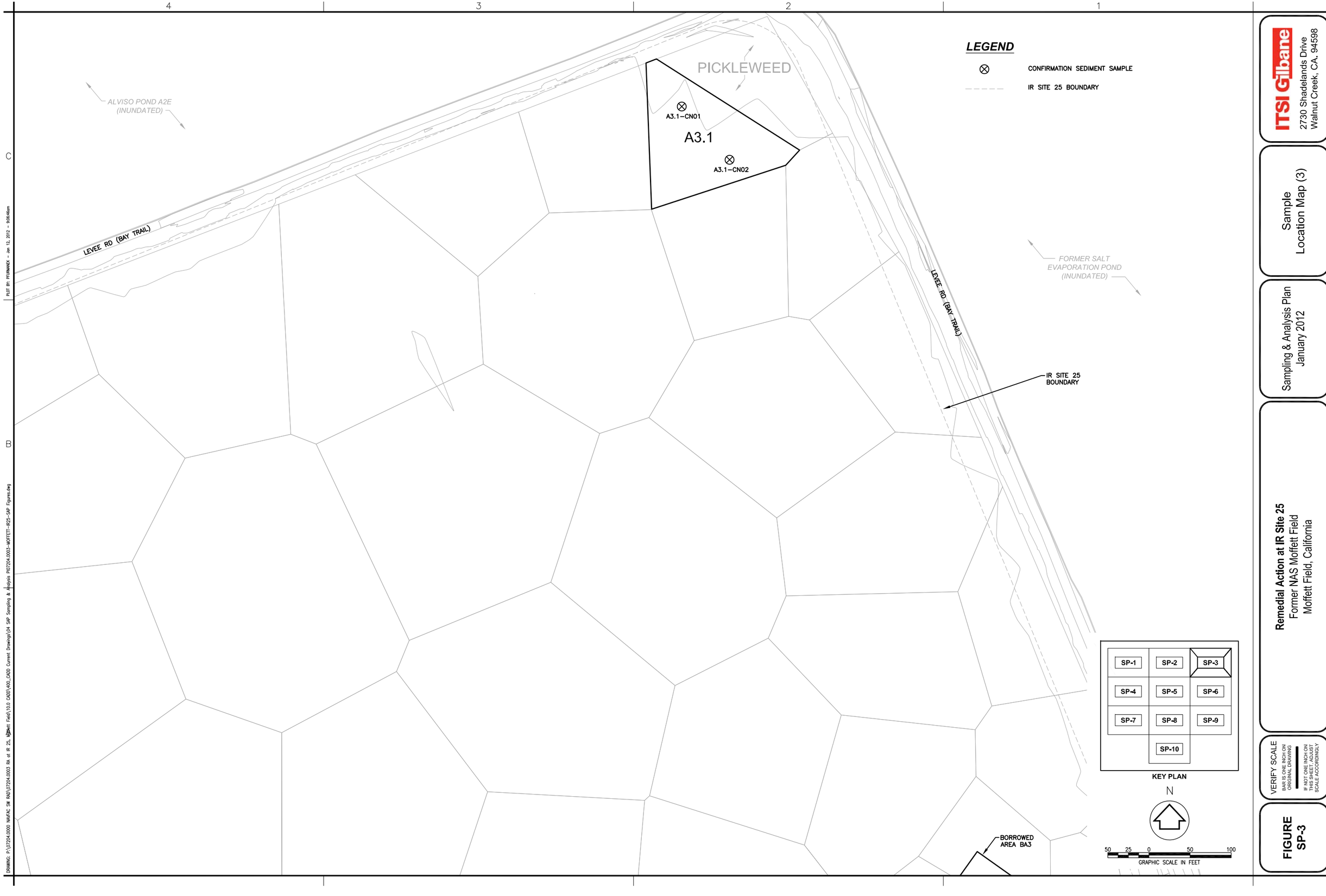
Sample
Location Map (2)

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE SP-2



ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA. 94598

Sample
Location Map (3)

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE SP-3

PLOT BY: PERMANEK — Jan 12, 2012 — 9:06:46am
DRAWING: P:\07204.0000 MMPAC SW RAD\07204.0003 RA at IR 25, Moffett Field\10.0 CADD\10_CADD Current Drawings\04 SAP Sampling & Analysis P07204.0003–MOFFETT–IR25–SAP Figures.dwg

LEVEE RD (BAY TRAIL)

A4.1–CN01 A4.1–CN02 A4.1–CN03

A4.1

A4.1–CN04 A4.1–CN05 A4.1–CN06 A4.1–CN07

SALT GRASS
WITH PICKLEWEED

A4.1–CN08 A4.1–CN09 A4.1–CN010

BA1–06 BA1–07 BA1–08

BA1–09 BA1–10 BA1–11

A4.2–CN01

A4.2

A4.2–CN02

PICKLEWEED

IR SITE 25
BOUNDARY

AOI 14
(NASA)

PICKLEWEED

A4.3–CN01

A4.3

A4.3–CN02

A4.4–CN01

A4.4

WELL

A4.5–CN02

A4.5

A4.5–CN01

LEGEND

- BORROW AREA SEDIMENT SAMPLE
- CONFIRMATION SEDIMENT SAMPLE
- (E) GW MONITORING WELL
- IR SITE 25 BOUNDARY

SP-1 SP-2 SP-3
SP-4 SP-5 SP-6
SP-7 SP-8 SP-9
SP-10

KEY PLAN

N

50 25 0 50 100
GRAPHIC SCALE IN FEET

PLOT BY: PFERNANDEZ — Jan 12, 2012 — 9:07:03am

DRAWING: P:\07204.0000 WAFAC SW RAO\07204.0003 RA at IR 25, Moffett Field\10.0 CAD\400_CAD Current Drawings\04 SAP Sampling & Analysis R07204.0003-MOFFETT-IR25-SAP Figures.dwg

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA. 94598

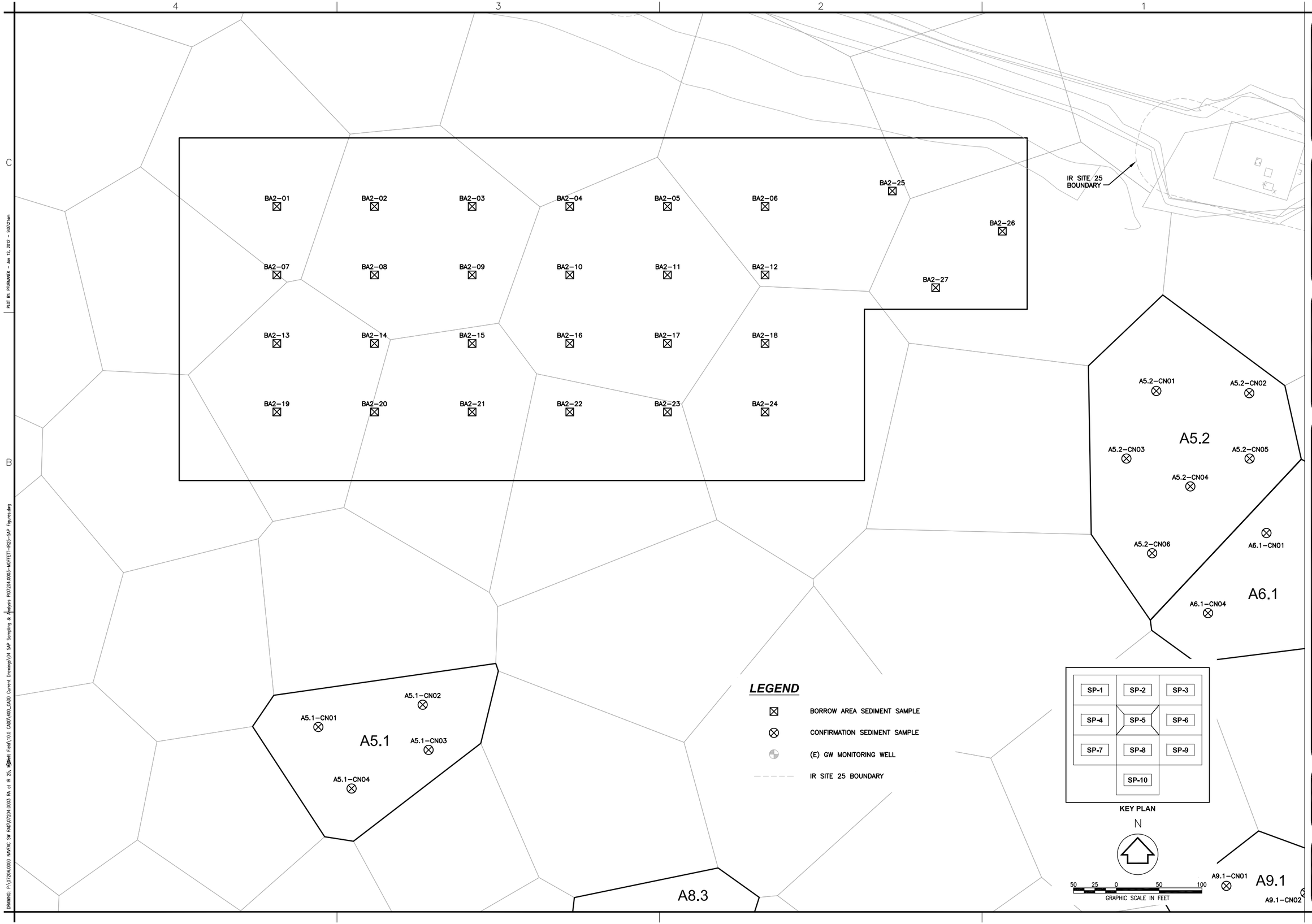
Sample
Location Map (4)

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET ADJUST SCALE ACCORDINGLY

FIGURE SP-4



ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA, 94598

Sample Location Map (5)

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE SP-5

PLOT BY: PFERNANDEZ — Jan 12, 2012 - 9:07:21am

DRAWING: P:\07204.0000 MAFAC SW PA01\07204.0003 RA at IR 25, Moffett Field\10.0 CAD\V00_CAD\ Current Drawings\04 SAP Sampling & Analysis P07204.0003-MOFFETT-IR25-SAP Figures.dwg

LEGEND

- BORROW AREA SEDIMENT SAMPLE
- CONFIRMATION SEDIMENT SAMPLE
- (E) GW MONITORING WELL
- IR SITE 25 BOUNDARY

KEY PLAN

N

50 25 0 50 100

GRAPHIC SCALE IN FEET

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA. 94598

Sample
Location Map (6)

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE SP-6

PLOT BY: PFERNANDEZ — Jan 12, 2012 — 9:07:40am

DRAWING: P:\07204.0000 NAVFAC SW RAC\07204.0003 RA at IR 25, Moffett Field\10.0 CADD\400_CADD Current Drawings\04 SAP Sampling & Analysis P07204.0003-MOFFETT-IR25-SAP Figures.dwg

WARNING: BURIED GAS TRANSMISSION LINE!
SEE NOTE ON FIGURE 4.

AOI 14
(NASA)

IR SITE 25
BOUNDARY

A8.1–CN01

A8.1

A8.1–CN04

IR SITE 25
BOUNDARY

NORTH PERIMETER RD

PG&E BURIED
GAS LINE

GATE 16C

10H02A

LEGEND

(E) GW MONITORING WELL

IR SITE 25 BOUNDARY

KEY PLAN

SP-1	SP-2	SP-3
SP-4	SP-5	SP-6
SP-7	SP-8	SP-9
	SP-10	

N

50 25 0 50 100
GRAPHIC SCALE IN FEET

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA. 94598

Sample
Location Map (7)

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE SP-7

PLOT BY: PFERNANDEZ — Jan 12, 2012 — 9:07:57am

DRAWING: P:\07204.0000 NAVFAC SW PA\07204.0003 RA at IR 25, Moffett Field\10.0 CADD\A90_CADD Current Drawings\04 SAP Sampling & Analysis P07204.0003–MOFFETT–R25–SAP Figures.dwg

SP-1 SP-2 SP-3
SP-4 SP-5 SP-6
SP-7 SP-8 SP-9
SP-10

KEY PLAN

N

50 25 0 50 100
GRAPHIC SCALE IN FEET

LEGEND

⊗ CONFIRMATION SEDIMENT SAMPLE

IR SITE 25 BOUNDARY

A8.1
A8.1–CN02
A8.1–CN03

A8.2
A8.2–CN01
A8.2–CN02

A8.3
A8.3–CN01
A8.3–CN02
A8.3–CN03
A8.3–CN04

A8.4
A8.4–CN01
A8.4–CN02

A8.5
A8.5–CN01
A8.5–CN02

A8.6
A8.6–CN01
A8.6–CN02

A8.7
A8.7–CN01

A8.8
A8.8–CN01
A8.8–CN02

A8.9
A8.9–CN01
A8.9–CN02

A8.10
A8.10–CN01

A8.11
A8.11–CN01

A8.12
A8.12–CN01
A8.12–CN02

A8.13
A8.13–CN01

A8.14
A8.1–CN01

A8.15
A8.15–CN01
A8.15–CN02

A8.16
A8.16–CN01

A8.17
A8.17–CN01

A8.18
A8.18–CN01

A8.19
A8.19–CN01

A8.20
A8.20–CN01

A8.21
A8.21–CN01

A8.22
A8.22–CN01

A8.23
A8.23–CN01

A8.24
A8.24–CN01

A8.25
A8.25–CN01
A8.25–CN02

A9.1
A9.1–CN03
A9.1–CN04

A9.2

A9.4

A9.7

A9.10
A9.10–CN01

A10.1
A10.1–CN02

A10.2

IR SITE 25 BOUNDARY

CATTAILS

PICKLEWEED

PICKLEWEED

PICKLEWEED

IR SITE 25 BOUNDARY

217 (NASA)

217A (NASA)

GATE 19B

NORTH PERIMETER RD

DRAWING: P:\07204.0000 MW\FIG_SW RAO\07204.0003 RA at IR 25, Moffett Field\10.0 CAD\A00_CAD Current Drawings\04 SAP Sampling & Analysis P07204.0003–MOFFETT–R25–SAP Figures.dwg

PLOT BY: PFRANKEK — Jan 12, 2012 – 9:37:34am

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA. 94598

Sample Location Map (8)

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE SP-8

A9.1

A6.2

A9.2

A9.2–CN01

A9.3–CN01

A9.3

A9.3–CN02

A9.4–CN01

A9.4

A9.6–CN01

A9.6

A9.6–CN02

A9.5–CN01

A9.5

A9.5–CN02

IR SITE 25 BOUNDARY

PG&E BURIED GAS LINE

A9.8–CN01

A9.8

A9.8–CN02

A9.7–CN01

A9.7

A9.9–CN01

A9.9

A9.11–CN01

A9.11

A9.10–CN01

A9.10

A9.12–CN01

A9.12

A9.12–CN02

PG&E BURIED GAS LINE

WELL

ZOOK RD

ZOOK RD

FLIGHT LINE

LEGEND

- CONFIRMATION SEDIMENT SAMPLE
- (E) GW MONITORING WELL
- IR SITE 25 BOUNDARY

WARNING: BURIED GAS TRANSMISSION LINE!
SEE NOTE ON FIGURE 4.

SP-1	SP-2	SP-3
SP-4	SP-5	SP-6
SP-7	SP-8	SP-9
	SP-10	

KEY PLAN

N

50 25 0 50 100

GRAPHIC SCALE IN FEET

ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA. 94598

Sample Location Map (9)

Sampling & Analysis Plan
January 2012

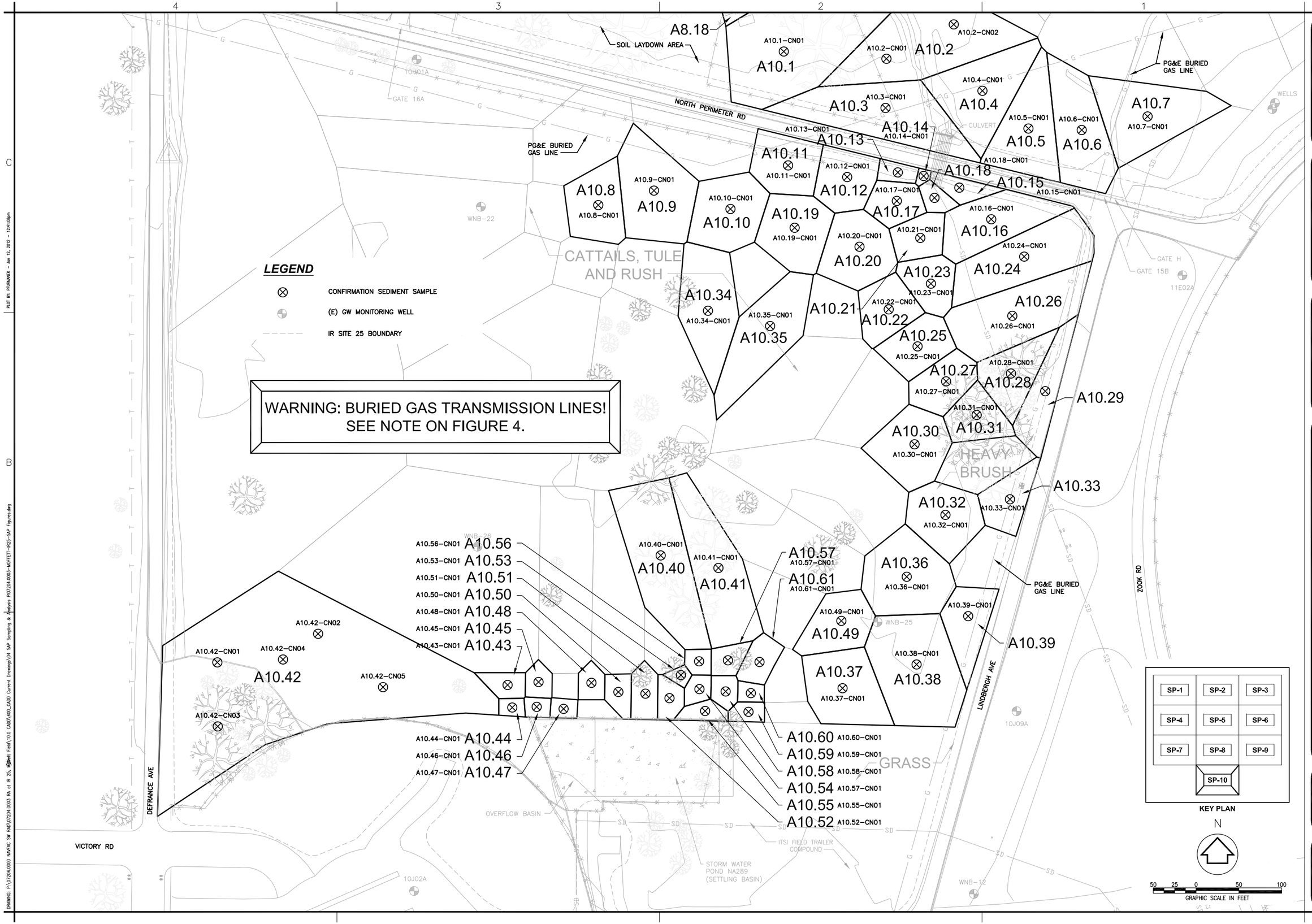
Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE SP-9

PLOT BY: PFERNANDEZ — Jan 12, 2012 - 9:09:38am

DRAWING: P:\07204.0000 NAVFAC SW PMO\07204.0003 RA at IR 25, Moffett Field\100 CAD\100_CAD Current Drawings\04 SAP Sampling & Analysis P07204.0003-MOFFETT-IR25-SAP Figures.dwg



ITSI Gilbane
2730 Shadelands Drive
Walnut Creek, CA. 94598

Sample Location Map (10)

Sampling & Analysis Plan
January 2012

Remedial Action at IR Site 25
Former NAS Moffett Field
Moffett Field, California

VERIFY SCALE
BAR IS ONE INCH ON ORIGINAL DRAWING
IF NOT ONE INCH ON THIS SHEET, ADJUST SCALE ACCORDINGLY

FIGURE SP-10

PLOT BY: PERNANEK — Jan 13, 2012 — 12:41:08pm

DRAWING: P:\07204.0000 MW\FAC SW RAD\07204.0003 RA at IR 25, Moffett Field\10.0 CAD\10.400_CAD\ Current Drawings\04 SAP Sampling & Analysis P\07204.0003-MXFFETT-IR25-SAP Figures.dwg

SAP ATTACHMENT 1
SAMPLE FIELD FORMS

Innovative Technical Solutions, Inc.
2730 Shadelands Drive, Suite 100
Walnut Creek, California 94598
(925) 946-3100 – (925) 256-8998 (fax)

Local Address: _____

Chain-Of-Custody

Project Name and Number: _____ Laboratory Name: _____ Date: _____

Project Manager: _____ Address: _____ Contact Name: _____ Page: _____ of _____

Site Location: _____ Phone: _____

Sample I.D.	Date	Time	Sample Depth	No. of Containers	Sample Matrix	Analysis:	Preservative: / Container Type: / Special Instructions/Comments

Sampled By: _____

Signature: _____

Special Instructions: _____

Send Results to: (w/fax #) _____

Turnaround Time: _____

Courier/Airbill No.:

Relinquished By/Affiliation:	Date:	Time:	Received By/Affiliation:	Date:	Time:

Original – Laboratory Yellow – Field/Office

Innovative Technical Solutions, Inc.
2730 Shadelands Drive, Suite 100
Walnut Creek, CA. 94598
(925) 946-3100 (TEL), (925) 256-8998 (FAX)

PROJECT:
PROJECT NO.:
SITE LOCATION:

SAMPLE COLLECTION LOG

DATE:
PAGE: of

SAMPLE NUMBER	DATE SAMPLED	TIME SAMPLED	SAMPLE LOCATION	SAMPLED BY	SAMPLE DESCRIPTION	FINAL SAMPLE DISPOSITION	
			TOTAL SAMPLES				*TOTAL TESTS*

SAMPLER(S) SIGNATURE(S)

ITSI Innovative Technical Solutions, Inc.

Sheet ___ of ___

MONITORING WELL DEVELOPMENT FORM

Project Name: _____ Project No.: _____

Well No.: _____ Tested By: _____ Date: _____

Measuring Point Description: _____ Initial Water Level (ft.): _____

Total Well Depth (ft.): _____ Final Water Level (ft.): _____

Water Level Measurement Method: _____ Time Start Develop: _____

Development Method(s): _____ Time End Develop: _____

Comments: _____

Well Volume Calculation	Total Depth (ft)	-	Depth to Water (ft)	=	Water Column (ft)	x	Multiplier for Casing Diameter (in)			=	Casing Volume (gal)
							2	4	6		
							0.16	0.64	1.44		

Time										
Depth to water										
Volume purged (gals)										
∑ volume purged (gals)										
∑ casing volumes										
Purge rate (gpm)										
Temperature (F°/C°)										
pH										
Specific conductivity (mS/cm)										
Dissolved oxygen (mg/L)										
Turbidity or Color										
Odor?										
De-watered?										

ITSI Innovative Technical Solutions, Inc.

Low-Flow Groundwater Purge Log

Project Name: _____ Project No.: _____

Well No.: _____ Tested By: _____ Date: _____

Measuring Point Description: _____ Static Water Level (ft.): _____

Total Well Depth (ft.): _____ Sample Depth (ft.): _____

Water Level Measurement Method: _____

Purge Method: _____ Sample Method: _____

Time Start Purge: _____ Field Filtering: _____

Time End Purge: _____ Time Sampled: _____

Purge Rate: _____

Comments: _____

Time	pH	Sp. Cond. (mS/cm)	Turbidity (NTU)	DO (mg/L)	Temp. (^{o}C)	ORP (mV)	Volume Pumped(L)	Comments

Project _____ Logged By _____ **Boring No.** _____

Project Number _____ Date Drilled _____ Sheet _____ Of _____

Location _____ Total Depth _____ Boring Location Sketch

Surface Elevation _____ Boring Diameter _____

Drillers _____

Method _____

Depth (Feet)	Sample Interval	Blow Counts	PID (ppm) B-zone/stem/sample	Water Level	Well Construction	Lithology / USCS	DESCRIPTION
5							
10							
15							
20							
25							
30							

Casing Diameter _____ Casing Length _____ From _____ To _____

Screen Size _____ Screen Length _____ From _____ To _____

Sand Type _____ From _____ To _____

Bentonite Type _____ From _____ To _____

Cement/Grout _____ From _____ To _____

Surface Completion _____

ITSI Innovative Technical Solutions, Inc.

Innovative Technical Solutions, Inc.

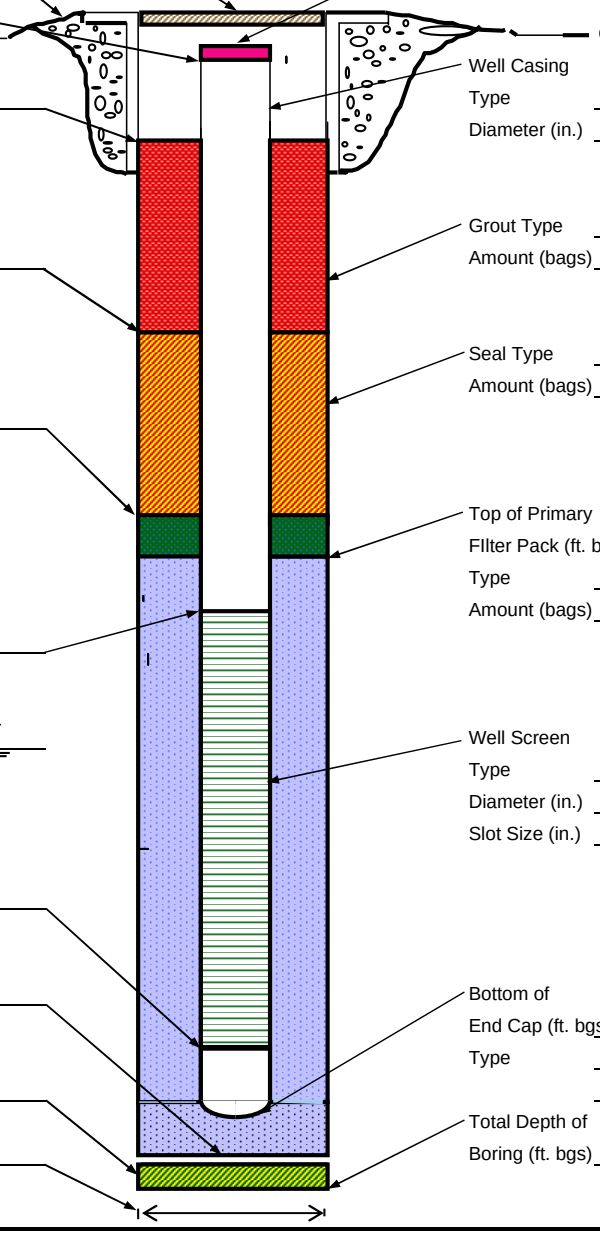
WELL CONSTRUCTION DETAIL

WELL NO: _____

Project Name: _____ **Project No:** _____ **Boring No.** _____ **Drilling Contractor:** _____

Site: _____ **Datum:** **Method:** _____ **Date Installed:** _____

Notes: _____



Flush Mount Well Completion Box _____

Concrete Surface Seal _____

Top of Well Casing _____

Top of Grout (ft. bgs) _____

Top of Seal (ft. bgs) _____

Top of Secondary Filter Pack (ft. bgs) _____

Type _____

Amount (bags) _____

Top of Well Screen (ft. bgs) _____

Groundwater (Static) _____

Depth (ft. TOC) _____

Date Measured _____

Bottom of Well Screen (ft. bgs) _____

Bottom of Filter Pack (ft. bgs) _____

Bottom Seal Thickness (ft.) None

Borehole Diameter (in) _____

Well Cap (Type) _____

Ground Surface

Well Casing Type _____

Diameter (in.) _____

Grout Type _____

Amount (bags) _____

Seal Type _____

Amount (bags) _____

Top of Primary FIlter Pack (ft. bgs) _____

Type _____

Amount (bags) _____

Well Screen Type _____

Diameter (in.) _____

Slot Size (in.) _____

Bottom of End Cap (ft. bgs) _____

Type _____

Total Depth of Boring (ft. bgs) _____

Innovative Technical Solutions, Inc.

2730 Shadelands Drive, Suite 100
Walnut Creek, California 94598
(925) 946-3100 (Tel), (925) 256-8998 (Fax)

PROJECT NAME:

PROJECT NUMBER:

SITE LOCATION:

DAILY ACTIVITY REPORT

DATE:

PAGE OF

DESCRIPTION OF FIELD ACTIVITIES AND EVENTS

PREPARED BY:	REVIEWED BY:
DATE:	DATE:
PREPARERS SIGNATURE:	REVIEWERS SIGNATURE:

* *Not appropriate for a field activity report when only one responsible person is in the field.*

INSTRUMENT CALIBRATION RECORD

Date	Instrument	Model	Serial Number	Standard Used	Adjustment Required ?	Measured Values Before Adjust/After Adjust	Calibrated by

Comments_____

Repair/rejection Comments_____

ITSI Innovative Technical Solutions, Inc.

SAP ATTACHMENT 2
SAMPLE QC FORMS

CORRECTIVE ACTION REQUEST FORM	
Project Name & Number:	
Document Control Number:	
Date of Problem:	Originator:
Description of Problem and Effect on System:	
Person Notified:	Title & Date:
Person Notified:	Title & Date:
Description of Corrective Action:	
Person Completing Action:	
Signature:	Title & Date:
Approval:	Title & Date:

Innovative Technical Solutions, Inc.

NON ROUTINE OCCURENCE REPORT

NRO Number:	Project:	ITSI Project No.:	Date:

PART A: Non Routine Occurrence Description (include specific requirement):

Identified by: Date:

Root cause:

PART B: Corrective Action to be taken (include date when action(s) will be completed:

Performed by:

PART C: Action to be taken to preclude occurrence:

Performed by:

PART D:

Acceptance by: _____ Date: _____
Project Manager

Acceptance by: _____ Date: _____
Quality Assurance Manager

Corrective Action(s) completed by:	Verification completed by:

SAP ATTACHMENT 3

ITSI SOPs

Standard Operating Procedure

Shallow Soil: Drive Sampler, Hand Auger or Test Pit

PR-TC-02.02.01.02

Effective Date: 1 October 2009

Prepared by: *Approved by e-mail* Date: 1 October 2009
Carlton Holte

Reviewed by: Date: 1 October 2009
Clare Gilmore, Senior Geologist

Approved by: Date: 1 October 2009
Jeffrey Hess, Program Director

Revision History:

Version	Changes	Affects Section/Pages	Effective date
1.0	Initial Issue		1 Oct 2009

Table of Contents

1.0 PURPOSE AND SCOPE

The purpose of this standard operating procedure is to describe the methods and procedure for sampling of shallow soils and other solids using drive sampler, hand auger, or test pit techniques.

Scope and Limitations
Hand drive sampler and hand auger sampling can be used when matrices are composed of relatively soft and non-cemented formations, to reach depths of up to 5-10 feet below ground surface, depending on site conditions. Test pits can be dug to much greater depths than can be reached by hand augering methods.

Note: Samples for VOC analysis should not be collected via hand auger methods. However, a hand auger may be utilized to penetrate to and expose the undisturbed material at the desired depth for sampling by more applicable methods.

2.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

Hand Auger. A sample collection device consisting of metal rods with a T-bar handle and a detachable metal head. The auger head is a hollow metal tube with two cutting edges at the bottom curved into each other to hold the material pushed up into the tube as the auger is forced deeper. All trace environmental samples should be collected using stainless steel auger heads. See ASTM D1452 for a description of various types of augers available for use.

Mud Auger. A type of auger head with the top several inches open at the sides to allow for reduction of suction during removal from wetted and highly plastic materials, such as mud and lagoon solids.

Sand Auger. A type of auger with the cutting edges bent toward and touching each other. The design allows for the trapping of loosed materials in the auger tube.

3.0 PROCEDURES

The intent of these procedures is to establish consistent and repeatable steps to be taken to assure that shallow soil samples are collected efficiently and that the samples accurately reflect current conditions for the location and matrix from which they are collected.

3.1 COLLECTION OF UNDISTURBED SOIL SAMPLE USING A DRIVE SAMPLER

The following steps should be followed to collect samples of undisturbed soil using a hand-driven drive sampler:

1. Don a pair of clean gloves.
2. If desired, place plastic sheeting around the targeted location to keep sampled material in place. Use a knife or scissors to cut an access hole for the sample location.
3. Remove any surficial debris (e.g., vegetation, rocks, twigs) from the sample location and the surrounding area.
4. If the desired sample interval is deeper than 0-6-inches, then auger to the top of the desired depth interval using a hand auger. Place the bucket of the hand auger on the ground with the teeth down, and, while holding the T-handle, rotate it in a clockwise direction while pushing straight downward until the bucket is full.
5. Extract the auger by pulling upward with a slight rocking or rotating motion (counterclockwise) until the head is fully out of the hole.
6. Measure the depth of the sample bottom with the rule or tape and compare to the desired sampling depth interval. If not at the top of the desired sample depth interval, continue deeper using the hand auger.
7. Remove the soil from the [bucket of the] auger with a spoon or scoop, and empty the auger bucket onto the ground or plastic and repeat steps 4-6 until the top of the desired sample interval is reached.
8. Using a clean drive sampler equipped with clean sample sleeve(s), drive the sampler head to the bottom of the desired sample interval (typically 6 inches) is reached. Remove the drive sampler from the ground and remove the sample sleeve(s) from the drive head.
9. If collecting a sample for VOC analysis, collect the sample from one end of the undisturbed material in the sample sleeve(s) using an approved VOC sampling device.
10. Place teflon tape and friction cap on the ends of the sample sleeve(s). Mark which end represents the top of the sample interval. Clean off the sample sleeve(s), then place sample labels directly on the sleeve(s), complete documentation, place the sample containers in a Ziplock® Freezer Bag or equivalent, and place into the sample cooler.
11. Measure the depth interval from which the sample was taken and record this interval (both top and bottom depth) along with the GPS coordinates in the field logbook or on the sample collection log.

12. Repeat steps 4-10 for deeper samples from the same hole.

3.2 COLLECTION OF A DISTURBED SOIL SAMPLE USING A HAND AUGER

The following steps should be followed to collect samples of undisturbed soil using a hand auger:

1. Don a pair of clean gloves.
2. If desired, place plastic sheeting around the targeted location to keep sampled material in place. Use a knife or scissors to cut an access hole for the sample location.
3. Remove any surficial debris (e.g., vegetation, rocks, twigs) from the sample location and the surrounding area.
4. Place the bucket of the hand auger on the ground with the teeth down, and, while holding the T-handle, rotate it in a clockwise direction while pushing straight downward until the bucket is full.
5. Extract the auger by pulling upward with a slight rocking or rotating motion (counterclockwise) until the head is fully out of the hole.
6. Measure the depth to the bottom of the auger hole with the rule or tape and compare to the desired sampling depth interval. If not at the top of the desired sample depth interval, continue deeper using the hand auger.
7. Remove the soil from the auger with a spoon or scoop, or empty the auger bucket onto the ground or plastic and repeat steps 4-6 until the top of the desired sample interval is reached.
8. Using a clean auger, continue deeper until the bottom of the desired sample interval is reached and confirmed by measuring. Place the soil from the entire sample interval into a clean stainless steel bowl.
9. If collecting a sample for VOC analysis, collect the sample from the relatively undisturbed material in the bowl using an approved VOC sampling device.
10. Homogenize the non-VOC sample [following the procedure outlined in SOP PR-TC-02.02.01.04] and transfer the sample directly into the sample container(s). Cap the sample container(s), label, complete documentation, place the sample containers in a Ziplock® Freezer Bag or equivalent, and place into the sample cooler.
11. Measure the depth interval from which the sample was taken and record this interval (both top and bottom depth) along with the GPS coordinates in the field logbook or on the sample collection log.
12. Repeat steps 4-10 for deeper samples from the same hole.

3.3 COLLECTION OF SOIL SAMPLE FROM A TEST PIT

Collecting soil samples from a test pit is generally performed by collecting soil samples from relatively undisturbed soil brought to the surface in the bucket of a backhoe or excavator. The actual sample is then collected in a manner similar to surface soil, either directly in the container (e.g., wide-mouth soil jar or sample sleeve), or using a spoon or scoop to collect the soil and

transfer to the sample container. Care should be taken to select soil from relatively large and intact chunks of soil, where possible, and from the center portion of the bucket to avoid contact with the sidewalls of the bucket.

The following steps should be followed to collect samples of relatively undisturbed soil from a test pit:

1. Don a pair of clean gloves.
2. Remove any surficial debris (e.g., vegetation, rocks, twigs) from the sample location and surrounding area until the soil is exposed. Once exposed, the soil surface is designated as "at grade", or 0 inches; sample depths are then measured from this datum.
3. Direct the backhoe or excavator to dig until the desired sample interval is reached. Stockpile soil adjacent to the test pit for use in backfilling the test pit, unless otherwise required.
4. Direct the backhoe or excavator operator to remove a scoop of soil from the sidewall of the test pit corresponding to the entire sample interval. Have the bucket brought to the surface and placed firmly on the ground so it can be approached safely.
5. Visually select a relatively intact chunk of soil in the center portion of the bucket. Use a spoon or trowel to scrape and remove the top 1/8 to 1/4 inch of soil to expose fresh soil.
6. If collecting a sample for VOC analysis, collect that sample first using an approved VOC sample device.
7. Drive a clean sample sleeve directly into the selected soil. Use a rubber or wooden mallet to drive the sleeve, if necessary. Extract the sample sleeve, cap with teflon tape and friction cap, clean and label. Alternatively, a clean wide-mouth jar can be used to scoop soil directly into the sample container, or a clean spoon or scoop can be used to collect the soil and transfer into a clean sample container.
8. Measure the depth interval where the bucket was placed to collect the soil using a rule or tape to verify the sampling depth interval along with the GPS coordinates and record in the field logbook or sample collection log.
9. Complete a test log of the test pit, including a lithologic description and plan view and cross-sectional view of the test pit showing the location of the samples.
10. Clean off the sample container(s), then place sample labels directly on the containers or sleeve(s), complete documentation, and place into the sample cooler.

3.3 EQUIPMENT

The following equipment and materials should be used when conducting hand auger sampling:

- Commercial drive sampler and/or hand auger, with the drive head and/or auger of stainless steel construction for trace environmental sampling.
- Approved VOC sampling device (if needed for collection of VOC samples).
- Engineer's rule or stiff measuring tape (marked at maximum intervals of 0.01 foot).

- Spoons or scoops (stainless steel or disposable).
- Decontaminated or dedicated stainless steel bowls (if homogenizing the samples).
- Plastic sheeting.
- Sample container(s), caps, labels, coolers, etc., as specified in the SAP or FSP.
- GPS
- Field logbook or sample collection logs.

3.4 QC SAMPLING

If sampling equipment is re-used between samples (i.e., auger, drive sampler), then equipment rinsate samples should be collected to verify proper decontamination between samples. A minimum of one equipment rinsate sample per major sampling device should be collected per event, if not more frequently (i.e., one per day).

4.0 REQUIRED DOCUMENTATION

The following records generated as a result of implementation of this procedure must be maintained as quality records.

- GPS or survey coordinates for each sample location
- Field notes
- Chains of Custody

5.0 ATTACHMENTS

None.

6.0 FORMS

None.

7.0 REFERENCES

Innovative Technical Solutions, Inc. (ITSI), 2006. *Final Chemical Data Quality Management Plan*, 8(a) Remedial Action Contract Number N68711-005-D-6403. January.

Standard Operating Procedure

Sample Handling, Packaging and Shipping

PR-TC-02.04.01.01

Effective Date: 30 September 2009

Prepared by: _____ Date: 30 September 2009
Kristen Carlyon, Program Chemist

Reviewed by: _____ Date: 30 September 2009
Evelyn Dawson, Program Chemist

Approved by: _____ Date: 30 September 2009
Jeffrey Hess, Program Director

Revision History:

Version	Changes	Affects Section/Pages	Effective date
1.0	Initial Issue		30 Sep 2009

Innovative Technical Solutions, Inc.

Standard Operating Procedure

Sample Handling, Packaging and Shipping

PR-TC-02.04.01.01 v1.1

Effective Date: 24 February 2010

Prepared by: Kristen Carlyon, Program Chemist Date: 30 September 2009

Reviewed by: Evelyn Dawson, Program Chemist Date: 30 September 2009

Approved by: Jeffrey Hess, Program Director Date: 30 September 2009

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1.0	Initial Issue	NA	30 Sep 2009	NA
1.1	Added perchlorate to the Sample Preservation and Storage Requirements Table	Attachment A	24 Feb 2010	J Hess

Table of Contents

1.0 PURPOSE

The objective of this procedure is to establish a uniform method for the handling of environmental samples. This includes using the appropriate sample containers and preservatives, following correct chain-of-custody procedures, and using appropriate sample shipment methods.

2.0 SCOPE AND APPLICABILITY

This procedure will be used during the collection and handling of all types of environmental media, including but not limited to, groundwater, surface water, soil, sediment, and air samples.

This procedure applies to the shipping and packing of all non-hazardous samples. Non-hazardous samples are those that do not meet any hazard class definitions found in 49 CFR 107-178, including materials designated as Class 9 materials and materials that represent Reportable Quantities (hazardous substances). In general, most soil, air, and aqueous samples do not meet any of DOT's hazardous materials definitions. However, samples for which screening has shown a potential hazard sufficient to meet a DOT definition or that are derived from a source known or suspected to meet a DOT definition must be packaged and shipped in accordance with applicable DOT and/or IATA requirements.

3.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

°C: degrees Celcius

Bubble wrap: Plastic sheeting with entrained air bubbles; used for protective packaging purposes.

CFR: Code of Federal Regulations

COC: Chain-of-custody

Cooler: Any hard-sided insulated container meeting DOT or IATA general packaging requirements.

DOT: U.S. Department of Transportation.

IATA: International Air Transport Association.

Packing material: Styrofoam beads ("peanuts"), or equivalent

PPE: Personal protective equipment.

QAPP: Quality Assurance Project Plan

Shipping container: *see* Cooler

VOA vial: 40-mL glass vial used for the collection of samples for volatile organic analysis.

4.0 EQUIPMENT AND MATERIALS

Equipment and materials that may be required to implement this SOP include the following:

- Bubble wrap
- Packing material
- Tape (packing tape, duct tape, or other tear-resistant material)
- Large plastic trash bags
- Ziploc bags (freezer grade, gallon and quart sizes)
- Ice
- Custody seals
- "This Side Up" arrows
- Address labels and/or airbills
- Chain of Custody forms
- Black waterproof pen (e.g., fine-point Sharpie marker).

5.0 PROCEDURE

5.1 GENERAL

The following method outlines general considerations for sample handling in the field and maintaining sample custody after collection.

Environmental samples are collected in the field in order to evaluate whether conditions in soil gas, soil, surface water, groundwater or atmosphere are hazardous. These samples therefore, should be handled with the utmost care to maintain sample integrity, so that analytical data represent field conditions as closely as possible. In addition, sample care, custody, and control are extremely important for establishing that sample integrity was maintained between field crews and the laboratory.

Details regarding collection of samples can be found in ITSI's SOP series number PR-TC-02.03.00.00 for Investigation and Sampling-Related Procedures.

General considerations for handling during sampling are:

- Always wear proper PPE when handling samples.
- Wrap sample container in a way that is both protective of the sample container and other surrounding sample containers.
- Document all procedures thoroughly in field logbooks and/or on sampling forms. There is never "too much information".

Samples must be stabilized for transport from the field to the laboratory through the use of the proper sample containerization and preservation. This is due to the potential chemical and/or biological degradation that may occur after samples are collected. Typical sample containerization and preservation are presented in Table 1. Unless otherwise indicated in the site-specific QAPP, sample containers should be cooled immediately after completion of sampling and maintained at a temperature not to exceed 4 ± 2 °C until received by the laboratory.

5.2 SAMPLE CONTAINERIZATION AND PRESERVATION

The appropriate sample container types, volumes, preservatives, and holding time requirements for soil and groundwater samples for the most commonly requested analyses are listed in Table 1, Sample Preservation and Storage Requirements.

Methods of sample preservation are intended to retard biological action, retard hydrolysis, and reduce sorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and protection from light.

All sample containers will be properly labeled and monitored for temperature control in the field and during laboratory transport and storage. Temperature blanks will be used in all coolers containing samples requiring preservation at reduced temperature (e.g., 4 ± 2 °C).

5.3 SAMPLE IDENTIFICATION AND LABELS

All samples will be properly labeled to prevent misidentification of samples. Generally, preprinted sample labels are encouraged to enhance legibility and reduce transcription errors at the laboratory. The label will be affixed to the sample container prior to transportation to the laboratory and will contain the following information:

- Project name, number, and location
- Site name
- Name of collector
- Date and time of collection
- Sample identification number
- Preservative, if any
- Requested test methods or analyses.

See SOP PR-TC-01.04.04.00 for sample identification protocols.

5.4 CHAIN OF CUSTODY

Chain-of-custody (COC) procedures are implemented to ensure that all samples are traceable from the time that they is collected until they, or their derived data, are used. A sample is considered to be “in custody” under the following conditions:

- It is in personal possession.

- It is in personal view after being in personal possession.
- It was in personal possession when it was properly secured.
- It is in a designated secure area.

Sample custody will be documented through the use of COC forms. These forms will be used to track sample custody from the point of sample collection through sample disposal. The security of samples will be ensured by the use of the procedures described below.

5.4.1 Chain-of-Custody Forms

A COC form will be filled out for and will accompany every group of samples sent to the analytical laboratory, to document sample care, custody, and control from the time of collection to sample receipt.

The following information will be recorded on the COC form:

- COC form number
- Company name, address, and telephone number
- Company contact person
- Laboratory name, address, and telephone number
- Laboratory contact person
- Sample identification
- Date and time of collection
- Sampler's name
- Analytical method(s) requested
- Sample volume (e.g., three 40-milliliter [mL] vials)
- Sample matrix (e.g., soil or groundwater)
- Preservative (e.g., hydrochloric acid [HCl])
- Request for matrix spike analysis or other QC analysis
- Signatures of individuals releasing and accepting samples
- Times of release and acceptance of samples
- Air bill number if shipping by commercial courier
- Any comments to identify special conditions or requests.

5.4.2 Custody Seals

Custody seals will be used when samples are shipped via courier service, and must be placed on the shipping container so that the seals have to be broken before the container can be opened. The seal must be signed and dated by the field personnel. Custody seals are not deemed

necessary when the samples will be in the continuous possession of project, field, or laboratory personnel.

5.5 PACKAGING FOR SHIPMENT

Samples will be packaged for shipment as follows:

- Use tape to seal off the cooler drain on the inside and outside to prevent leakage.
- Place packing material (bubble wrap and/or other adsorbent material) on the bottom of the shipping container (cooler) to provide a soft impact surface.
- Place a 55-gallon or equivalent plastic bag into the cooler (to minimize the possibility of leakage during transit).
- Place each sample bottle or set of volatile organic analysis (VOA) vials in a separate plastic bag and seal the bag. Squeeze air from the bag before sealing.
- Starting with the largest glass containers, wrap each container with sufficient bubble wrap to ensure the best chance to prevent breakage of the container.
- Pack the largest glass containers in bottom of the cooler, placing packing material between the containers to partially cover the sample containers (more than halfway) to avoid breakage from bumping. Cardboard separators may be placed between the containers at the discretion of the shipper.
- Double-bag ice chips or cubes in gallon or quart freezer-grade Ziploc plastic bags and wedge the ice bags between the sample containers.
- Add bagged ice across the tops of the samples.
- Continue filling the shipping container in the same manner (e.g., using bubble-wrap and ice) with smaller sample containers/vials.
- When the container is sufficiently full (or all samples have been packed), seal the inner protective plastic bag (with twist-ties and/or packing tape), and place additional packing material on top of the bag to minimize shifting of containers during shipment.
- Tape a gallon Ziploc bag to the inside of the cooler lid, place one copy of the completed COC document for the shipment inside, and seal the bag shut.
- Tape the shipping container (cooler) shut using packing tape, duct tape, or other tear-resistant adhesive strips. Taping should be sufficient to ensure that the lid will not open during transport.
- Place custody seals on two separate portions of the cooler, to provide evidence that the lid has not been opened prior to receipt by the intended recipient.

5.5.1 Labeling

Label the shipping container/cooler as follows:

- Attach a “This Side Up” arrow securely to each side of the cooler. Affix "fragile" or other labels on the cooler as appropriate.

- Attach a label with the name and address of the receiver and the shipper to the top of the cooler.
- If the cooler is to be shipped by overnight carrier, attach a properly completed airbill to the top of the cooler.

6.0 ATTACHMENTS

- Attachment A: Sample Preservation and Storage Requirements

7.0 FORMS

The following forms are attached:

- Chain of Custody Form

8.0 REFERENCES

ITSI, 2006. *Final Chemical Data Quality Management Plan, 8(a) Remedial Action Contract Number N68711-005-D-6403*. January.

U.S. Army Corps of Engineers (USACE), 2001. *Requirements for the Preparation of Sampling and Analysis Plans.* EM200-1-3.

U.S. Department of Transportation Regulations, 49 CFR Parts 108-178.

International Air Transport Association (IATA), Dangerous Goods Regulations, current edition.

Attachment A

Sample Preservation and Storage Requirements
PR-TC-02.04.01.01 v1.1

Matrix	Analytical Group	Preparation* / Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Water	VOC	Gasoline Range Organics (GRO) 5030B / 8015B	3 X 40 mL VOA vials with Teflon septa	HCL to pH < 2 / 4°C	14 days analysis
Water	VOC	GCMS VOCs 5030B / 8260B	3 X 40 mL VOA vials with Teflon septa	HCL to pH < 2 / 4°C	14 days analysis
Water	VOC	GC VOCs 5030B / 8021B	3 X 40 mL VOA vials with Teflon septa	HCL to pH < 2 / 4°C	14 days analysis
Water	SVOC	Phenols 3510C, 3520C / 8041A	2 X 1.0 liter amber glass with Teflon liners	4°C	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Organochlorine Pesticides 3510C, 3520C / 8081A	2 X 1.0 liter amber glass with Teflon liners	4°C	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Polychlorinated Biphenyls (PCBs) 3510C, 3520C / 8082	2 X 1.0 liter amber glass with Teflon liners	4°C	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Organophosphorus Pesticide 3510C, 3520C / 8141A	2 X 1.0 liter amber glass with Teflon liners	4°C	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Chlorinated Herbicides 8151A / 8151A	2 X 1.0 liter amber glass with Teflon liners	4°C	7 days extraction 40 days analysis (after extraction)
Water	SVOC	GCMS SVOC 3510C, 3520C, 3535A / 8270C	2 X 1.0 liter amber glass with Teflon liners	4°C	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Dioxins and Furans 8280A / 8280A; 8290 / 8290	2 X 1.0 liter amber glass with Teflon liners	4°C	30 days extraction 45 days analysis (after extraction)
Water	SVOC	Polycyclic Aromatic Hydrocarbons 3510C, 3520C / 8310	2 X 1.0 liter amber glass with Teflon liners	4°C	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Nitroaromatics and Nitramines 8330A / 8330A; 8330B / 8330B	2 X 1.0 liter amber glass with Teflon liners	4°C	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Diesel and Oil Range Organics (DRO and ORO) 3510C, 3520C / 8015B	2 X 1.0 liter amber glass with Teflon liners	4°C	7 days extraction 40 days analysis (after extraction)
Water	Metals	ICP-AES Metals 3005A, 3010A, 3015A, 3020A, 3050B, 3051A / 6010B	1 X 500 mL plastic (HDPE)	NO3 to pH < 2	6 months analysis
Water	Metals	ICP-MS Metals 3005A, 3010A, 3015A, 3020A,	1 X 500 mL plastic (HDPE)	NO3 to pH < 2	6 months analysis

Attachment A

Sample Preservation and Storage Requirements
PR-TC-02.04.01.01 v1.1

Matrix	Analytical Group	Preparation* / Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
		3050B, 3051A / 6020			
Water	Metals	Mercury by CVAA 7470A / 7470A	1 X 500 mL plastic (HDPE)	NO3 to pH < 2	28 days analysis
Water	Inorganic	Hexavalent Chromium 7196A / 7196A 7197 / 7197	1 X 250 mL plastic HDPE)	4°C	24 hours analysis
Water	Inorganic	Anions by IC 300.0 / 300.0 9056A / 9056A	1 X 250 mL plastic HDPE)	4°C	48 hours for nitrate, nitrite, and orthophosphate analysis 28 days for chloride, sulfate, bromide, and fluorideanalysis
Water	Inorganic	Nitrate and Nitrite as N Total 353.2 / 353.2	1 X 250 mL plastic (HDPE)	H2SO4 to pH <2 / 4°C	28 days analysis
Water	Inorganic	Kjeldahl Nitrogen 351.2 / 351.2	1 X 250 mL plastic (HDPE)	H2SO4 to pH <2 / 4°C	28 days analysis
Water	Inorganic	Chemical Oxygen Demand (COD) 410.2 / 410.2	1 X 250 mL plastic (HDPE)	H2SO4 to pH <2 / 4°C	28 days analysis
Water	Inorganic	Alkalinity SM2320B / SM2320B 310 / 310	1 X 250 mL plastic (HDPE)	4°C	14 days analysis
Water	Inorganic	Total Dissolved Solids (TDS) SM2540C / SM2540C 160.1 / 160.1	1 X 250 mL plastic (HDPE)	4°C	7 days analysis
Water	Inorganic	pH SM4500-H+B / SM4500-H+B 150.1 / 150.1	1 X 250 mL plastic (HDPE)	4°C	24 hours analysis
Water	Inorganic	Conductivity SM2510B / SM2510B 120.1 / 120.1	1 X 250 mL plastic (HDPE)	4°C	28 days analysis
Water	Inorganic	Perchlorate 6850 / 6850	1 X 125-ml plastic (HDPE)	20°C/ Store sample with headspace	28 days analysis
Soil	VOC	Gasoline Range Organics (GRO) 5035 / 8015B	3 X 5g EnCore®	4°C or frozen	analysis - 24 hours or 7 days if frozen

Attachment A

Sample Preservation and Storage Requirements
PR-TC-02.04.01.01 v1.1

Matrix	Analytical Group	Preparation* / Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Soil	VOC	GCMS VOCs 5035 / 8260B	3 X 5g EnCore®	4°C or frozen	analysis - 24 hours or 7 days if frozen
Soil	VOC	GC VOCs 5035 / 8021B	3 X 5g EnCore®	4°C or frozen	analysis - 24 hours or 7 days if frozen
Soil	SVOC	Phenols 3540C, 3550C / 8041A	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	4°C	extraction - 14 days analysis - 40 days
Soil	SVOC	Organochlorine Pesticides 3540C, 3541, 3550C / 8081B	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	4°C	extraction - 14 days analysis - 40 days
Soil	SVOC	Polychlorinated Biphenyls (PCBs) 3540C, 3541, 3550C / 8082	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	4°C	extraction - 14 days analysis - 40 days
Soil	SVOC	Organophosphorus Pesticides 3540C, 3541, 3550B / 8141A	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	4°C	extraction - 14 days analysis - 40 days
Soil	SVOC	Chlorinated Herbicides 8151A / 8151A	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	4°C	extraction - 14 days analysis - 40 days
Soil	SVOC	GCMS SVOCs 3540C, 3541, 3545A, 3550C, 3560, 3561 / 8270C	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	4°C	extraction - 14 days analysis - 40 days
Soil	SVOC	Dioxins and Furans 8280A / 8280A; 8290 / 8290	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	4°C	extraction - 30 days analysis - 45 days
Soil	SVOC	Polycyclic Aromatic Hydrocarbons 3540C, 3550C / 8310	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	4°C	extraction - 14 days analysis - 40 days
Soil	SVOC	Nitroaromatics and Nitramines 8330A / 8330A;	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	4°C	extraction - 14 days analysis - 40 days
Soil	SVOC	Nitroaromatics and Nitramines 8330B / 8330B	1.5 grams of soil in specially prepared locking plastic bag	4°C	extraction - 14 days analysis - 40 days
Soil	SVOC	Diesel and Oil Range Organics 3540C, 3541, 3545A, 3550C, 3560 / 8015B	brass, stainless steel or teflon™ sleeves with teflon™ end caps or 4 oz glass jar	extraction - 14 days analysis - 40 days	extraction - 14 days analysis - 40 days

Attachment A

Sample Preservation and Storage Requirements
PR-TC-02.04.01.01 v1.1

Matrix	Analytical Group	Preparation* / Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Soil	Metals	ICP-AES 3050B / 6010B	1 X 4 oz glass jar	4°C	analysis - 6 months
Soil	Metals	ICP-MS 3050B / 6020	1 X 4 oz glass jar	4°C	analysis - 6 months
Soil	Metals	Mercury by CVAA 7471A / 7471A	1 X 4 oz glass jar	4°C	analysis - 28 days
Soil	Inorganics	Conductivity 9050A/ 9050A	1 X 4 oz glass jar	4°C	analysis - 28 days
Soil	Inorganics	Hexavalent Chromium 7196A / 7196A 7197 / 7197	1 X 4 oz glass jar	4°C	analysis - 24 hours
Soil	Inorganics	pH 9045D / 9145D	1 X 4 oz glass jar	4°C	analysis - 24 hours
Soil	Inorganics	Perchlorate 6850 / 6850	1 X 4 oz amber jar	< 20 °C, Store with headspace	analysis - 28 days

*Extraction methods are not limited to the ones listed in the table above. Please refer to the individual methods, EPA Method 3500 and Chapters 2 and 3 from (SW846 for guidance in choosing the appropriate extraction procedure.

Abbreviations:
AES = Atomic Emission Spectrometry
°C = degrees centigrade
CVAA = Cold Vapor Atomic Absorption
GC = Gas Chromatography
HCl = Hydrochloric Acid
H2SO4 = Sulfuric Acid
IC = Ion Chromatography
ICP = Inductively Coupled Plasma
mL = milliliters
MS = Mass Spectrometry
oz = ounce
SVOC = Semi-volatile Organic Compounds
VOA = Volatile Organic Analysis
VOC = Volatile Organic Compounds

Standard Operating Procedure

Field Analytical Methods: Innov-X Alpha 4400 Portable X-Ray Fluorescence Analyzer (XRF) for Metals in Soil

PR-TC-02.05.02.01

Effective Date: 30 September 2009

Prepared by:	*Approved by e-mail* Rogerio Leong	Date:	30 September 2009
Reviewed by:	*Approved by e-mail* Dennis Rich	Date:	30 September 2009
Reviewed by:	Kristen Carylon, Program Chemist	Date:	30 September 2009
Approved by:	Jeffrey Hess, Program Director	Date:	30 September 2009

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Version	Changes	Affects Section/Pages	Effective date
1.0	Initial Release		30 Sep 2009

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1.0 PURPOSE AND SCOPE

The purpose of this standard operating procedure is to describe the steps required for the start-up, operation, calibration, and routine use of the Innov-X Systems Alpha Series (Innov-X Alpha) hand-held field portable x-ray fluorescence (XRF) instrument. The Innov-X Alpha XRF instrument is a hand-held, battery-operated energy dispersive x-ray fluorescence analyzer used in the detection and quantification of metals and other elements. The analyzer is a direct readout instrument that does not require external calculations. Typical environmental applications include: detection/quantification of heavy metals in soil (in-situ or samples collected from the surface or with the subsoil probe); sediments, and sludge; and detection/quantification of heavy metal air particulates collected on membrane filters, either from personal samplers or from deployable particulate samplers.

Although the Innov-X Alpha can be used to screen for a variety of metals and lead-based paint in different media, the appropriate software must be installed and factory calibrated for the different uses. Currently ITSI has two units, both of which are set up for metals in soil by EPA Method 6200 (antimony, arsenic, barium, cadmium, chromium, cobalt, copper, lead, mercury, nickel, selenium, silver, thallium, tin, vanadium, and zinc), and one of which is also set up for mining-related elements (gold, silver, and platinum group metals) and lead-based paint testing. Additionally, ITSI has a test stand for fixed operation, along with software for operation of the unit while in the test stand via a portable computer instead of the onboard Pocket PC.

This SOP covers the operation of the unit for metals testing in soil. Other uses of the Innov-X Alpha (e.g., lead-based paint testing) will be covered in a separate SOP. Specific procedures covered in this SOP include the following methods of testing metals in soil:

- **In-situ soil testing**: Performing soil testing directly on the surface of the ground. Operators must remove any plant growth, rocks and/or other foreign objects so that the analyzer probe is flush to the surface of the ground.
- **Ex-situ bagged soil testing**: Collecting a soil sample in a thin plastic bag (i.e., a "baggie") and testing directly through the baggie.
- **Ex-situ prepared soil testing**: Preparing the sample through drying (if moisture content is above 20%); sieving in a #10 mesh sieve; and thoroughly homogenizing it. The prepared sample is then placed back into an XRF cup or baggie for analysis.

Note: The XRF analyzer shall only be used by properly trained (and licensed, as appropriate) operators. Required training includes, but is not limited to, training specific to the Innov-X Alpha offered by the manufacturer.

2.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

GPS: global positioning system
LOD: Level of detection
NIST: National Institute of Standards and Testing
USACE: United States Army Corps of Engineers
USEPA: U.S. Environmental Protection Agency
XRF: X-ray fluorescence

3.0 PROCEDURES

XRF spectroscopy is a non-destructive, qualitative and quantitative analytical technique used to determine the chemical composition of samples. The Innov-X Alpha is a complete, hand-held, portable x-ray tube-based analyzer powered from a rechargeable lithium ion battery. Under ideal conditions, the battery will provide 4 to 8 hours of use before re-charging is necessary.

To analyze a sample, the sample is positioned in front of the x-ray tube/detector window. The trigger on the unit is engaged or the appropriate button on the iPAQ unit is tapped to initiate the analysis; this exposes the sample to x-rays emitted by the tube. The energy from the x-rays excites electrons within the sample, causing the atoms that comprise the elements in the soil sample to become unstable. Since these atoms seek stability, electrons in each atom of the elements emit energy to regain the stability lost when they became excited. This is called fluorescence. The fluorescence produced is characteristic of a specific element. Fluorescent and backscattered x-rays from the sample enter the unit through the detector window. They are counted by the detector, and then analyzed by the detachable iPAQ computer. The specific elements detected in the soil and their concentrations are then stored in memory and displayed on the iPAQ screen.

3.1 GENERAL INSTRUMENT PROCEDURES

3.1.1 Instrument Handling

The manufacturer recommends operating the unit within the following conditions:

- Temperature range: 0 to 40 °C (32° - 104°F)
- Humidity range: 10% to 90% relative humidity (with no condensation)
- Altitude: below 2,000 meters (approximately 6,600 feet).

The Innov-X Alpha should always be stored in its waterproof, drop-proof carrying case. The battery charger should only be used in dry conditions. Battery packs should be changed only in dry conditions.

Sample moisture has two effects on results: 1) it alters the soil chemistry, since water is another chemical compound that comprises the soil matrix; and 2) it impedes the ability to properly prepare samples.

3.1.2 Instrument Start-up

(NOTE: Prior to operating the Innov-X Alpha, make sure the iPAQ is charged.) Insert a fully charged battery pack into the Innov-X Alpha unit's handle. The charge on the battery can be tested by pressing the "check" button on the side of the battery and noting the LED reading. Make sure the battery is fully inserted, seats properly and the battery housing latch can close.

Complete the following steps to prepare and set up the Innov-X Alpha unit for use:

- Slip the iPAQ into the slot on top of the XRF and ensure that the connecting pins are engaged. (Do not push with force, as the pins can break).
- Power the analyzer on and let it warm up for at least 15 minutes. (The power switch is located on the left side of the back of analyzer).
- Power on the iPAQ by depressing the On/Off button located in the upper right-hand corner of the iPAQ.
- Using the iPAQ's stylus, scroll to the XRF software by clicking "START" and then "INNOV-X." to initiate the Innov-X program. When the "NOTICE" screen appears, select and tap the "START" icon to begin the hardware and software self-testing process n. This will take approximately 60 seconds. Once the unit has been initiated and tested/verified, the "MAIN MENU" screen will appear. From this screen, select the appropriate analytical mode from the menu, or select additional mode options from the "MODE" menu located at the bottom of the screen.
- Standardize the instrument according to the procedures outlined in Section 3.4 below..
- Release the software trigger lock by tapping the "locked" icon (bottom right of the screen) on the iPAQ and tapping "yes" in response to the prompt. (Reminder: the standardization clip must be removed before samples can be analyzed.
- Analyze NIST calibration standards to verify instrument performance.
- The unit is now ready to perform field/sample analyses.

Note: For information on use of the unit's Bluetooth wireless capabilities, see Appendix B.

3.1.3 Basic Operation

The Innov-X Software is controlled through three main screens:

- **Main Menu Screen**: The main menu appears upon startup. It is used to select the analysis mode, open the results screen, and perform certain administrative functions such as changing your login password..

Use the Main Menu to select the desired analysis mode (Soil). The analysis mode can be selected by either tapping on the name of the method (shown in blue) or by selecting the appropriate mode from the Modes menu.

It is possible to go directly from the Main Menu screen to the Results screen by selecting "*View→Results*". If the Results screen is opened in this manner, it is possible to view results when the iPAQ is not connected to the analyzer.

- **Analysis Screen**: This screen is used to change settings, edit libraries, and perform tests. Selecting a mode from the Main Menu screen opens the Analysis window for that mode. All data acquisition and analysis is controlled from this window. This window allows the user to start or stop an analysis, change testing parameters, and modify the fingerprint.

  (NOTE: The Analysis screen runs continually during normal instrument operation. From the Results menu, it is always possible to go back to the Analysis screen by selecting "*File→Exit*" or by tapping the X in the upper right hand corner of the screen.)

  The Analysis screen displays the name of the mode that is currently active, a Start/Stop icon (which is inactive in most cases), an Information button that is selected to enter descriptive information for any given test, a trigger lock, and a battery indicator. In addition, a message appears directly below the name of the mode to indicate the current state of the analyzer. Typically it reads "*Ready to Test,*" but it provides other information in certain circumstances. Any mode-specific information will be displayed at the bottom of the screen, above the available menu choices.

- **Results Screen**: This screen displays results from current reading, allows scrolling back to previous test results, and allows recorded data to be exported to a comma-delimited file that is compatible with Microsoft Excel.

  The Results screen displays the current reading or old/stored data. All data-handling functions (such as exporting and deleting readings) are carried out from this screen. Once the Results screen is open, the user may start a new test without going back to the Analysis screen by pulling and holding the trigger on the Innov-X Alpha unit. Tapping the X in the upper right hand corner of the Results screen will return the user to the Analysis screen without starting a test. If no analysis mode is running, an Exit button will appear; selecting "Exit" will close the Results screen.

3.1.4 Standardization and Calibration

Before performing tests, it is necessary to standardize the instrument. This automated procedure involves collecting a spectrum on a known standard (Alloy 316) and comparing a variety of parameters to values stored when the instrument was calibrated at the factory. If there are any problems with the instrument, they will be indicated by an error message. The standardization procedure takes about one minute. Standardization must be done any time the analyzer hardware is initiated or restarted and must be repeated if the instrument is operating for more than 4 hours.

Each time the analyzer is restarted, the operator will be prompted to standardize the instrument before performing any measurements. This is indicated by the message "*Standardization Required. Please place a standardization clip over the analyzer window. Then tap here to standardize.*" on the Analysis screen.

To initiate the standardization procedure, snap the standardization clip on the front of the instrument. Verify that it completely covers the analyzer window. Tap the gray box in the center of the iPAQ screen or select "*File→Standardize*" to begin. When standardization is in progress, the red light on the top of the instrument will blink, indicating that the X-ray tube is energized and the shutter is open. In addition, a status bar will appear, tracking the progress of the measurement. When standardization is complete, the message "*Successful Standardization*" will appear, along with the resolution of the instrument. Tap "OK" to acknowledge and clear the message. The instrument is now ready for sample evaluation.

Standardization Errors - The analyzer performs several diagnostic checks during the standardization process. If the standardization fails, the instrument will prompt the user regarding the next step. When standardization fails, verify that the standardization clip is in place, and attempt standardization again. To standardize after a failure, tap the gray box in the center of the display, or choose "*File→Standardize*". If standardization fails again, exit the analysis screen and power off the instrument. Restart and re-standardize. If the standardization fails a third time, you will be prompted to perform a soft reset of the iPAQ. Selecting "*Yes*" on this screen automatically initiates a soft reset the iPAQ. When this is complete, power down the analyzer, restart, and re-standardize. If the standardization fails again, replace the battery in the instrument and attempt standardization again. If this fails, contact the Innov-X Systems service center at (866) 446-6689 or via email at technicalsupport@innovxsys.com.

Calibration Checks – Several NIST certified standards (See Appendix B for certified values of the NIST Standards provided) are kept with the analyzer for calibration verification. Prior to initiating soil testing (and periodically throughout a day's testing), a low, medium, and high standard should be measured for a minimum of two minutes each, depending on the elements being analyzed. Elemental concentrations for elements of interest plus or minus the error on the reading should be within 20% of the standard values. The standards provided with the XRF analyzer are contained in XRF sample cups with a Mylar window (through which the soil can be viewed) on one side, and a solid cap on the other side. Samples should be measured in the sample cup, through the Mylar window. The best way to measure a prepared sample is using the test stand.

3.1.5 Sample Testing

After the instrument has been standardized and the appropriate minimum and maximum test times have been entered using the Analysis screen, testing can begin. Simply pull the trigger or select "*Start*" on the iPAQ screen to begin the test. The red warning light on the top of the instrument will blink, indicating X-rays are being emitted. The screen will display the words "*Test in progress*" and the time elapsed. The word "*Testing*" will blink on and off in the lower right-hand corner of the screen.

The Innov-X Alpha is equipped with a software trigger lock that prevents the trigger from being activated unintentionally. The lock is released by tapping the "Lock/Unlock" icon on the iPAQ screen. Once the lock is released, it will remain unlocked for subsequent tests. If more than five minutes elapses between tests, the trigger lock will re-activated and must be disabled before additional testing can be done.

Information such as a sample name/ID, and identifying characteristics can be stored with each measurement. This is done from the Test Information screen that can be accessed from the Analysis Screen for any mode by tapping the "Info" button or selecting "*Edit→Edit Test Information*". The Test Information screen consists of eight fields. (NOTE: The name and format of each field can be changed using the "*Modify Test Info Template*". Consult the operator's manual for more information on adding test information.)

After the specified minimum test time has elapsed, intermediate results will be displayed on the screen. Until the maximum test time has elapsed, the words "*WAITING FOR DATA*" will appear on the iPAQ screen. Each line of results displayed shows the name of an element, its calculated concentration, and the error in the measurement. The error will decrease with increased testing time. Minimum and maximum test times can be adjusted by the user by selecting "*Options→Set Testing Times*". A screen appears prompting entry of testing times.

Too many elements are measured in the sample to display them all on a single iPAQ screen. Use the scroll bar at the right of the chemistry display to view other elements. The complete display shows detected elements first, followed by elements that are below the detection limit of the instrument. These elements are shown as less than a calculated LOD. When the measurement is complete (i.e., the maximum test time has elapsed or the trigger has been released [if a manual test was being conducted]), the Final Results screen will open, displaying the final results of the measurement.

The Final Results screen in the "*Soil Mode*" displays the concentration (in ppm) and the error in measurement for each detected element, followed by the list of non-detected elements with the calculated limit of detection for each element for that test. If the display does not show soil chemistry results, change the display by selecting "*View→Results*". The standard display can be modified by using the View menu. As with all analytical modes, it is possible to view spectra and Test Information.

The results (analytical reports) and spectra that have been stored in the Innov-X Alpha iPAQ internal or flash memory must be downloaded and captured as files on a PC to reduce loss of data. Microsoft ActiveSync® provides software for this purpose. Results or spectra may be exported to text files for importing into a spreadsheet. Refer to the operator's manual for details.

3.1.6 Instrument Maintenance

Keep the Innov-X Alpha clean and dry. When the Mylar window on the front of the instrument becomes dirty, the performance of the analyzer unit will be affected. Clean the window gently with cotton swabs. Clean the body of the instrument with a soft cloth. Never use water, detergents, or solvents, as these may damage the instrument.

For all other maintenance and repair issues, send the unit to the manufacturer. Do not attempt to make repairs yourself, as opening the case of the Innov-X Alpha voids the warranty. Also, to ensure continued safe operation, the Innov-X Alpha should be returned to the manufacturer every 2 years for performance testing, routine maintenance and/or any necessary software upgrades.

Refer to the Innov-X Alpha user manual for additional detailed operational and/or maintenance and troubleshooting instructions. Contact Innov-X Systems for any further assistance.

3.2 IN-SITU SOIL TESTING

In-situ testing is performed by placing the Innov-X Alpha directly on the ground. The procedure for in-situ testing of soils is as follows:

1. Prepare an area (12 x 12 inches) for in analysis by removing large rocks, vegetation, and debris.
2. Ensure that the soil surface in the location to be analyzed is flat and compacted.
3. Place the Innov-X Alpha firmly in contact with the soil surface (use the soil foot to hold the unit in place and maximize contact with the ground).
4. Initiate analysis; DO NOT move the unit during analysis.
5. Record the results of each analysis and save the test in the iPAQ memory for subsequent retrieval at the end of the work day.
6. Since dirt can accumulate on the analyzer window, the window should be wiped clean, and checked to ensure it is not ripped or punctured, after each analysis.

Note: Avoid analysis of water-saturated soils or sediments.

3.3 EX-SITU BAGGED SOIL TESTING

Samples are generally collected or received in labeled plastic bags, and should be prepared for testing as follows:

1. If moisture is above 20%, then dry the samples (generally by air drying in direct exposure to the sun) and return the dried sample to the labeled baggie for analysis.
2. Either (a) place the bag in the test stand (window side down), close the safety shield, and initiate the analysis, or (b) place the bag on a flat surface, place the Innov-X Alpha directly on the bag, and initiate analysis. **Never hold bagged samples while testing!**

NOTE: Results for chromium, vanadium, and barium will be lower by 20-30% when sampled through a baggie.

The test application software assumes the sample is infinitely thick. For in-situ measurements, this is generally the case. However, for bagged samples, it is important to fill the bags nearly full. Make sure that sufficient sample material exists in the bag to cover the analyzer window completely, with a sample thickness of at least one-half inch. (Additionally, ensure that sample thickness is as uniform as possible from analysis to analysis during a sampling event.)

3.4 EX-SITU PREPARED SOIL TESTING

Prepared soil samples are analyzed when maximum accuracy is required. Samples are generally received in labeled plastic bags or glass jars, and should be prepared for testing as follows:

1 If moisture is above 20%, then dry the sample by either placing it (in a glass or stainless steel bowl) in a conventional oven and drying at ### degrees, or simply air-drying it by direct exposure to the sun.

2. Thoroughly mix each sample and sieve it through a #10 sieve.

3. Place the processed sample into a new labeled baggie for analysis following the "Ex-Situ Bagged Sampling" process described above or, if using a sampling cup, then perform the following steps:

    - Fill a labeled polyethylene x-ray sample cup to the snap ring with a portion of the processed sample.
    - Seal the x-ray sample cup with 0.2-mil (5 micrometer) thick polypropylene x-ray window film.
    - Pack the sample evenly against the window film by tapping the x-ray sample cup against a tabletop or other clean, flat surface.
    - Place the sample cup in the test stand (window side down), close the safety shield, and initiate the analysis.

The test application assumes the sample is infinitely thick. For in-situ measurements, this is generally the case. However, for sample cups and/or bagged samples, it is important to fill the sample cups or bags nearly full. Fill sample cups to a depth of at least one-half inch of packed soil. When analyzing bagged samples, make sure that sufficient sample exists in the bag to cover the window completely with a sample at least one-half inch thickness, and ensure that sample thickness is as uniform as possible from analysis to analysis. The test stand should be used when analyzing samples in sample cups for the most accurate results. Never hold prepared or bagged samples while testing!

3.5 EQUIPMENT

The following basic equipment and materials are required when conducting analyses using the Innov-X Alpha in the field:

- Innov-X Alpha unit
- iPAQ unit
- Two Li-ion batteries (each battery typically last approximately 4-6 hours) and battery charger
- AC adapter system battery module
- iPAQ cradle and charger
- Standardization clip and NIST calibration cups with certified values for specific metals of interest (i.e. lead, arsenic, etc…) (Note: The Innov-X System has a number of NIST

standard test soils plus "clean" silicon dioxide [SiO_2] available for performing routine calibration checks and verifying the XRF accuracy.)

- Spare analyzer window
- Optional: Soil foot (tripod for placing unit directly on soil surface for in-situ testing)
- Optional: Test stand (bench–top unit for ex-situ testing only)
- Optional: PC laptop equipped with software for remote operation of the unit.

3.6 QC SAMPLING

The following general QA procedures apply to all XRF analyses:

- All sample data, pre-operational calibration/standardization, and operational checks must be documented in the instrument utilization or analysis logs.
- The instrument must be operated in accordance with this SOP and the manufacturer's recommendations.
- Preventive maintenance is conducted at the intervals recommended by the manufacturer. An instrument utilization log must be maintained to document specific corrective actions taken to alleviate any instrumental problems, and for recording all service performed.
- Results must be saved electronically on a hard drive or portable media.
- Standardization Check. The self-standardization must be performed each time the instrument is used, and every four hours during sample analysis, to maintain proper detector calibration.
- Target Element Calibration Check. The purpose of the target element calibration check is to ensure that the instrument and the selected application are working properly prior to performing sample analysis. This check should be performed at the beginning of the day and after every tenth sample analyzed. Use the NIST SRM 2709, 2710, and 2711 standards provided with the unit to check the Soil Mode application. These samples should be measured using the same acquisition times used for sample analysis. Save the sample check results/spectra for documentation.
- Detection of Matrix Interference. To determine if analyses are being influenced by matrix interference, a repeat analysis shall be performed on 20% of the tests performed in situ or in accordance with project-specific requirements. If the variation between the initial and repeat sample analyses at a sampling point is greater than 20%, then the analysis must be repeated on an ex-situ prepared sample. Sampling locations for repeat analysis should be selected at random. Additional repeat analyses may also be performed when there are observable differences in soil particle size at a sampling location.
- Confirmation Analysis. Confirmatory analyses on a subset of the screening samples (minimum 10%) shall be used to determine if the XRF data meet the Target Sampling Quality Objectives for sampling activities. Confirmatory samples should ideally be selected randomly from the sample set. Confirmatory samples should be submitted to an accredited laboratory. Samples selected for performing a comparison between XRF and laboratory results should first be thoroughly homogeneized for the entire bag of sample,

and then a sub sample with sufficient volume (as required by the laboratory) should be collected from the bag for XRF testing. After performing XRF testing, the same sub sample should be sent to the laboratory for analysis.

3.7 POTENTIAL INTERFERENCES

Total method error for XRF analysis is a combination of instrument precision and user- or application-related error. Instrument precision is typically the least significant source of error in XRF analysis. User- or application-related error is generally more significant, and will vary with each site and the method(s) used. The most common user- and application-related errors are discussed below.

Sample Placement

This is a potential source of error because the X-ray signal decreases as the distance from the x-ray source is increased. This error may be minimized by maintaining the same distance between the sample and the analyte window for all tests. Sample geometry with respect to the x-ray tube/detector is also important. A tilted sample may cause analytical error.

Sample Representativity

To characterize site conditions accurately, samples collected must be representative of the site or area under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in contaminant presence and concentration range throughout a site. Variables affecting sample representativeness include: (1) geologic variability, (2) contaminant concentration variability, (3) collection and preparation variability, and (4) analytical variability. Attempts should be made to minimize these sources of variability by collecting and analyzing a sufficient number of samples across the test area or area of concern.

Physical Matrix Effects

Physical matrix effects are the result of variations in the physical character of the sample and include parameters such as particle size, uniformity, homogeneity, and surface conditions. For example, consider a sample in which the analyte exists in the form of very fine particles within a matrix composed of much coarser material. If two separate aliquots of the sample are prepared in such a way that the matrix particles in one are much larger than in the other, then the relative volumes occupied by the analyte-containing particles will be different. When measured, a larger amount of the analyte will be exposed to the source X-rays in the sample containing finer matrix particles, resulting in a higher intensity reading for that sample and, consequently, an apparently higher measured concentration for that element.

4.0 SAFETY CONSIDERATIONS

The safe and proper operation of the XRF instrument is the highest priority because it generates ionizing radiation in which a direct exposure can cause serious injuries. The Innov-X Alpha is a safe instrument when used according to manufacturer's recommended safety procedures. Refer to the User Manual for a detailed discussion of radiation safety practices and precautions.

THE XRF SHOULD NOT BE POINTED AT ANY PART OF THE BODY, WHETHER OR NOT THE UNIT IS ENERGIZED!

The Innov-X Alpha must be handled in accordance with the following radiological control practices.

- Keep your hands and all body parts away from the front end of the instrument when the shutter is open. Under no circumstances should the analyzer be pointed at the operator or surrounding personnel. A red light on the top of the unit will blink when the Innov-X Alpha is active and x-rays are being emitted. (Note: Although the exposure rate directly in front of the detector window can be as high as 28 R/hr, no measurable dose to an operator is expected if the manufacturer's instructions are followed, and no interlocks are bypassed.)
- When performing an analysis, the Innov-X Alpha should always be in contact with the surface of the material being analyzed, and the material should completely cover the aperture when the x-ray tube is on (shutter is open). Do not remove a sample or move the unit while the x-ray tube is on.
- When using the Innov-X Alpha, make sure that surrounding people are at least 3 feet away.

Under no circumstances should the x-ray tube be on and emitting x-rays when the instrument is not in use. The warning lights on the instrument will blink on and off whenever the x-ray tube is on and emitting x-rays. The Innov-X Alpha should not be exposed to conditions of excess shock or vibration, nor should it be used in high moisture environments.

5.0 REQUIRED DOCUMENTATION

The following records generated during implementation of the sample analyses described in this procedure must be maintained as quality records.

- Equipment Utilization Log
- Field Analytical Forms or Log Book
- Copies of analytical reports and spectra from the iPAQ unit
- Chain-of-Custody forms for confirmation samples.

6.0 ATTACHMENTS

A. Illustrated Bluetooth Quick Start Guide

B. Certified Standard Reference Material (SRM) Values

C. Analytical Program Recovery Procedures

D. Determining Data Usability of In-Situ Testing

7.0 FORMS

None.

8.0 REFERENCES

Innov-X Systems. *Alpha Series User Manual, Version 2.1.*

U.S. Environmental Protection Agency (USEPA), Environmental Response Team (ERT), 1991. *Representative Sampling Guidance, Volume 1 – Soil*. OSWER Directive 9360. 4-10. November.

USEPA/ERT, 1991. *Field-Portable X-Ray Fluorescence*. Quality Assurance Technical Information Bulletin, Volume 1, Number 4, May.

Attachment A

Illustrated Bluetooth Quick Start Guide

Illustrated Bluetooth Quick Start Guide

This Quick Start Guide is not a substitute for reading the manufacturer's XRF manual. Using Bluetooth wireless technology enables the Innov-X Alpha operator to use more efficiently the capabilities of this instrument. A laptop computer in the field allows sample names/locations to be entered easily with a PC keyboard as compared to using a stylus. The file transfer "sync" function of the "Pocket Controller" software allows one to more efficiently transfer data from the Innov-X Alpha to a PC wirelessly without removing the iPAQ from the Innov-X Alpha body. This configuration also precludes the possibility of damaging fragile electronic connector contacts, the iPAQ unit itself and any requirement for using an iPAQ cradle & a connector cable. This configuration keeps the iPAQ securely attached to the Innov-X Alpha and enhances the instrument's field capabilities.

This Quick Start Guide requires the following:

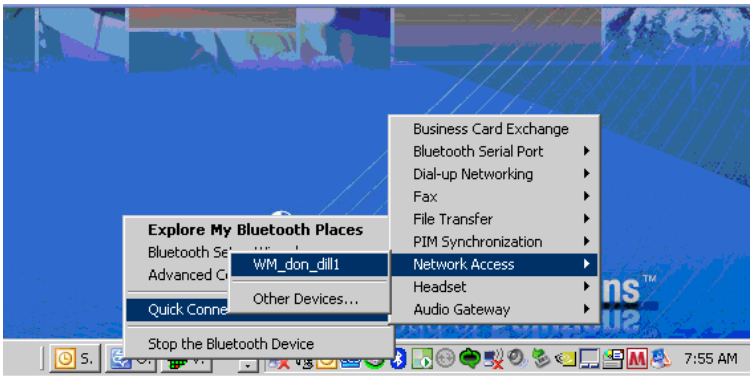
- XRF user is certified to operate the Instrument.
- Microsoft ActiveSync has been installed on computer.
- USB Bluetooth Dongle (i.e., Targus model ACB10US) is used for connectivity.
- Controller software such as SOTI, Inc. "Pocket Controller" has been installed on computer.

Step 1: Start-up

Turn on XRF & iPAQ→Let XRF warm up for 10 minutes/ Put dongle in USB port of Laptop/PC

Step 2: Bluetooth Dongle Settings

Start Bluetooth device on computer by <right clicking> on Bluetooth Icon in Tool Bar → <click> Start the Bluetooth Device (blue indicator light will turn on Dongle – non-flashing) → <click> Quick Connect → go to "Network Access" → <click><your specific computer ID>



Then, allow several minutes for Bluetooth device to acquire computer IP address. The below icon will appear while the IP address is being acquired & disappear after the IP address has been

acquired (Note: under Configure → General Tab, if the iPAQ IP is 127.0.0.1, the IP address has not been acquired – the above steps must be repeated)

Step 3: iPAQ Setup

<click> Start → <click> iPAQ Wireless → <click> Bluetooth icon (Note: Bluetooth button on iPAQ will turn Green & the indicator lights will flash Blue for Bluetooth Connectivity & Amber if connected to 115 volt charging source). Start "Pocket Controller" software → If iPAQ buttons are programmed, <click> Button #1 on iPAQ to start Innov-X software.

Attachment B

Certified Standard Reference Material (SRM) Value

Certified Standard Reference Material (SRM) Values

Certified SRM Values (in mg/kg)

Element	#2709[(1)] (Low Standard)	#2711[(1)] (Medium Standard)	#2710[(1)] (High Standard)
Antimony (Sb)	7.9 ± 0.6	19.4 ± 1.8	38.4 ± 3.0
Arsenic (As)	17.7 ± 0.8	105 ± 8	626.0 ± 38.0
Barium (Ba)	968 ± 40	726 ± 38	707.0 ± 51.0
Cadmium (Cd)	0.38 ± 0.01	41.7 ± 0.25	21.8 ± 0.2
Chromium (Cr)	130 ± 4	NA	NA
Cobalt (Co)	13.4 ± 0.7	NA	NA
Copper (Cu)	34.6 ± 0.7	114 ± 2	2950 ± 130
Lead (Pb)	18.9 ± 0.5	1162 ± 31	5532 ± 80
Manganese (Mn)	538 ± 17	638 ± 28	10100 +/- 40
Mercury (Hg)	1.40 ± 0.08	6.25 ± 0.19	32.6 ± 1.8
Nickel (Ni)	88 ± 5	20.6 ± 1.1	14.3 ± 1.0
Selenium (Se)	1.57 ± 0.08	1.52 ± 0.14	NA
Silver (Ag)	0.41 ± 0.03	4.63 ± 0.39	35.3 ± 1.5
Strontium (Sr)	231 ± 2	245.3 ± 0.7	NA
Thallium (TI)	0.74 ± 0.05	2.47 ± 0.15	NA
Vanadium (V)	112 ± 5	81.6 ± 2.9	76.6 ± 2.3
Zinc (Zn)	106 ± 3	350.4 ± 4.8	6952 ± 91

[(1)] NIST Certificates of Analysis is available on the NIST web site https://srmors.nist.gov. The terms high, medium, and low refer to the overall level of contamination within the standard, rather than the concentration of individual constituents.

NA - Not Applicable

Certified SRM Values (in weight %)

Element	#2709 (Low Standard)	#2711 (Medium Standard)	#2710 (High Standard)
Aluminum	7.5 +/- 0.06	6.53 +/- 0.09	6.44 +/- 0.08
Calcium	1.89 +/- 0.05	2.88 +/- 0.08	1.25 +/- 0.03
Iron (Fe)	3.5 +/- 0.11	2.89 +/- 0.06	3.38 +/- 0.1
Magnesium (Mn)	1.51 +/- 0.05	1.05 _/- 0.06	0.853 +/- 0.042
Phosphorus	0.062 +/- 0.005	0.086 +/- 0.007	0.106 +/- 0.0015
Potassium	2.03 +/- 0.06	2.45 +/- 0.08	2.11 +/- 0.11
Silicon	29.66 +/- 0.23	30.44 +/- 0.19	28.97 +/- 0.18
Sodium	1.16 +/- 0.03	1.14 +/- 0.03	1.14 +/- 0.06
Sulfur	0.089 +/- 0.002	0. 042 +/- 0.001	0.24 +/- 0-.006
Titanium (Ti)	0.342 +/- 0.024	0.306 +/- 0.023	0.283 +/- 0.10

(1) NIST Certificates of Analysis are available on the NIST web site https://srmors.nist.gov. The terms high, medium, and low refer to the overall level of contamination within the standard, rather than the concentration of individual constituents.

NA - Not Applicable

Attachment C

Analytical Program Recovery Procedures

Analytical Program Recovery Procedures

This attachment describes several common maintenance and troubleshooting procedures for the iPAQ and XRF units.

C-1. Dealing with an iPAQ that has lost all charge.

Sometimes it is necessary to have an Innov-X Systems instrument in storage or shipped over long periods of time. As far as possible, the iPAQ should be left on charge overnight regularly to maintain the memory and all shortcuts within the software. While readings and the actual program files will not be lost by a full battery drain, some of the program links that are kept in local memory can be, as the local memory is powered by the battery. Below are instructions for bringing an iPAQ for an Innov-X Systems instrument back up from "deep storage".

Charging the iPAQ and setting time and date:

- Put the iPAQ on charge for at least 5 hours. The amber light at the top should blink while the unit is charging. If it does not boot up or blink after the first hour, use the stylus to poke the "soft reset" pinhole at the base of the iPAQ. That should boot up the iPAQ and start the light blinking for charge. Once that light is solid, the unit is fully charged.
- Make sure to set the time and date correctly. Date and time are important for reading storage on the iPAQ, and can cause issues with software and how readings are displayed if not set correctly. Year is especially important. To set the time and date, click on the date from the starting screen of the iPAQ. There will be a local time and a travel time. Setting the local time is all that is essential for instrument performance. Be sure to click "OK" to save the changes once you have put in the current date and time. (Additional information about resetting the date is provided in section C-3 below.)
- Once the unit is fully charged, do a soft reset as instructed above. Then go to the Start menu, and click Innov-X. If the unit gives a "cannot find shortcut" error, use the instructions below to re-establish the shortcut.

C-2. Lost Shortcut on iPAQ

On occasion, low battery life, or the loosening of the memory card can cause a "cannot find shortcut" error message in the Innov-X Systems software on the iPAQ. If that message is displayed , following these steps will restore the shortcut. (In addition, make sure that the time and date on the iPAQ are correct, as those items being incorrect can cause issues with viewing results.)

1. Remove the iPAQ from the analyzer.
2. Remove and reinsert the compact flash (CF) card in the back of the iPAQ to ensure that it is seated properly.
3. Go to Start->Programs->File Explorer.
4. To the right-hand side will be a menu that lists where you are currently looking in the system. Drop this down and choose "My Device."

5. Choose the CF or Storage Card.
6. Click on the Innov-x Folder.
7. Look for the file called "Main" and tap on it.
8. This will bring up the main Innov-X Systems menu. Tap "File," then "Exit."
9. Go to the Start menu and the Innov-X Systems icon should be there.

Note: If the unit goes into the main menu, but then does not go into any of the software options, and displays a "default IME" error at the top of the screen, do a "soft reset" as instructed above to reset the memory and restore the shortcut.

C-3. Resetting the date.

If the battery has run low on your iPAQ, the date may have reset to the original factory install date of the iPAQ. As the software stores the results data chronologically, it is difficult to find results when the date on the iPAQ is incorrect. To reset the date:

1. Make sure you are on the main iPAQ screen.
2. Tap on the incorrect date at the top. This will open up the "Edit Date" screen.
3. Tap on the keyboard symbol in the lower right corner. Then you will be able to tap on the numbers and edit them using the keyboard.
4. Once the date is correct, tap on the "OK" button in the upper right-hand corner and then approve the change.
5. You will then return to the main iPAQ screen and can confirm that the change has taken effect.

Attachment D

Determining Data Usability of In-situ Testing

Determining Data Usability of In-situ Testing

For operators relying extensively on in-situ testing, it is important to determine the data usability of this testing at a given site. This protocol described below is not intended for every sample, but rather for a small percentage of samples considered representative of the site. If the operator can demonstrate that quantitative data are being achieved with little or no sample preparation, then the site characterization can be completed much more quickly.

For example, an operator may be able to demonstrate that the XRF result changes considerably when samples are passed through a 2 mm sieve, but that XRF results do NOT change appreciably upon finer sieving. In this case, the operator can conclude that good XRF data are achievable with only 2 mm sieving. Sieving only to this level requires far less time than a more robust sample preparation. A protocol to determine the appropriate level of sample preparation is the following:

1. Select a region of soil approximately four inches square.
2. Perform several in-situ tests in this area, or collect the top (approximately) quarter inch of soil from this region, bag the soil, and test it through the bag. In either case, average the results and record as Test 1.
3. If you did not bag the in-situ test sample, collect the top (approximately) quarter inch of soil from this region and sieve through the 2 mm (10-mesh) sieve. Otherwise, sieve the bagged sample used for the in-situ test. Thoroughly mix the sieved sample, and place some of the sieved material into an XRF cup or a new baggie. Perform a test of this sample and record the results as Test 2.
4. If the results of Test 2 differ less than 20% from the average in-situ results (Test 1), this indicates the soil in this region is reasonably homogeneous and the data are semi-quantitative and usable.
5. If the results of Test 1 and Test 2 differ by more than 20%, the soil is not homogeneous, and particle size effects are likely affecting your in-situ measurements. In this case, grind and sieve the sample through the 250 µm (60 mesh) sieve. Mix this sample and place a sub-sample into an XRF cup for testing. Record this test as Test 3.
6. If the results of Test 2 and Test 3 differ by less than 20%, passing soil through the 10-mesh sieve provides generally usable data.
7. If the results of Test 2 and Test 3 differ by more than 20%, particle size effects are still influencing the XRF results. In this case, samples for quantitative analysis should be sent to the laboratory to assure data quality.

Innovative Technical Solutions, Inc.

Standard Operating Procedure

Sample Tracking and Electronic Data Management

PR-TC-02.12.02.00

Effective Date: 30 September 2009

Prepared by: Kristen Carlyon, Program Chemist — Date: 30 September 2009

Reviewed by: Wing Tse, Database Manager — Date: 30 September 2009

Approved by: Jeffrey Hess, Program Director — Date: 30 September 2009

Revision History:

Version	Changes	Affects Section/Pages	Effective date
1.0	Initial Issue		30 Sep 2009
1.1	Clarification of Project Chemist responsibilities, and corrections to section numbering.	Pages 2-4	30 Sep 2009

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1.0 PURPOSE AND SCOPE

The purpose of this standard operating procedure is to describe the requirements and procedures for tracking environmental samples in a manner that will provide a systematic means of notifying our electronic data management group (including our chemists, database administrators, and other interested parties) of upcoming sampling events, ensuring the correct samples are collected and correct analyses are requested, tracking the receipt of analytical data from the laboratory for the sampling efforts, facilitate upload of electronic data to the database from the field crew and laboratories, and provide a reference for reconciliation of laboratory invoices.

2.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

COC: Chain-of-Custody form

CDQMP: Chemical Data Quality Management Plan

DQO: Data Quality Objective

EDD: Electronic Data Deliverable

eDMS: Environmental Data Management System, ITSI's in-house environmental data management system.

ERPIMS: Environmental Restoration Program Information Management System

FSP: Field Sampling Plan

Geotracker: A database and geographic information system (GIS) hosted by the California State Water Resources Control Board (SWRCB) that provides online access to environmental data.

GPS: Global Positioning System

LIMS: Laboratory Information Management System

NIRIS: Naval Installation Restoration Information Solution

ORP: Oxidation-reduction potential

PID: Photo-ionization detector

QAPP: Quality Assurance Project Plan

SAP: Sampling and Analysis Plan

SEDD: Staged Electronic Data Deliverable

XRF: X-ray fluorescence

3.0 PROCEDURES AND RESPONSIBILITIES

Systematic sample tracking and efficient data management require that the procedures presented in this SOP be followed by all parties involved in the flow of environmental data. Figure 1 outlines the flow of sample information and laboratory results from initial sampling through reporting of the validated results. Responsibilities, by role, for managing the accurate collection and reporting of the data are discussed below and specific procedures are presented in Sections 3.1-3.3.

- **Project Manager**. Establishes and communicates the goals and objectives (DQOs) of the sampling event to the team, and providing specifics regarding the number and type of samples, analytical methods, and any special reporting requirements. Authorizes payment of laboratory and validation invoices upon successful submittal of complete EDD.
- **Field Personnel**. Responsible for the proper collection of environmental samples according to the approved SAP or FSP/QAPP. Responsible for accurate, defensible documentation of sample collection per CDQMP and all project planning documents.
- **Sample Coordinator**. Responsible for tracking the samples from time of collection through laboratory acceptance. Updates the Sample Tracking Log daily. Reconciles coolers contents against COCs prior to transfer to laboratory. Inputs COC and field information, including sample coordinates and field parameters, into eDMS daily. Submit samples to the laboratory. Sends COCs, Sample Collection Logs and other field forms to project team. Resolves completeness issues with laboratory (e.g. broken bottles, missing samples, etc.).
- **Project Chemist** (or designee). Prepares SAP or FSP/QAPP and sample tracking log. Sets up site in eDMS and portal, if necessary. Loads eQAPP and sample schedule into eDMS. Uploads project planning documents to ITSI project portal. Reviews COC and field information in eDMS. Reviews LIMS login report and resolves analytical issues with laboratory. Assists lab in data upload of results. Reviews and approves results/validation qualifiers, releasing data for use. Updates Sample Tracking Log once samples are in the laboratory and for subsequent activities (e.g., data validation). Notify Project Manager upon successful submittal of completed EDDs.
- **Data Management Staff**. Work with Project Chemist on setting up project and/or site, if new. Identify EDD reporting requirements (ERPIMS, NIRIS, SEDD, etc.) based on Contract and Task Order requirements. Manage any new user accounts needed based on staffing of project team. Work with Project Chemist on designation of sample IDs (and new location IDs, if required). Runs completeness test on EDDs against COCs to insure all data has been received, and finalize data in eDMS.

3.1 FIELD STAFF RESPONSIBILITIES

3.1.1 Notification of Sampling

At the beginning of each project involving the acquisition of environmental data, a preliminary meeting will be held by the project manager, project chemist and members of the electronic data

management group to discuss the data quality objectives (DQOs), sampling requirements, and plan out preparation of the SAP or FSP/QAPP (including location and sample IDs). Once fieldwork is scheduled, a meeting between the project manager, project chemist and the field personnel prior to deployment will be conducted to discuss the specific requirements of the project. Specific information regarding the number and type of samples to be collected will be presented, along with recommended field procedures, sequence of work, and identity of the primary and secondary analytical laboratories. Sample naming protocols will be discussed to insure proper sample identification in the field and on Chain-of-Custodies (COCs). The electronic data management group will be notified of the start of sampling at this time.

For multi-phase or recurring projects such as quarterly monitoring, both the project chemist and electronic data management group will be notified prior to the beginning of each sampling event, and a copy of the Sample Tracking Log will be provided to all interested parties prior to the initiation of sampling (The SAP or FSP/QAPP should contain a copy, but please provide updated version if any changes have been made prior to field sampling).

3.1.2 Chain-of-Custody

During field sampling activities, a copy of the COC will be forwarded daily by the sample coordinator to both the ITSI Project Office (to the Project Chemist) and ITSI Corporate Office (to the electronic data management group).

- If paper COCs are used, then ITSI COCs with a unique identification number should be used and can be requested through the COC coordinator (Dan Wedeking at 925-946-3349) in ITSI's Corporate Office prior to the beginning of sampling. The location and sample IDs and the sample depths (top and bottom) for non-aqueous samples should be written on the copies of the COCs. The COCs may be uploaded to the server or emailed directly to the ITSI Offices. If upload to the server is chosen, the ITSI offices will be notified by email at the time of the first upload. In those cases where there is no internet access available, COCs are to be faxed daily and sent by FedEx at the end of each week.
- If Forms II Lite is used, a copy of the output files should be forwarded electronically to the Project Chemist and electronic data management group, along with a copy of the hard copy COC from the field printer for reference.

Once the data management system is fully deployed, the information from the COCs will be entered into the system daily from the field by the sample coordinator, where connectivity is available. If no connectivity is available, the project team will arrange for the information to be entered by others from the COCs provided to the project and Corporate offices.

3.1.3 Sample Coordinates and Other Field Notes

Along with the COCs, the sample coordinator will forward (GPS) sample coordinates to be loaded into the Sample Tracking Log (or online data management system) for each sample collected, with the exception of waste and some process samples, or recurring sample locations where coordinates already exist (e.g., previously surveyed monitoring wells). Please confirm the existence of valid coordinates for each sample location prior to sampling, otherwise collect GPS coordinates just in case. In addition, all field notes including boring logs, water levels, and field measurements will be entered in the online data management system (once fully deployed) no

later than weekly (ideally on a daily basis), and/or sent on a daily basis to the data management group for entry into the system.

3.2 PROJECT CHEMIST RESPONSIBILITIES

3.2.1 Development of SAP or FSP/QAPP and Appropriate Location and Sample IDs

The Project Chemist is responsible for preparation of the SAP or QAPP/FSP and development of appropriate location and sample IDs. A list of location IDs and their associated sample IDs should be sent to the data manager or their designee for approval before they are incorporated in the sampling plan. The location and sample IDs should conform to the location and sample ID nomenclature requirements listed in SOP PR-TC-01.04.04.00. Once the project plans are approved by the client, the Project Chemist shall set up the site in the eDMS and the portal and upload the eQAPP to eDMS and the work plan to the portal.

3.2.2 Creation of Sample Tracking Log

The project chemist shall develop a Sample Tracking Log at the inception of the project. The log shall track the following items:

Pre-Sampling Post-Sampling

- Location ID
- Sample ID
- Sample matrix
- Required analyses
- Sample Date
- Date Submitted to Lab
- Laboratory Sample ID
- Status of data packages.

3.2.2.1 QC of Entry of COCs in eDMS

The project chemist will QC the data entry of the COC information entered into eDMS. The sample identifications, analyses requested, sampling methods, dates and times of sample collections, and proper assignment of quality control samples will all be checked. The Project Chemist will also verify that the Sample Tracking Log has been updated, and will update the log if it has not been updated.

3.2.2.2 Cross-checking of Laboratory Receipt Form

Upon receipt of the samples by the laboratory, a completed chain-of-custody and laboratory receipt form shall be forwarded to the Project Chemist and crosschecked to the Sample Tracking Log (or online data management system) within 48 hours. Transcription errors and any minor differences will be resolved right away and documented through email correspondence. Major problems will be documented through the use of corrective action forms.

3.2.3 Receipt of Data and Data Uploads

As laboratory data packages are prepared and submitted to ITSI, receipt of these data packages will be recorded on the Sample Tracking Log (or online data management system). The Project Chemist will assist the lab in data upload of results. If not directly submitted, the electronic data deliverables (EDDs) in acceptable format (Enhanced ERPIMS unless otherwise approved) will be forwarded to the electronic data management group right away. The completeness of the EDDs will be verified upon receipt by the electronic data management group. eDMS will screen the results against the eQAPP. The Project Chemist will then review and approve the results in eDMS, checking the tracking log for completeness.

Validation using eDMS or a third-party validator occurs at this point. The validation codes are applied to eDMS and a validation report is prepared. The Project Chemist reviews and approves the qualifiers and again updates the Sample Tracking Log. At this point the data is approved for general use.

Upon completion of the receipt of the last sample for the sampling event (for example, one complete round of groundwater monitoring), a copy of the completed Sample Tracking Log will be forwarded to the electronic data management group for organization purposes

3.2.4 Reconciliation of Invoices

Upon receipt of laboratory invoices, the Project Chemist or his designee will cross-check the invoices against the sample tracking log to verify the receipt by ITSI of all billed sample analyses, completed final data packages, and EDDs (accepted by the electronic data management group) before notifying the project manager that the invoices should be authorized for payment.

3.3 DATA MANAGEMENT GROUP RESPONSIBILITIES

3.3.1 Support Input of Field Data

The data management group will support the Project Chemist and sample coordinator in promptly entering field data into the online data management system until the online data management system is fully deployed, following that, in cases where access by the field staff is limited.

3.3.2 Upload of Sample Information to Database

The data management group will use the information in the sample tracking log and the COCs as they are received in preparation for the upload of the electronic data deliverables (EDDs) directly from the laboratory or from laboratory provided electronic files.

Upon receipt of the sample-tracking log, the electronic data management group will review the log. Any immediate potential problems (for example, the use of the dash '-' instead of an underscore '_' in the laboratory data system) that may follow in the preparation of the EDD will be identified and corrected.

3.3.2.1 Entry of COC s

The data management group will enter the COC information into eDMS. The sample identifications, analyses requested, sampling methods, dates and times of sample collections, and proper assignment of quality control samples will all be cross-checked for accuracy. The Sample Tracking Log will be updated as each COC is entered.

3.3.2.2 Entry of Other Field Data

Other data to be entered by the electronic data management group will include water levels, field stability parameters (dissolved oxygen, ORP, turbidity, etc.), and GPS or survey coordinates. Additional data may include results of XRF field sampling, immunoassay test kit sampling, PID measurements, or other information deemed important by the Project Manager for data review and analysis, and ultimately for reporting to the client.

3.3.3 Creation of Final EDDs

The data management group will consolidate the validated EDDs from the in-house and/or third-party data validation firm, and input the field information needed to complete the required EDD package. The final EDD will then be submitted to the government in the required format (ERPIMS, NIRIS, Geotracker, etc.)

4.0 REQUIRED DOCUMENTATION

The following records generated as a result of implementation of this procedure must be maintained as quality records.

- GPS coordinates for each sample collected
- Field notes
- Chains of Custodies
- Sample Collection Logs
- Sample Tracking Form.

5.0 ATTACHMENTS

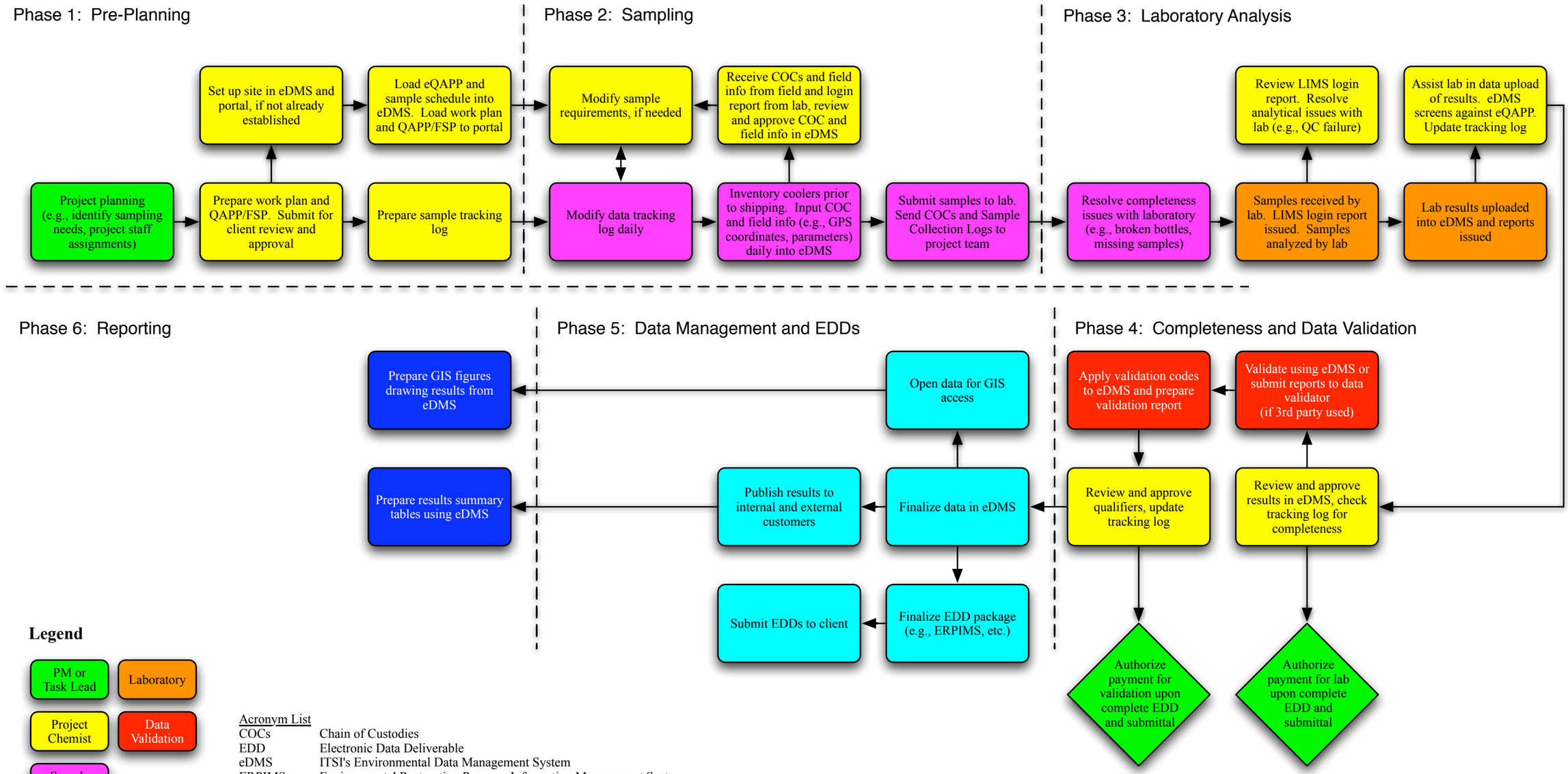
None.

6.0 FORMS

- Sample Tracking Log

7.0 REFERENCES

ITSI, 2004. *Data Management Program: Planning, Collection, Analysis, Verification/Validation, and Reporting of Chemical Data from Environmental Sampling Events, Version 1.1*. January



Legend

- PM or Task Lead
- Laboratory
- Project Chemist
- Data Validation
- Sample Coordinator
- Data Manager
- Data Customer

Acronym List

COCs	Chain of Custodies
EDD	Electronic Data Deliverable
eDMS	ITSI's Environmental Data Management System
ERPIMS	Environmental Restoration Program Information Management System
eQAPP	Electronic Quality Assurance Project Plan
FSP	Field Sampling Plan
GIS	Geographic Information Systems
GPS	Global Positioning System
LIMS	Laboratory Information Management System
PM	Project Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control

Figure 1
ITSI Data Management Workflow

Sample Tracking Log

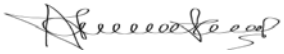
Event	Location ID	Sample Type	Field QC	Date Sampled	Time	Sample ID	Date Shipped	Date Received	Chain of Custody	Laboratory Work Order No.	Laboratory Sample No.	Analyses					Report Received	EDD Received	Data on eDMS	Notes
												TPH-DRO & MO by EPA 8015B	VOCs by EPA 8260	SVOCs by EPA 8270C	Title 22 Metals by 6010/6020/7000	Percent Moisture by ASTM D2216				

SAP ATTACHMENT 4

LAB SOPs

Metals by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
SW846 Method EPA 6010B

Approved by:  Laurie Glantz – Murphy – Laboratory Director Date: 10/8/2010

Approved by:  Guergana Gueorguieva – QA Officer Date: 10/8/2010

Annual Review

Reviewed by: Hang Dinh Date: 3/12/2009

Reviewed by: Hang Dinh Date: 12/23/2009

Reviewed by: Dolores Queja Date: 10/8/2010

Reviewed by: _____ Date: _____

Document Control

Issued To	Issue Date	Comment
Metals ICP	10/8/10	

Title- Metals by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

Method Reference: SW846 Method 6010B Revision 2 December 1996

Revision: Section(s) added section 6.3; 7.2; 9.16; Calibration Standard table 10.1.3; 10.3; 11.12 analytical sequence run; section 12.3.4.1 daily ICP care

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the determination of metals in water, wipes, sludges, sediments, and soils. Sample matrices are pretreated following SW846 methods for digestions of soil, sediment, sludge, wipe (Method 3050B) or waste water samples and extracts (Method 3010A). Refer to specific digestion SOP's for more information on digestion techniques.

1.2 List of metals can be analyzed by ICP :Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sr, Tl, Sn, Ti, V, and Zn

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods.

2.2 This method describes multi-elemental determinations by ICP-AES using simultaneous optical systems and axial and radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices and are processed and controlled by a computer system

3.0 DEFINITIONS

3.1 BATCH. A group of samples that behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

3.2 Calibration standards – A solution prepared from the primary dilution standard solutions or stock standard solutions and are used to calibrate the instrument.

3.2.1 Initial Calibration Standards – A series of calibration standard solutions used to initially establish the linear operating range of the instrument by developing calibration curves for individual target elements.

3.2.2 Instrument Performance Check (IPC) solution – The IPC (Highest calibration standard) solution is run as if it were a sample after the last calibration standard

and is used to verify instrument performance during analysis. The IPC solution is the same source as the calibration standard.

3.2.3 Initial Calibration Verification (ICV) Standard – A standard solution prepared from a standard solution different than that of the calibration standard and analyzed immediately following daily calibration, prior to any sample analysis. This standard verifies the accuracy of the calibration curve.

3.2.4 Continuing Calibration Verification (CCV) Standard – A standard solution which is analyzed after every tenth field sample analyses, and at the end of an analytical run. The CCV verifies the previously established calibration curves and confirms accurate analyte quantification for the previous ten field samples analyzed. At the laboratory's discretion, an ICV may be used in lieu of the CCV. If used in this manner, the ICV should be at a concentration near the mid-point of the calibration curve.

3.2.5 Calibration Blank – A volume of deionized, distilled water acidified to the same concentrations of acid added in the standard and is also used as an initial (ICB) and continuing calibration blank (CCB).

3.2.6 Interference Check Standard (ICSA/ICSAB) – A solution containing known concentrations of interfering elements that will provide an adequate test of the correction factors and is analyzed at the beginning of each analytical run.

3.2.7 Spectral Interference Check (SIC) Solution – A solution of selected method analytes of higher concentrations which is used to evaluate the procedural routine for correcting known interelement spectral interferences with respect to a defined set of method criteria

3.3 Method Detection Limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is generated from processing 7 replicates of reagent water or a clean solid through all of the same steps as a field sample.

3.4 Linear Dynamic Range (LDR) – The concentration range over which the analytical curve remains linear.

3.5 Batch QC – Batch QC refers to the QC samples that are analyzed in an analytical batch of 20 or less field samples, such as, the MB, LCS, MS and MSD or Replicate.

3.5.1 Method Blank (MB) – An aliquot of reagent water or other blank matrices that contain all the reagents in the same volume as used in the processing of the samples.

3.5.2 Laboratory Control Sample (LCS) – An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory and analyzed like a sample. Its purpose is to determine whether the laboratory procedures are in control. This QC measure can be used to assess the laboratory's accuracy and precision when applying this procedure in duplicate.

3.5.3 Matrix Spike (MS) – An aliquot of an environmental or field sample to which known quantities of the method analytes are added in the laboratory and

analyzed like a sample. Its purpose is to determine whether the sample matrix contributes bias to the analytical results.

3.5.4 Replicate (DUP) – Two sample aliquots analyzed separately with identical procedures

4.0 INTERFERENCES

4.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements.

4.1.1 Spectral interference – (1) Background emission or stray light; (2) Spectral overlap of emissions.

4.1.2 Physical interferences are caused by high viscosity or high particulates present in samples which can clog the nebulizer.

4.1.3 Chemical Interferences are characterized by compound formation, ionization effects, and solute vaporization.

4.1.4 Memory Interferences are carry-over from samples

5.0 SAFETY

5.1 Corrosives - Because all standards and samples are prepared in a 5% hydrochloric acid and 2% nitric acid matrix, appropriate care must be taken when handling these solutions. Safety glasses and gloves must be worn when preparing and handling these solutions. All digestion procedures must be performed in a fume hood and any acid spills must be cleaned up and disposed of in an appropriate manner. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 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 data handling sheets such as MSDS and SOP must be made available to all personnel involved in these analyses and must be read before doing the analysis.

5.3 Prevent Shock from High Voltage - The power unit supplies high voltage to the RF generator, which is used to form the plasma. **Do not attempt to repair and/or adjust any components inside the power unit. There are no user-serviceable parts inside and only trained service technicians should access the RF generator.**

5.4 UV Light - When lit, the plasma produces a very intense light which must not be viewed with the naked eye. Protective lenses are in place on the instrument which protects the operator from UV radiation. The instrument has safety interlocks on the plasma shield which prevents the plasma from operating when the door is opened on the Perkin-Elmer OPTIMA 5300.

6.0 EQUIPMENT AND SUPPLIES

6.1 Perkin-Elmer ICP OPTIMA 5300 DV axial, radial dual view with solid state RF generator

6.1.1 Peristaltic pump integrated with the instrument

6.1.2 AS-93 autosampler

6.1.3 Water chiller

6.1.4 "Win Lab 32" software for data handling

6.2 Class A Volumetric flask

6.3 Auto pipettes with tips. These must be calibrated as outlined in the autopipette calibration SOP

7.0 REAGENTS AND STANDARDS

7.1 Reagents such as acids and hydrogen peroxide used in the preparation of standards and for sample processing must be analyzed prior to use to confirm that there are no analyte concentration above the reporting limit.

7.1.1 Hydrochloric acid, concentrated (HCl) (sp.gr. 1.19) – EMD, HX0607/2

7.1.2 Nitric Acid, concentrated (HNO_3) (sp.gr. 1.41) – JT Bake, 9598-34

7.1.3 HCl (5%) and HNO_3 (2%) – Add 50 mL of concentrated HCl and 20 mL of concentrated HNO_3 to 400 mL of reagent water (DI water) and dilute to 1000 mL.

7.2 Calibration Stock Standard solutions can be purchased from an appropriate supplier (SPEX, CPI, SCP) and must be traceable to NIST. The manufacturer's expiration date is used to determine the storage time for each standard. The manufacturer, lot number, and the expiration date for each standard is recorded in the standard logbook and is assigned a unique number when first received. The certificate of analysis must be kept on file for each standard.

Calibration Stock Standard	Vendor	Part Number
MIXSTD1@various conc.	SPEX	MIXSTD1-100
MIXSTD2@various conc.	SPEX	MIXSTD2 -100
MIXSTD3@various conc.	SPEX	MIXSTD3 -100
MIXSTD4@various conc.	SPEX	MIXSTD4 -100
MIXSTD5@various conc.	SPEX	MIXSTD5 - 100
Titanium, 1000 mg/L	SPEX	PLT19-2Y
Strontium, 1000 mg/L	SPEX	PLSR2-2Y
Tin, 1000 mg/L	SPEX	PLSN2-2X

7.3 Quality Control Standard – A solution obtained from outside source having known concentration values is used to verify the calibration standards. This can also be use as a spiking solution for the LCS and MS/MSD.

Quality Control Standard	Vendor	Part Number
QC-7, 100 mg/L	CPI	4400-002
QC-21, 100 mg/L	CPI	4400-010
Tin, 1000 mg/L	CPI	4400-1000612-100
Interferent A (ICSA)	CPI	4400-INTA1-00
Alternate Analyte B1 & B2 (ICSAB)	CPI	4400-INTB1-100 4400-INTB2-100

7.3.1 All stock standards once opened will have expiration of 1 year from date opened.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 The pH of all aqueous samples must be tested immediately before taking an aliquot for digestion or for direct analysis to ensure the sample has been properly preserved.

8.2 For the determination of dissolved metals, the sample must be filtered through a 0.45 µm membrane filter at the time of collection or as soon as possible upon receipt in the laboratory. Acidify the filtrate with (1+1) nitric acid immediately following the filtration to pH <2. The sample must be held at pH <2 for 24 hours before digestion can begin.

8.3 If acid preservation in the field is not feasible, preservation may take place in the laboratory if the sample is received unpreserved. The sample must be held at pH <2 for 24 hours before digestion can begin.

8.4 Only plastic or PTFE containers should be used for the boron and silica determination.

8.5 The holding time for metals sample is 6 months if properly acid preserved. Solid samples are stored in a refrigerator at ≤6ºC. After analysis, sample digestates are stored for about 1 month and are properly disposed of into the drum designated for acidic waste.

9.0 QUALITY CONTROL

9.1 This section outlines the minimum QA/QC requirements necessary to meet the analytical requirements for method SW846 Method 6010B.

9.2 It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

9.2.1 An analyst initial demonstration of capability must be performed. This is the initial analysis of four aliquots of a mid range standard. The average percent recovery and the standard deviation of the four percent recoveries must be calculated. The average recovery must be ±20% of the true value.

9.2.2 Method Detection Limits (MDLs). MDLs should be established for all analytes, using a solution spiked at approximately 3 times the estimated detection limit. Use the MDL to verify LOD at 1-4 times and LOQ at low calibration standard. The LOQ will always be greater than LOD.

9.2 Instrument Dection Limits (IDLs). IDL's should be done **a minimum of once per year** for all analytes at three non-consecutive days or whenever instrument conditions have significantly changed. The IDLs (in ug/L) are determined by multiplying three times the standard deviation of 10 reading of a calibration blank. Each measurement shall be performed as though it were a separate analytical sample (i.e., each measurement shall be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs shall be determined and reported for each wavelength used in the analysis of the samples. IDLs shall be ≤LOD.

9.3 Linear Calibration ranges. The upper limit of the linear calibration ranges should be established for each analyte by determining the signal responses from at least three concentration standards, one of which is close to the upper limit of the linear range. The linear calibration range which may be used for the analysis of samples should be 90

percent of the highest standard that is within 10 percent of the true value. Linear calibration ranges should be determined every 6 months or whenever there is a significant change in instrument response.

9.4 Initial Calibration Verification Standard (ICV). After each calibration, a standard from a different source than the calibration standard should be analyzed with an initial calibration blank (ICB). For the ICV, all elements to be reported must be within 10 percent of the true value and the replicates exceed 5 times the reporting limit should have a relative standard deviation of < 5%. The ICB results should be less than three times the IDL or less than 1/10 of the concentration of the action level of interest. (All ICB values should be less than the reporting limits for the elements).

9.5 Continuing Calibration Verification. Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample or every two hours during an analysis run, whichever is more frequent, and at the end of the sample run. If the continuing CCV solutions are not within 10 percent of the true value, the CCV should be reanalyzed to confirm the initial value. If the CCV is still outside the acceptance limits after the reanalysis, no samples for the failing element(s) can be reported in the area bracketed by the failing CCV. An exception is if the recovery of the CCV is in the range of 111% to 125% and the sample results are less than the reporting limit. (Note: If a CCV fails due to a calibration shift, rather than a one time problem, then the instrument should be recalibrated and the QC rechecked before any additional samples are analyzed.)

9.5.1 If the CCV recovery is within specification and the relative standard deviation is high, then the CCV can be reanalyzed as long as the 10% frequency is met. If the reanalysis is within specifications, then the bracketed samples can be reported.

9.5.2 If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Results for specific analyte in all samples since the last acceptable CCV should be flag.

9.6 Continuing Calibration Blank. Analyze the continuing calibration verification solution and the continuing calibration blank immediately following the daily calibration, after every tenth sample or every 2 hours during an analysis, whichever is more frequent, and at the end of the sample run. The CCB results should be **less than three times the IDL** or less than 1/10 of the concentration of the action level of interest. (All CCB values should be less than the reporting limits for the elements). If the CCB does not meet this criterion, it can be reanalyzed two more times. The average of the three readings must be within 3 standard deviations of the background mean. If these criteria are not met, then no samples for the failing element(s) can be reported in the area bracketed by the failing CCB.

9.7 Low Level Calibration Check (CRI). The CRI standard contains the elements of interest at levels near the low end of the curve or less than or equal to reporting limit. This method does not require the analysis of a CRI solution at the beginning of the run. However, some specifics client such as DoD require that the calibration curve be verified with a low calibration check after every calibration. Run the CRI solution at the beginning of the analysis unless otherwise instructed by the metals supervisor or manager.

9.7.1 In no limits are specified, then in house acceptance criteria **of 50 to 150 percent** will be applied. If an analyte is outside the specified range, check with metals supervisor.

9.7.2 For work following the DOD requirement, the low level check must be at the level of the reporting limit for each element. In addition, acceptance criteria of ±20% of the true value should be applied. If an analyte is outside the specified range, correct the problem, then reanalyze. No samples may be analyzed without a valid low-level calibration check standard. Check with metals supervisor.

9.8 Serial Dilution Analysis. For one sample per preparation or analysis batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution should be prepared. For the serial dilution, a 1:5 dilution should be made on the sample. The results of the 1:5 dilutions should agree within 10 percent of the true value as long as the sample result is greater than 50 times the LOQ. If the dilution does not agree, then remake and reanalyze the serial dilution. If the reanalysis is again not within the limit, the failing elements should be footnoted indicating that there were possible matrix interferences. Alternatively, a serial dilution can be done with larger dilutions, and the sample can be reported from the dilutions. For example, a sample that failed the serial dilution criteria using the straight sample and a 1:5 dilution may pass the serial dilution criteria using a 1:2 dilution and a 1:10 dilution. In that case, the sample would be reported from the 1:2 dilution and the results would be footnoted that a dilution was required due to matrix interference. The calculation to be used for serial dilutions is shown below.

$$\frac{\text{(Sample Result - Serial Dilution Result)}}{\text{Sample Result}} \times 100 = \text{Serial Dilution RPD}$$

9.9 Post Digestion Spike Addition. Post-digest spikes may also be used to determine potential interferences. Check with the metals supervisor for further information on when a post-digest spike should be performed. Recovery limits of 75 to 125 percent should be used to assess post-digest spikes. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

9.10 Interference Check Standard. An interference check standard must be analyzed at the beginning of each analytical run. For all spiked elements, the analyzed results must be within 20 percent of the true values. For unspiked elements, the interfering element solutions should contain less than the absolute value of two times the reporting limit for each element. If these criteria are not met, then no samples containing the elements in question can be reported in the area bracketed by this QC unless the samples contain no significant interferent. This method does not require the analysis of an interfering element check solution at the end of the run. However, this may be required ~~due~~ to meet other method and/or client requirements. Run the ICSA and ICSAB solutions every 8 hours unless otherwise instructed by the metals lab supervisor or manager.

9.10.1 Some project specifics or client such as DoD may require that absolute value of concentration for all non-spiked analytes < LOD in the ICS-A and ±20 percent of the true values in ICS-AB. If ICS fails, terminate analysis, locate and correct

problem, reanalyze ICS and rerun samples. If an analyte is outside the specified range, check with metals supervisor.

9.11 Method Blank. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. The method blank must contain elements at less than the reporting limit for each element. If the method blank contains over that limit, the samples must be re digested or reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

9.12 Lab Control Sample/Spike Blank. The laboratory must digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. For a running batch, a new lab control sample or spike blank is required for each different digestion day. The laboratory should assess laboratory performance of an aqueous lab control or spike blank against recovery limits of **80 to 120 percent**. For solid lab controls, the elements should be within the range given by the lab control supplier. If the lab control or spike blank is outside of the control limits for a given element, all samples must be redigested and reanalyzed for that element.

9.13 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. The control limits for the matrix spike recovery are from **75 to 125 percent recovery**. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

9.13.1 For work following DoD requirement, the control limit for matrix spike recovery should use QC acceptance criteria specified by DoD for LCS.

$$\left(\frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Amount Spiked}}\right) \times 100 = \text{matrix spike recovery}$$

9.14 Some clients may require a post-digest spike if the matrix spike is outside of the control limits. Spiking levels should be set as per client requirements. Either CLP type spike levels or SW846 spike levels may be used, depending on client needs. Check with the metals supervisor for more information.

9.14.1 Method 6010B specifies that the post-digest spike be in the range of 10 to 100 times the detection limit. Limits of 75 to 125 percent are normally applied, unless a client specifies a different limit. No action is necessary if the post-digest spike is outside of this limit, unless a preparation problem is suspected with the spike, in which case the post-digest spike should be remade and reanalyzed. The post-

digest spike recovery should be footnoted on the matrix spike recovery for the element of interest.

9.14.2 Matrix Spike Duplicate. The laboratory must digest a matrix spike duplicate sample for a minimum of 1 in 20 samples. A duplicate may be used in place of a matrix spike duplicate on client request. The relative percent difference (RPD) between the matrix spike and the matrix spike duplicate should be assessed. The matrix spike duplicate or duplicate RPD is calculated as shown below. The control limit for the duplicate is <20% RPD. If a matrix spike duplicate or duplicate is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

$$\frac{(|\text{Matrix Spike Result} - \text{Matrix Spike Duplicate Result}|) \times 100}{\text{Matrix Spike Result} + \text{Matrix Spike Duplicate Result})/2} = \text{Duplicate RPD}$$

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration /Rinse Blank (Standard 1). The calibration blank is prepared by diluting a mixture of 20 ml of concentrated nitric acid and 50 ml of concentrated hydrochloric acid to a final volume of 1 liter with deionized water.

10.2 Calibration Standards: These can be made up by diluting the stock solutions to the appropriate concentrations.

10.2.1 Standards should be approximately matrix matched to the samples. For most samples, a 3 percent nitric acid and 5 percent hydrochloric acid will approximate the acid matrix of the sample and limit nebulization problems. If it is known that the samples contain a significantly different acid matrix, then the matrix of the standards should be modified or the samples should be diluted so that they are in a similar matrix to the curve.

10.2.2 Standards should be prepared so that there is minimal spectral interference between analytes.

10.2.3 To a 100-mL volumetric flask, add approximately 80 ml of DI water, 2.0 ml of conc. HNO_3 and 5.0 ml conc. HCl. Swirl to mix. Carefully pipette 1.0 ml of the following purchased mix stock standards and Single Element Standards at various volumes as outlined below. Dilute to 100 ml with DI water. Refer to the standards book for specific information on the standards and stock solutions. Refer to Table below for the levels of standards to be used in the calibrations.

10.2.3.1 Calibration Standard 2

Mix Std	Elements	Final Conc. (mg/L)
1	Be **50mg/L**	**0.5**
1mL	Cd **150mg/L**	**1.5**
	Pb **500mg/L**	**5.0**
	Mn **100mg/L**	**1.0**
	Se **200mg/L**	**2.0**
	Zn **150mg/L**	**1.5**

Mix Std	Elements	Final Conc. (ug/mL)
3	As **500mg/L**	**5.0**
1mL	Mo **100 mg/L**	**1.0**
	Si **100mg/L**	**1.0**

Mix Std	Elements	Final Conc. (mg/L)
4	Al **200mg/L**	**2.0**
1mL	Ca **1000mg/L**	**10.0**
	Cr **20mg/L**	**0.2**
	Ni **20mg/L**	**0.2**
	K **400mg/L**	**4.0**
	Na **200mg/L**	**2.0**

Mix Std	Elements	Final Conc. (mg/L)
4	Al **200mg/L**	**2.0**
1mL	Ca **1000mg/L**	**10.0**
	Cr **20mg/L**	**0.2**
	Ni **20mg/L**	**0.2**
	K **400mg/L**	**4.0**
	Na **200mg/L**	**2.0**

Mix Std	Elements	Final Conc. (mg/L)
5	Sb **200mg/L**	**2.0**
1mL	B**100mg/L**	**1.0**
	Mg 1000**mg/L**	**10.0**
	Ag **50mg/L**	**0.5**
	Tl 200 mg/L	**2.0**

Single Element Std	Standard conc	Vol(mL)	Final Conc. (mg/L)
Sr	1000mg/L	0.050	0.5
Ti	1000mg/L	0.100	1.0
Sn	1000mg/L	0.200	2.0
Li	1000mg/L	0.200	2.0
Ba	1000mg/L	0.100	1.0
Co	1000mg/L	0.100	1.0
Cu	1000mg/L	0.100	1.0
V_	1000mg/L	0.100	1.0

10.2.3.2 Calibration Standard 3 - Minerals

Single Element Std	Standard conc	Vol(mL)	Final conc. mg/L
Al	**10,000mg/L**	0.500	**50.0**
Ca	**10,000mg/L**	0.500	**50.0**
Fe	**10,000mg/L**	0.500	**50.0**
Mg	**10,000mg/L**	0.500	**50.0**
K	**10,000mg/L**	0.500	**50.0**
Na	**10,000mg/L**	0.500	**50.0**

Refer to Table 20.1 for List of Calibration **STANDARD, ICV, CCV, CRIA and CRID LEVELS**

10.3 Continuing Calibration Verification Check (CCV). This solution is also known as the Instrument Performance Check (IPC) Solution. This solution is prepared by adding either mixed or single element metals solutions to a solution containing 2 percent nitric acid and 5 percent hydrochloric acid and diluting to a fixed final volume with this acid mixture. The metals should be at concentrations near the middle range of the calibration curve. (Note: This check is run after the calibration, after every 10 samples or every 2 hours during an analysis run, whichever is more frequent, and at the end of the sample run.)

10.3.1 To a 100 mL volumetric flask (Nalgene®), add approximately 80 mL of DI H2O, 5 mL conc HCl and 2 mL conc HNO3. Swirl to mix. Carefully pipette 0.5 mL of the following purchased multi element calibration standards and Single Element Standards at various volumes as outlined below. Dilute to mark with DI water and mix.

Single Element Std	Standard conc	Vol(mL)
Sr	**1000mg/L**	0.025
Ti	**1000mg/L**	0.050
Sn	**1000mg/L**	0.100
Li	**1000mg/L**	0.100
Ba	**1000mg/L**	0.050
Co	**1000mg/L**	0.050
Cu	**1000mg/L**	0.050
V_	**1000mg/L**	0.050
Al	**10,000mg/L**	0.240
Ca	**10,000mg/L**	0.200
Mg	**10,000mg/L**	0.200
K	**10,000mg/L**	0.230
Na	**10,000mg/L**	0.240
Fe	**10,000mg/L**	0.250
Mixed Standard		Vol(mL)
1	various concentration	0.5
3		0.5
		0.5
5		0.5

Final Concentration in ICCV/CCV				
Elements	Final Conc. 'mg/L		Elements	Final Conc. 'mg/L
Aluminum	25		Manganese	0.5
Antimony	1		Molybdenum	0.5
Arsenic	2.5		Nickel	0.1
Barium	1		Potassium	25
Beryllium	0.25		Selenium	1
Boron	1		Silicon	0.5
Cadmium	1		Silver	0.25
Calcium	25		Sodium	25
Chromium	0.1		Strontium	0.25
Cobalt	1		Thallium	1
Iron	25		Tin	1
Lead	2.5		Titanium	0.5
Lithium	1		Vanadium	0.5
Magnesium	25		Zinc	0.75

10.4 Matrix Spike and Spike Blank Solution. (For aqueous samples and TCLP/STLC leachates)

10.4.1 A 0.25 ml of two QC stock standard solutions (7.3) should be added to spike blank and the matrix spike before they are digested and brought to a final volume of 50 ml. In situations where any odd elements, such as Bi, Li, Si, Sn, W and Pd, is of interest for a specific project, a spike blank spiked with these elements is also digested.

10.4.2 For TCLP/STLC samples, the lab control should be made using blank leachate solution rather than DI water.

QC-21 (0.25-ml of 100 ppm) QC-7 (0.25-ml of 100 ppm) Tin (50uL of 1000ppm)	Final Concentrations (0.5mg/L) Al B Cu Mg Ag Ti Sb, Ca Fe Mo Na V As Cd Pb K(5.0) Sr Zn Ba Cr Ni Se Tl Be Co Mn Sn Si (0.25)

10.5 Lab Control Solution. This solution is prepared by adding either mixed or single element metal solutions to DI water and diluting to a fixed final volume. 50 ml of this solution is digested and brought to a final volume of 50 ml.

10.6 CRIA Standards. The CRI standards contain the elements of interest at levels near the low end of the curve. However, low checks are at twice the reporting limit or the reporting level.

10.7 ICSA/ICSAB Interfering Element Solutions.

10.7.1 The ICSA solution contains only the interfering elements. The recommended concentrations are shown below. . If the linear ranges on a given instrument are lower than these levels, the concentrations may be set near the top of the linear range for those elements.

Al	500 mg/L
Ca	500 mg/L
Fe	200 mg/L
Mg	500 mg/L

10.7.2 ICSAB Solution. The ICSAB solution contains both the interferents and the analytes of interest. The recommended concentrations are shown below. . If the linear ranges on a given instrument are lower than these levels, the concentrations may be set near the top of the linear range for those elements.

Ag	1.0 mg/L	Cu	0.50 mg/L
Al	500 mg/L	Fe	200 mg/L
Ba	0.50 mg/L	Mg	500 mg/L
Be	0.50 mg/L	Mn	0.50 mg/L
Ca	500 mg/L	Ni	1.0 mg/L
Cd	1.0 mg/L	Pb	1.0 mg/L
Co	0.50 mg/L	V	0.50 mg/L
Cr	0.50 mg/L	Zn	1.0 mg/L
As	1.0 mg/L	Tl	1.0 mg/L
Se	1.0 mg/L	Mo	0.5 mg/L
Sb	1.0 mg/L	Pd	0.5 mg/L

10.8 Internal Standard Solution. To a 1 liter flask containing approximately 800 ml of DI water, add 0.5 ml of 10,000 mg/l yttrium. Add 20 ml concentrated nitric acid and bring to a final volume of 1000 ml ad mix well. This solution is added to all samples and standards as the instrument is running using an internal standard kit line on the peristallic pump.

11.0 PROCEDURE

11.1 Perkin-Elmer ICP 5300 DV

11.1.1 Set up the ICP using the proper operating parameters as established for the ICP-5300 DV.

11.1.1.1 Make sure the argon gas on, cooler on, change new acid rinse into the bottle, and make sure internal standard solution is sufficient.

11.1.1.2 Make sure the torch, radial/axial windows and nebulizer in the optimized condition. The torch and radial/axial windows can be replaced weekly or as needed.

11.1.1.3 Turn on instrument and allow the instrument to become thermally stable for about 30 minutes prior to calibration.

11.1.2 Align the ICP using the 1mg/L Mn solution (this is also known as Profile). Open the File menu, select workspace then Align ICP. Aspirate the solution for about 60 seconds and hit Align view. (for both Axial and Radial). For the Axial instrumentation, this step should be performed daily. When done, place the probe in DI water to rinse the system.

11.1.2.1 On a daily basis, monitor the counts of 1 mg/L Mn of Axial and radial view. If there is significant shift (greater than 10%), investigate the shift before starting the run.

11.1.3 Select the appropriate workspace or method for the sample to be analyzed.

11.1.4 Set up the autosampler using the sample information editor to load sample ID, autosampler location of samples and details such as weight, dilution, and units. In order to minimize loading errors, print the autosampler loading list prior to loading the sample into the tray and include the documentation with the raw data file.

11.1.5 Analyze the sample using the Automated Analysis Control or Manual Control.

11.1.6 Save all data including the spectra under Result Data Set Name.

11.2 From the Automated Analysis Control Panel, click on the Calibrate button to analyze standards only. Examine the spectra if needed.

11.3 After calibration is completed, begin analyzing the CCV and CCB for each element. The analyzed value must be within 5% of the true value or the calibration should be recalibrated.

11.4 After the instrument is properly calibrated, begin by analyzing the ICV and ICB check standards. For the ICV, all elements to be reported must be within 10 percent of the true values. For both ICV and CCV, all replicates exceed 5 times the reporting limit should have a relative standard deviation of less than 5 percent. Both ICB and CCB results should be less than three times the IDL or less than 1/10 of the concentration of the action level of interest. (ICB and CCB values should be less than the reporting limits for the elements).

11.5 Run the CRI solution (Low level Calibration Check) For the CRI, all elements of interest must be within ± 50% of the true value or within client specified limits. Check with the metals supervisor for more information.

11.5.1 For work following the DOD requirement, the low level check must be at the level of the reporting limit for each element. In addition, acceptance criteria of ±20% of the true value should be applied. No samples may be analyzed without a valid low-level calibration check standard. Check with metals supervisor.

11.6 Before analyzing any real samples, the interfering element solutions must be checked. The ICSA/ICSAB solutions must be run on a daily basis. For all the spiked elements, the analyzed results must be within 20 percent of the true results. For unspiked elements, the interfering element solution should contain less than the absolute value of the reporting limit for each element.

11.6.1 Some project specifics or client such as DoD may require that absolute value of concentration for all non-spiked analytes < LOD in the ICS-A and ±20 percent of the true values in ICS-AB. If ICS fails, terminate analysis, locate and correct problem, reanalyze ICS and rerun samples. If an analyte is outside the specified range, check with metals supervisor.

11.6.2 If the interfering element solution is not within specifications and that element must be reported, then new interfering element correction (IEC) factors will need to be generated. If new IEC's are generated, then the run must be restarted from the ICSA, ICSAB quality control samples and new CCV checks must be run before any samples can be reported.

11.7 CCV and CCB should be analyzed before any of samples run. For the CCV, all elements to be reported must be within 10 percent of the true values. For the elements with a CCV greater than 5 times the reporting limit, the relative standard deviation for the replicate should be less than 5%.

11.7.1 If the CCV is not within 10 percent of the true values, no sample can be reported in the area bracketed by the failing CCV for the failing element.

11.8 After the initial analytical quality control has been analyzed, the samples and the preparation batch quality control should be analyzed. Each sample analysis must be a minimum of 3 readings using at least a 5 second integration time. For samples containing levels of elements greater than approximately 5 times the reporting limits, the relative standard deviations for the replicates should be less than 5%. If not, reanalyze the sample. If, upon reanalysis, the RSDs are acceptable, then report the data from the reanalysis. If RSD's are not acceptable on reanalysis, then, on the reviewer's discretion, the results for that element may be footnoted that there are possible analytical problems indicated by a high RSD between replicates. In some cases, an additional dilution analysis may be needed. Check with the area supervisor or manager for additional information.

11.9 Between each sample, flush the nebulizer and solution uptake system with a blank rinse solution for a minimum of 60 seconds or for the required period of time to ensure that

analyte memory effects are not occurring. A time of 120 seconds is recommended for most analyses.

11.9.1 A rinse time of 100 seconds combined with the step-ahead function will give an approximate total rinse time of 120 second.

11.10 Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth samples or every 2 hours during an analysis run, whichever is more frequent, and at the end of the sample run.

11.10.1 If the continuing CCV solution is not within 10 percent of the true value, no samples can be reported in the area bracketed by the failing CCV for the failing element. Additionally, for the elements with a CCV greater than 5 times the reporting limit, the relative standard deviation for the replicates should be less than 5 percent.

11.10.2 The CCB results should be less than three times the IDL or less than 1/10 of the concentration of the action level of interest and less than the reporting limit. If the CCB does not meet this criterion and is less than the reporting limit, it can be reanalyzed two more times. The average of the three readings must be within 3 standard deviations of the background mean. If these criteria are not met, then no samples can be reported in the area bracketed by the failing CCB for the failing element. The exception is that samples that are less than the reporting limit may be reported if the CCB is biased high, but is still less than the reporting limit.

11.10.2 If the initial CCB is above the reporting limit, then all samples should be submitted for reanalysis. Do not rerun the CCB if the initial reading is above the reporting limit.

11.10.3 If reanalysis of the CCB is required, reanalyze a new pair of CCV, CCB before proceeding with the analysis of any additional samples.

11.11 For one sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution should be prepared. For the serial dilution, a 1:5 dilution should be made on the sample. The results of the 1:5 dilutions should agree within 10 percent of the true value as long as the sample is greater than 50 times of the instrument detection limit for that element.

11.12 Post-digest spikes may also be used to determine potential interferences. Check with the metals supervisor for further information on when a post-digest spike should be performed.

11.13 For any readings that exceed the 90% of the linear range for a given element, a dilution is required. After a high reading, the sample following the high one must be examined for possible carryover. Verification may be necessary by rinsing the lines with an acid solution and then rereading the sample.

11.14 For the interelement spectral interference corrections to remain valid during sample analysis, the interferent concentration must not exceed its linear range. If the interferent exceeds its linear range, or its correction factor is big enough to affect the element of interest even at a lower concentration, sample dilution with reagent blank and reanalysis is required. In these circumstance analyte detection limits are raised. Check with metals supervisor for more information.

11.14.1 Anytime that the interference is large relative to the sample, dilution may be required. Check with the metals supervisor for more information.

11.15 This method does not require the analysis of an interfering element check solution at the end of the run. However, this may be required to meet other method and/or client requirements. Run the ICSA and ICSAB solutions every 8 hours unless otherwise instructed by the metals lab supervisor or manager.

11.16 For any readings where the internal standard is outside of the range of 60 to 125% of the internal standard level in the calibration blank, then the sample should be diluted until the internal standard is within that range.

11.16.1 For work following DoD document, internal standard intensity within 30-120% of the intensity of the IS in the ICAL.

11.17 All the data are automatically saved during the analysis to **Results.mdb** file. The data can be export to LIMS using Data Management software during the run or at the end of the run. The data must be reviewed in the LIMS as outlined in the inorganic data review SOP.

11.18 Example of an ICP Analytical Sequence (See Table 11-1)

Table 11-1

A/S Location	Sample Type	Sample Description
1	QC	Standard 1(Calib. Blank)
2	QC	Standard2
3	QC	Standard 3
3	QC	CCV
1	QC	ICB
8	QC	ICV
1	QC	ICB
3	QC	ICCV
1	QC	ICB
6	QC	CRI
4	QC	ICSA
5	QC	ICSAB
3	QC	CCV
1	QC	CCB
	Batch QC	MB1
	Batch QC	B1 (BSP)

A/S Location	Sample Type	Sample Description
	Field	Sample 1
	Field	Serial Dilution (SD1)
	Field	S1 (MS)
	Field	S2 (MSD)
	Field	Sample 5
	Field	Sample 6
	Field	Sample 7
3	QC	CCV
1	QC	CCB
	Field	Sample 8
	Field	Sample 9
	Field	Sample 10
	Field	Sample 11
	Field	Sample 12
	Field	Sample 13
	Field	Sample 14
	Field	Sample 15
	Field	Sample 16
	Field	Sample 17
3	QC	CCV
1	QC	CCB
	Field	Sample 18
	Field	Sample 19
	Field	Sample 20
	Field	Sample 19
	Field	Sample 20
	Field	Sample 5 dilution 5
	Batch QC	MB1
	Batch QC	B1(BSP)
3	QC	CCV
1	QC	CCB

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Calculations

12.1.1

$$\frac{\text{(Spiked Result)}}{\text{Amount Spiked}} \times 100 = \text{Blank Spike (LCS) Recovery}$$

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{Amount Spiked}} \times 100 = \text{Matrix Spike Recovery}$$

$$\frac{(\,|\text{Sample Result} - \text{Duplicate Result}|\,)}{\text{(Sample Result + Duplicate Result)/2}} \times 100 = \text{Duplicate RPD}$$

or

$$\frac{(\,|\text{MS Result} - \text{MSD Result}|\,)}{\text{(MS Result + MSD Result)/2}} \times 100 = \text{MSD RPD}$$

12.1.2 Determine the concentration of silicon and calculate silica

2.139 [Si] = mg/L SiO_2

12.1.3 Determine separate concentrations of Ca and Magnesium. Use that information for calculation of Total Hardness as mg equivalent $CaCO_3$/L (SM2340B)

2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]= mg equivalent $CaCO_3$/L

12.2 DOCUMENTATION REQUIREMENTS

12.2.1 If samples or QC checks require reanalysis, a brief explanation of the reason must be documented in the raw data. All instrument data should be exported to the LIMS system. IEC factors must be printed out each day and a copy is included with the raw data for all associated runs.

12.2.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed. The Accutest Lot Number must be cross-referenced on the standard vial.

12.2.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

12.2.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.2.5 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

12.3 INSTRUMENT MAINTENANCE

12.3.1 Recommended periodic maintenance includes the items outlined below.

12.3.2 Change the pump tubing weekly or as needed.

12.3.3 Clean the filter on the recirculating pump approximately once a month and dust off the power supply vents every one to two weeks.

12.3.4 Clean the nebulizer, torch, and injector tube every two to four weeks or more often as needed.

12.3.4.1 Replace torch and injector tube with clean assembly. Soak in 1:1 nitric acid and rotate daily with a clean one.

12.3.4.2 Rinse spray chamber with DI water

12.3.4.3 Replace nebulizer with a clean nebulizer. Soak nebulizer in Fluka RBS 25 (surfactant) solution and in 1:1 Nitric acid and rotate daily.

12.3.5 Change the sampler tip as needed (every one to two months).

12.3.6 Clean the slides on the autosampler with methanol and wipe them with a KimWipe saturated with Teflon spray a minimum of once per day.

12.3.7 Check and clean the following filters every one to two weeks:

- 2 filters on the back of the polychromator controller compartment
- one filter on the back of the power source
- one filter below the torch compartment

12.4 Archiving Data – Archived data every 2 months or as often as needed. Refer to MET005-0 Archiving and Restoring ICP data SOP.

13.0 POLLUTION PREVENTION

13.1 All methods will refer to the Waste Handling SOP or Safety Manual

14.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QC MEASURES

14.1 All methods will refer to Section 9.0

15.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

15.1 All method will refer to Section 9.0

16.0 CONTINGENCIES OF HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

16.1 Analysts will report any out of control or unacceptable data to the Laboratory Director, Operations Manager, or QA Manager prior to re-processing samples. A non-conforming QC data report is generated and issued to the QA Manager.

17.0 WASTE MANAGEMENT –All methods will refer to the Waste Handling SOP or Safety Manual

17.1 Non hazardous aqueous wastes.

17.2 Hazardous aqueous wastes.

17.3 Chlorinated organic solvents.

17.4 Non-chlorinated organic solvents.

17.5 Hazardous solid wastes.

18.0 METHOD PERFORMANCE

18.1 U.S. EPA SW846 6010B, Revision 2, December 1996

19.0 REFERENCES

19.1 U.S. EPA SW846 6010B, Revision 2, December 1996

19.2 Hardness by Calculation, SM2340B, Standard Methods for the Examination of Water and wastewater. 18th Edition, 1992

19.3 Perkin Elmer, OPTIMA 5300 DV ICP-AES manual

21.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

Table 20.1 HIGH STANDARD, ICV, CCV, CRIA and CRID LEVELS

Elements	Calibration Standard ppm	Initial Verification Calibration ppm	Continuing Calibration Verification ppm	CRIA Conc. mg/l	CRID Conc. mg/l
Al	50	1	25	0.2	0.1
Sb	2	1	1	0.03	0.01
As	5	1	2.5	0.03	0.02
Ba	1	1	0.5	0.1	0.01
Be	0.5	1	0.25	0.005	0.001
B	1	1	0.5	0.1	0.01
Cd	1.5	1	0.75	0.002	0.001
Ca	50	1	25	1	0.2
Cr	0.2	1	0.1	0.005	0.002
Co	1	1	0.5	0.005	0.002
Cu	1	1	0.5	0.005	0.002
Fe	50	1	25	0.05	0.025
Pb	5	1	2.5	0.01	0.006
Li	2	1	1	0.01	0.005
Mg	50	1	25	1	0.2
Mn	1	1	0.5	0.005	0.002
Mo	1	1	0.5	0.005	0.004
Ni	0.2	1	0.1	0.005	0.004
K	50	10	25	0.5	0.25
Se	2	1	1	0.03	0.02
Si	1	5	0.5	0.1	0.05
Ag	0.5	0.5	0.25	0.005	0.002
Na	50	1	25	1	0.5
Sr	0.5	1	0.25	0.005	0.005
Tl	2	1	1	0.03	0.002
Sn	2	1	1	0.05	0.05
Ti	1	1	0.5	0.002	0.002
V	1	1	0.5	0.005	0.003
Zn	1.5	1	0.75	0.01	0.005
Bi					
W					

TABLE 1A: ANALYTICAL LINES ON THE OPTIMA 5300 DV	
Element	**Wavelength**
Al	308.215
Sb	206.834
As	188.980
Ba	493.408
Be	313.03
B_	249.680
Cd	228.800
Ca	317.972
Cr	267.710
Co	228.613
Cu	324.756
Fe	259.941
Pb	220.352
Li	610.362
Mg	279.078
Mn	257.607
Mo	202.031
Ni	231.604
K_	766.490
Se	196.027
Si	251.609
Ag	328.068
Na	589.601
Sr	421.551
Tl	190.794
Sn	189.927
Ti	334.940
V	292.402
Zn	213.857
Y	371.029

ACCUTEST LABORATORIES
Standard Operating Procedure

Lab Manager

QA Manager

Effective Date: 8-4-10

TEST NAME **DETERMINATION OF ORGANOCHLORINE PESTICIDES USING GC SYSTEM**

METHOD REFERENCE SW846 8081A (Revision 1, December 1996), SW846 8000C, EQA044 (Manual integration)

***Revised sections*: 9.6.2, 10.2.1 and Table 2**

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the analytical procedures, which are utilized by Accutest to acquire samples for analysis of organochlorine pesticides and screening of polychlorinated biphenyls (PCBs) by gas chromatography with Electron Capture Detectors (ECD).

1.2 The method is applicable to extracts from solid and liquid matrices. The compounds listed in Table 1 are determined by a dual-column analysis system.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 15 g for solids) is extracted using the appropriate matrix-specific sample extraction technique. Liquid samples are extracted at neutral pH with methylene chloride using Method 3510 (separatory funnel). Solid samples are extracted using Method 3545, Pressurized Fluid Extraction. A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes. Cleanups include Florisil (Method 3620), silica gel (Method 3630), gel permeation chromatography (Method 3640), and sulfur (Method 3660).

2.2 After cleanup, the extract is analyzed by injecting a 2-µL sample that is split between dual narrow-bore fused silica capillary columns that are mounted in a single gas chromatograph with electron capture detectors (GC/ECD).

2.3 The peaks detected are qualitatively identified by comparison to retention times specific to the known target list of compounds on two different column types (primary and confirmation).

2.4 If sensitivity permits, the positive hit should be confirmed by GC/MS method 8270.

2.5 Once identified the compound is quantitated by external standard techniques with an average calibration factor generated from a calibration curve.

3.0 REPORTING LIMIT & METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. RL's may vary depending on matrix and sample volumes or weight and percent moisture. Refer to Table 1 for current reporting limits.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. The different types of blanks are Method Blank, Instrument Blank, Storage Blank, and Sulfur Blank.

FIELD BLANK – an analytical sample prepared from organic-free water and carried through the sampling handling protocol serves as a check for contamination.

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTINUING CALIBRATION - analytical standard run every 12 hours and at the end of analytical sequence to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the term's continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the electron capture detector to the target compounds.

MATRIX - the predominant material of which the sample to be analyzed is composed. A sample matrix is either water or soil/sediment. Matrix is <u>not</u> synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD DETECTION LIMITS (MDLs) – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs should be determined approximately once per year for frequently analyzed parameters.

PERCENT DIFFERENCE (%D) - to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

RELATIVE PERCENT DIFFERENCE (RPD) - to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

RELATIVE RESPONSE FACTOR (RRF) - a measure of the instrument response of an analyte. Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RETENTION TIME (RT) - the time required (in minutes) for a standard compound to elute from a chromatographic column.

SURROGATES - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recoveries. Surrogate are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

INSTRUMENT BLANK - a system evaluation sample containing lab reagent grade water with internal standards and/or surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. 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 data handling sheets must be made available to all personnel involved in these analyses.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and the BHCs. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/Mass approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 INTERFERENCES

6.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

6.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other stages of sample processing. Refer to "The Preparation of Glassware for Extraction of Organic Contaminants" SOP for practices utilized in the extraction department.

6.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary from source to source. Interferences such as sulfur and phthalate are treated with copper and alumina by organics preparation respectively.

6.3.1 The presence of elemental sulfur will result in broad peaks that interfere with detection of early-eluting organochlorine pesticides. Method 3660 is suggested for removal of sulfur.

6.3.2 Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interference from phthalate esters.

6.4 Waxes, lipids, and other high molecular weight materials can be removed by method-3640 (Gel Permeation Chromatography-GPC column cleanup).

6.5 To reduce carryover when high-concentration samples are sequentially analyzed, the syringe must be rinsed out between samples with solvent.

6.6 In the case where an unusually concentrated sample is encountered, it should be followed by the analysis of an instrument blank. An instrument blank is a sample containing hexane with surrogate standards added at 20 ppb. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

7.0 SAMPLE PRESERVATION AND HOLDING TIME

7.1 PRESERVATION

7.1.1 Water Samples

7.1.1.1 Collect samples in 1 liter glass amber bottles without preservatives.

7.1.1.2 A liter of an unpreserved sample is required for extraction. Additional sample volume is necessary for any samples used for matrix spike and matrix spike duplicates. Therefore, 3 liters of at least one sample in every group of 20 field samples are required for analysis to accommodate all quality control requirements.

7.1.2 Soil Samples

7.1.2.1 Samples are collected in a 300-ml amber glass sample bottle. No preservative is required.

7.1.3 Sample should be taken with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing possible phthalate contamination.

7.1.4 The samples must be protected from light and refrigerated at $4^0C(\pm 2^0C)$ from the time of receipt until extraction and analysis.

7.2 HOLDING TIME

7.2.1 Aqueous sample must be extracted within 7 days of sampling.

7.2.2 Soil sample must be extracted within 14 days of sampling.

7.2.3 Extracts must be analyzed within 40 days following extraction.

8.0 APPARATUS AND MATERIALS

8.1 GAS CHROMATOGRAPH SYSTEM

8.1.1 Gas Chromatograph – Agilent or Hewlett Packard Models 6890 and 5890. The analytical system is completed with a temperature programmable gas chromatograph and all required accessories including syringes, capillary chromatographic columns, and gases. The capillary column is directly coupled to the source. The injection port is designed for splitless injection with capillary columns.

8.1.2 Columns

8.1.2.1 Column pair 1

8.1.2.1.1 30 m x 0.32 mm ID, 0.5 µm film thickness fused silica, DB-1701 narrow-bore capillary column or equivalent.

8.1.2.1.2 30 m x 0.32 mm ID, 0.5 µm film thickness fused silica, DB-5 narrow-bore capillary column.

8.1.2.2 Column pair 2

8.1.2.2.1 30 m x 0.32 mm ID, 0.5 µm film thickness fused silica, RTX CLPI narrow-bore capillary column or equivalent.

8.1.2.2.2 30 m x 0.32 mm ID, 0.25 µm film thickness fused silica, RTX CLPII narrow-bore capillary column or equivalent.

8.1.3 Detectors

8.1.3.1 Electron Capture Detectors (HP).

8.1.3.2 Micro Electron Capture Detectors (HP).

8.2 AUTOSAMPLER

8.2.1 Agilent or Hewlett Packard Models: 7673A, 7683, 7643A capable of holding 100 of 2ml crimp vials.

8.3 DATA SYSTEM

8.3.1 MSD interfaced to the gas chromatograph which allows the continuous acquisition and storage on machine-readable media (disc) of all chromatographic data obtained throughout the duration of the analysis.

8.3.2 The ENVIROQUANT (PC) data system is capable of quantitation using multipoint calibration.

8.3.3 Legato Networker with lookup database on 4mm DAT tape for long term, off line magnetic storage of data.

8.4 SYRINGES

8.4.1 Manually held ul graduated syringes, various volumes (Hamilton or equiv.).

8.4.2 10 µl graduated, auto sampler (Hamilton or equiv.).

8.5 VOLUMETRIC FLASKS, Class A.

9.0 REAGENTS AND STANDARDS

9.1 Refer to Accutest Sample Preparation SOPs EOP001 and EOP040 for reagents and standards used for sample extraction.

9.2 Solvents - Ultra pure, chromatography graded Hexane.

9.3 Stock Standard Solutions

9.3.1 Two separate sources of commercially prepared standards with traceability documentation are used.

9.3.1.1 Pesticides Mixtures containing one or more of the following compounds: alpha-BHC, beta-BHC, delta-BHC, gamma-BHC(Lindane), Heptachlor, Aldrin, Heptachlor Epoxide, Endosulfan I, Dieldrin, 4,4'-DDE, Endrin, Endosulfan II, 4,4'-DDD, Endosulfan sulfate, 4,4—DDT, Methoxychlor, Endrin ketone, Endrin Aldehyde, alpha-Chlordane & gamma-chlordane.

9.3.1.2 Individual standards containing Toxaphene, Chlordane and Mirex.

9.4 Working Solutions

9.4.1 Prepare working solutions, using stock solution, in hexane, as needed, that contain the compounds of interest, either singly or mixed together. Refer to Table 3 for details.

9.5 Calibration Standards

9.5.1 Initial Calibration Standards

9.5.1.1 Calibration standards are prepared at a minimum of five concentrations, including surrogates, from the above working solutions. Suggested levels and preparations are shown in Table 4A.

9.5.1.2 Separate calibration standards are required for each multi-component target analyte (i.e., Toxaphene and Chlordane). Unless otherwise necessary for a specific project, such as Ohio VAP or the Dept. of Defense (DoD), a single calibration standard near the mid-point of the expected calibration range of each multi-component analyte is employed. Refer to Table 4B and 4C for preparation scheme. Optional curves as shown on Table 4D and 4E may also be used for a multi-point calibration per project's specification.

9.5.2 Continuing Calibration Verification (CCV)

9.5.2.1 Continuing calibration checks containing all the single-component analytes are prepared at concentrations of 10 µg/l, 25 ug/l and 50 µg/l as described in Table 5. During analysis, these alternate concentrations are run to check the initial calibration.

9.5.2.2 In situations where only Toxaphene or Chlordane is of interest for a specific project and for Ohio VAP multi-level calibration checks of each multi-component analyte of interest may be prepared and analyzed throughout the analytical sequence.

9.6 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

9.6.1 Prepare the ICV standards from separate sources of stock standards from the calibration curve. An ICV must be run for the individual pesticides and the multi-component compounds, toxaphene and chlordane.

9.6.2 The ICV must be analyzed immediately following the initial calibration.

9.7 Surrogates

9.7.1 Tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are used as surrogate standards for this method.

9.7.2 A calibration range must be constructed for the surrogate compounds. Accordingly, appropriate amounts of surrogates are mixed with each calibration solution to define a range similar to the target compounds.

9.7.3 Surrogate compounds are also contained in continuing calibration checks, and second source calibration check standard.

9.7.4 Spike each sample, QC sample and blank with an appropriate amount of corresponding surrogate spiking solution, prior to extraction, for a final concentration in the extract of 40 µg/l of each surrogate compound.

9.8 Breakdown Evaluation Solution

9.8.1 The DDT and Endrin breakdown evaluation solution is prepared in hexane as outlined in Table 6.

9.9 Storage of Standards

9.9.1 Store unopened stock standard solutions according to the manufacturer's documented holding time and storage temperature recommendations. Protect from light.

9.9.2 Store all other working standard solutions in glass vials with Teflon lined screw caps at 4°C (± 2°C) in the dark.

9.9.3 Opened stock standard solutions must be replaced after 6 months or sooner if manufacturer's expiration date comes first or comparison with quality control check samples indicates a problem.

9.9.4 All other standards must be replaced after six months or sooner if routine QC indicates a problem or manufacturer's expiration date comes first.

10.0 CALIBRATION

10.1 Initial Calibration

10.1.1 The calibration range covered for all single-component analytes employs at least five of the following standards: 2, 5, 10, 25, 50, and 100* µg/l (*this point may be dropped if it exceeds the linear range of the instrument). The method reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Lower concentration standard may be needed to meet the reporting limit requirements of state specific regulatory program. The linear range covered by this calibration is the highest concentration standard. Calibration is performed for both the primary and secondary columns.

10.1.2 A calibration range must be constructed for each surrogate compound. Accordingly, add appropriate amounts of each surrogate compound to the calibration solution to define a range similar to the target compounds.

10.1.3 Unless otherwise necessary for a specific project, the analysis of the multi-component analytes (such as: Toxaphene or Chlordane) employs a single-point calibration. This single calibration standard is included with the initial calibration of the single component analytes for pattern and retention time recognition. For Ohio VAP and Dept. of Defense (DoD) projects an initial 5 point calibration is required for these analytes if there are positive hits.

10.1.4 Aliquot proper amount of each calibration standard into a 2-ml crimp top vial.

10.1.5 Each analyte is quantitatively determined using the external standard technique. The Calibration Factor (CF) is defined in Section 14.1.

10.1.6 For the initial calibration to be valid, the percent relative standard deviation (% RSD) (see Section 14.2) must be less than 20 % for each analyte of interest on each column. If any analyte exceeds the 20% acceptance limit for a given calibration, corrective action must be taken.

10.1.6.1 If the problem is associated with a standard, reanalyze the standard and recalculate the RSD.

10.1.6.2 Alternatively, narrow the calibration range by replacing the low or the high calibration standard that cover a narrow range.

10.1.6.2.1 The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

10.2 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

10.2.1 The initial calibration is verified with a second source calibration check standard from an external source (Section 9.6). It must be performed right after the initial calibration. An ICV must be run for the individual pesticides and the multi-component compounds, toxaphene and chlordane.

10.2.2 The percent difference (%D) (Section 14.3) for this standard must meet the %D criteria of 15% used for calibration verification on each column. For toxaphene and chlordane, the standard must meet 15%D of the total average of the peaks.

10.2.2.1 If %D is greater than 15%, reanalyze the second source check. If the limit cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

10.2.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, prepare a third source and repeat the process. Make fresh calibration standards using one of the two standard sources that matches each other.

10.3 Continuing Calibration Verification (CCV)

10.3.1 Continuing calibration check standards (Section 9.5.2) must be acquired at the beginning of each 12 hour shift, after every 10 injections not to exceed 12 hours and at the end of the analysis sequence. Analysts should alternate the use of the two different concentration mixtures for calibration verification depending on the range of the curve.

10.3.2 For the continuing calibration to be valid, the percent difference (%D) must be less than 15 % for each compound of interest on each column.

10.3.3 Each sample analysis must be bracketed by periodic analyses of acceptable calibration verification standards followed by an instrument blank, run after 10 injections or 12-hours, whichever is more frequent. If %D criteria fails during a mid sequence calibration check or at the end of the analysis sequence, a continuing calibration check is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed. In situations where the first check fails to meet the criteria, the instrument logbook should have clear documented notations as to what the problem was and what corrective action was implemented to enable the second check to pass.

10.3.4 When a calibration verification standard fails to meet the QC criteria at the end of the analysis sequence, all samples injected after the last standard that met the QC criteria must be evaluated to prevent mis-quantitations, and re-injection of the sample extracts may be required.

10.3.4.1 The analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed when the calibration standard response is above the initial calibration response.

10.3.4.2 Whether the analyte was detected in the specific samples analyzed during the analytical shift, or the calibration standard response is below the initial calibration response, then the extracts for those samples need to be reanalyzed.

10.3.5 Each subsequent injection of a continuing calibration standard must be checked against the retention time windows established in Section 11.0. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

11.0 RETENTION TIME WINDOWS

11.1 Retention time windows must be calculated for each analyte and surrogate on each GC column and whenever a new chromatographic column is installed, when a new initial calibration is analyzed or when there are significant changes in the operating conditions. The retention time windows must be reported with the analysis results in support of the identifications made.

11.2 Employ the following approach to establish retention time windows.

11.2.1 Make three injections of all single component standard mixture and multi-response products at approximately equal intervals during the 72-hr period.

11.2.2 Calculate the mean and standard deviation of the three absolute retention times- recording the retention time to three decimal places (e.g. 10.015 min)- for each single component pesticide.

11.2.3 For multi-response pesticides, choose five major peaks and calculate the mean and standard deviation of the three retention times for those peaks. The peak chosen should be fairly immune to losses due to degradation and weathering in the samples.

11.2.4 In those cases where the standard deviations of the retention time window for a particular pesticide is <0.01 minutes, the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

11.2.5 The width of the retention time window for each analyte and surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation is employed, the width of the window will be 0.03 minutes.

11.2.6 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

12.0 PROCEDURE

12.1 Sample Extraction

12.1.1 In general, water samples are extracted at a neutral pH with methylene chloride using a separate funnel (Method 3510) (Refer to SOP: EOP001). Solid samples are extracted using Method 3545, Pressurized Fluid Extraction. (Refer to SOP: EOP040).

12.2 Sample Cleanup

12.2.1 Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. Refer to the appropriate SOPs for details.

12.2.1.1 If a sample is of biological origin, or contains high molecular weight materials, the use of Method 3640 (GPC cleanup - pesticide option) is recommended. Frequently, one of the adsorption chromatographic cleanups (alumina, silica gel, or florisil) may also be required following the GPC cleanup.

12.2.1.2 Method 3610 (alumina) may be used to remove phthalate esters.

12.2.1.3 Method 3620 (florisil) may be used to separate organochlorine pesticides from aliphatic compounds, aromatics, and nitrogen-containing compounds.

12.2.1.4 Method 3630 (silica gel) may be used to separate single component organochlorine pesticides from some interferants.

12.2.1.5 Elemental sulfur, which may be present in certain sediments and industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur should be removed by the technique described in Method 3660.

12.3 Instrument Conditions

12.3.1 Recommended instrument conditions are listed in Table 2. Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification should be approved by team leader/manager.

12.4 DDT and Endrin Breakdown Evaluation

12.4.1 DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin aldehyde indicates breakdown.

12.4.2 Before the initial calibration and at the beginning of each 12-hour shift, inject 1 µl of an evaluation standard directly on column. (Refer to Section 9.8).

12.4.3 Calculate the percent breakdown for Endrin and DDT (Section 14.7) and save the breakdown report in the LIMS system.

12.4.4 If degradation of either DDT or Endrin exceeds 15%, injector maintenance should be completed before proceeding with calibration. Refer to EQA036-01 for GC system maintenance utilized in the lab.

12.5 Initial Calibration

12.5.1 See Section 10.1.

12.6 Initial Calibration Verification (ICV)

12.6.1 Refer to Section 10.2.

12.7 Continuing Calibration Verification (CCV)

12.7.1 Refer to Section 10.3.

12.8 Sample Analysis (Primary)

12.8.1 All samples and quality control samples are injected into the Gas Chromatograph using the autosampler. Program the sampler for an appropriate number of syringe rinses and a 1ul or 2 µl injection size. A splitless injection technology is used.

12.8.2 Sample concentrations are calculated by comparing sample responses with the initial calibration of the system (Section 14.4). If sample response exceeds the limits of the initial calibration range, dilute the extract and reanalyze. Extracts should be diluted so that all peaks are on scale, as overlapping peaks are not always evident when peaks are off scale.

12.8.3 Sample injections may continue for as long as the calibration verification standards meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

12.8.4 If chromatographic peaks are masked by the presence of interferences, further sample cleanup is necessary. Refer to Section 12.2 for extract cleanup alternatives.

12.8.4.1 If extract cleanup is required, all QC samples must also be processed through the cleanup method.

12.9 Confirmation Analysis

12.9.1 Confirmation analysis is to confirm the presence of all compounds tentatively identified in the primary analysis.

12.9.1.1 All instrument performance quality control criteria for calibration and retention times must be satisfied on the confirmation analysis.

12.9.2 Each tentative identification must be confirmed using either a second GC column of dissimilar stationary phase or using another technique such as GC/MS.

12.9.2.1 The primary and secondary analysis is conducted simultaneously in the dual-column analysis.

12.9.2.2 GC/MS confirmation may be used in conjunction with dual-column analysis if the concentration is sufficient for detection in GC/MS, normally a concentration of approximately 10 ng/µl in the final extract for each single component compound is required. Method 8270 is recommended as a confirmation technique when sensitivity permits.

12.9.3 Once the identification has been confirmed, the agreement between the quantitative results on both columns should be checked.

12.10 Sample Dilution

12.10.1 Establish dilution of sample in order to fall within calibration range or to minimize the matrix interference.

* Utilize screen data (specific project only).
* Utilize acquired sample data.
* Utilize the history program or approval from client/project.
* Sample characteristics (appearance).

12.10.2 If no lower dilution has been reported, the dilution factor chosen should keep the response of the largest peak for a target analyte in the upper half of the initial calibration range of the instrument.

12.10.3 Preparing Dilutions.

12.10.3.1 Prepare sample dilutions quantitatively. Dilute a stored sample extract, if available with hexane using logical volume to volume ratios, i.e., 1:5, 1:10, 1:50, etc.

12.10.3.2 Syringe dilutions. – Refer to Table 7 for dilutions. A calibrated 1ml syringe must be used to prepare dilutions. Gently shake to disperse the extract throughout the solvent prior to loading on the auto-sampler tray for further analysis.

12.10.3.3 Volumetric Flask Dilutions - Dilutions can also be made with a Class A volumetric flask. Measure the appropriate sample extract volume in a calibrated syringe and bring to final volume with dilution solvent in a Class A volumetric flask. Gently shake to disperse the extract throughout the solvent and transfer to auto-sampler vial for analysis.

12.11 Data interpretation

12.11.1 Qualitative identification

12.11.1.1 Analyst shall identify the targeted compounds with competent knowledge interpreting retention time and/or chromatographic pattern by comparison of the sample to the standard of the suspected compound. The criteria required for a positive identification are:

12.11.1.1.1 The sample component must elute at the absolute retention time window (Refer to Section 11.0) for both primary and confirmation run.

12.11.1.1.2 For the multi-response pesticides, at least five major peaks are selected. The retention time window for each peak is determined from the initial calibration analysis. Identification of a multi-component analyte in the sample is based on pattern recognition in conjunction with the elution of these five peaks within the retention time windows of the corresponding peaks of the standard on both GC columns.

12.11.1.1.3 Be aware of matrix interfering effects on peak shape and relative peak ratios that could distort the pattern. Interpretation of these phenomena may require a highly experienced chromatographer or at least a second opinion.

12.11.2 Quantitative analysis

12.11.2.1 When a target compound has been identified, concentration (see section 14.4) will be based on the integrated area/or height of the peak and calculated by external standard technique. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined.

12.11.2.2 For multi-response pesticides, usually the areas of 5 peaks are used for quantitation to calculate the calibration factors for those peaks, but fewer may be used depending on the extent of matrix interferences. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the resulting concentrations are averaged to provide the final result for the sample.

12.11.2.3 When sample results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results must be evaluated after the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula in Section 14.6. Report the lower result.

12.11.2.3.1 A program to perform the RPD calculation had been developed and incorporated into ENVIROQUANT software.

12.11.2.3.2 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

12.11.2.3.3 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the lower result with the footnote (remark) indicating "More than 40% RPD for detected concentrations between two GC columns".

13.0 QUALITY CONTROL

13.1 QC Requirements Summary

DDT and Endrin Breakdown Evaluation	Every 12-hour shift
ICV -Second Source Calibration	Following initial calibration
Continuing Calibration Checks	Every 12-hour shift or 10 injections(whichever is more frequent) and at the end of analysis sequence
Method Blank	One per extraction batch* or per day for a running batch
Blank Spike	One per extraction batch* or per day for a running batch
Matrix Spike	One per extraction batch*
Matrix Spike Duplicate	One per extraction batch*
Surrogate	Every sample and standard

*The maximum number of samples per batch is twenty or per project specification.

13.2 DDT and Endrin Breakdown Evaluation

13.2.1 Refer to Section 12.4.

13.3 Initial Calibration Verification (ICV) - Second Source Calibration Check

13.3.1 Refer to Section 10.2.

13.4 Continuing Calibration Verification (CCV)

13.4.1 Refer to Section 10.3.

13.5 Method Blank

13.5.1 The method blank is either DI water or sodium sulfate (depending upon the sample matrix) which must be extracted with each set of 20 or less samples. For a running batch, a new method blank is required for each different extraction day. The method blank are then extracted and run through any clean-up procedures along with the other samples in that batch.

13.5.2 If the method blank contains a target analyte above its MDL in LIMS, the entire batch must be re-extracted and re-analyzed.

13.5.3 Surrogate compounds are added to the method blank prior to extraction and analysis. If the surrogate accuracy in the blank does not meet criteria in LIMS, the entire batch must be re-extracted and re-analyzed.

13.6 Blank Spike

13.6.1 A blank spike must be extracted with each set of 20 or less samples. For a running batch, a new blank spike is required for each different extraction day. The blank spike consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. A separate blank spike may be needed if the sample requires Chlordane and/or Toxaphene. It is spiked with the same analytes at the same concentrations as the matrix spike/matrix spike duplicate.

13.6.1.1 For single-component analytes, the blank spike is prepared at 0.25 µg/l or 8.33 µg/kg on a dry weight basis.

13.6.1.2 For Toxaphene only analysis or per project specification, the blank spike is prepared at 5 µg/l or 167 µg/kg on a dry weight basis.

13.6.1.3 For Chlordane only analysis or per project specification, the blank spike is prepared at 4 µg/l or 133 µg/kg on a dry weight basis.

13.6.2 The blank spike recoveries should be assessed using in house limits specified in LIMS.

13.6.3 If a blank spike is out of control, the following corrective actions must be taken. In the case where the blank spike recovery is high and no hits reported in associated samples and QC batch the sample results can be reported with footnote (remark) and no further action is required.

13.6.3.1 Check to be sure that there are no errors in the calculations, or spike solutions. If errors are found, recalculate the data accordingly.

13.6.3.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample batch.

13.6.3.3 If no problem is found, re-extract and reanalyze the sample batch.

13.7 Matrix Spike (MS)/Matrix Spike Duplicate (MSD)

13.7.1 One sample is randomly selected from each extraction batch of similar matrix types and spiked in duplicate to determine whether the sample matrix contributes bias to the analytical results.

13.7.2 A separate matrix spike and matrix spike duplicate set may be needed if the sample requires Chlordane and/or Toxaphene. Matrix spikes are prepared by spiking an actual sample for a concentration of 0.25 µg/l or 8.33 µg/kg on a dry weight basis for pesticides, 5 µg/l or 167 µg/kg for Toxaphene, 4 µg/l or 133 µg/kg for Chlordane.

13.7.3 Assess the matrix spike recoveries and relative percent difference (RPD) against the in house control limits in LIMS.

13.7.4 If the matrix spike accuracy of any individual compound is out of control, the accuracy for the compound in the blank spike must be within control. Matrix interference is assumed and the data is reportable. No further corrective action is required.

13.8 Surrogates

13.8.1 Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogate standards. All blanks, samples, QC samples, and calibration standards contain surrogate compounds which are used to monitor performance of the extraction, cleanup (when used), and analytical system.

13.8.2 The recoveries (refer to Section 14.5) of the surrogates must be evaluated versus the surrogate control limits In LIMS developed by the laboratory.

13.8.3 If surrogate recovery is not within established control limits, corrective action must be performed if surrogate recoveries indicate that a procedural error may have occurred during the analysis of the sample.

13.8.3.1 Check the surrogate calculations for calculation or integration errors and perform corrections if detected.

13.8.3.2 Re-analyze the extract if calculation errors are not detected. If the surrogate recoveries for the re-analyzed extract are in control, report data from the re-analysis only.

13.8.3.3 If data from the reanalysis is also out of control, re-extract and reanalyze the sample.

13.8.3.4 If, upon reanalysis, the surrogate recoveries are acceptable, report the reanalysis data. If the holding time has expired prior to the reanalysis, report both the original and reanalysis results and note the holding time problem.

13.8.3.5 If the recovery is again not within limits, the problem is considered to be matrix interference. Submit both data sets with the original analysis being reported.

13.8.4 The retention time shift for surrogate must be evaluated after the analysis of each sample. The sample should be reanalyzed when the retention time of any surrogate compound is outside the retention window.

13.8.4.1 Reanalysis may not be required for samples having visible matrix interference, defined as excessive signal levels from target or non-target interfering peaks. This judgment should be approved by team leader or supervisor.

14.0 CALCULATIONS

14.1 Calibration Factor (CF).

$$CF = \frac{A_s}{C_s}$$

where: A_s = Area of the peak for the compound being measured.
C_s = Concentration of the compound being measured (µg/l).

14.2 Percent Relative Standard Deviation (% RSD).

$$\%RSD = \frac{SD}{CF_{av}} \times 100$$

where:
SD = Standard Deviation.
CF_{av} = Average calibration factor from initial calibration.

14.3 Percent Difference (% D).

$$\% D = \frac{|CF_{av} - CF_c|}{CF_{av}} \times 100$$

where:
CF_c = CF from continuing calibration (CBCHK).

14.4 Concentration (Conc.).

14.4.1 <u>For water:</u>

$$\text{Conc. } (\mu g/l) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{V_i}$$

14.4.2 For soil/sediment (on a dry weight basis):

$$\text{Conc. } (\mu g/kg) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{W_s \times S}$$

where:
A_c = Area of peak for compound being measured.
V_f = Final Volume of total extract (ml).
D = Secondary dilution factor.
V_i = Initial volume of water extracted (ml).
W_s = Weight of sample extracted (g).
S = (100 - % moisture in sample) / 100 or % solid/100.
M = Multiplier.

14.5 Percent Recovery (% R).

$$\%\ R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

14.6 Relative Percent Difference (RPD).

$$RPD = \frac{|C_1 - C_2|}{(1/2)(C_1 + C_2)} \times 100$$

where:
C_1 = Matrix Spike Concentration or the result on column 1.
C_2 = Matrix Spike Duplicate Concentration or the result on column 2.

14.7 Percent Breakdown.

$$\%\ \text{Breakdown for DDT} = \frac{\text{Total DDT degradation peak area}}{\text{Total DDT peak area}} \times 100$$

where:
Total DDT degradation peak area = DDE + DDD
Total DDT peak area = DDT + DDE + DDD

$$\%\ \text{Breakdown for Endrin} = \frac{\text{Total Endrin degradation peak area}}{\text{Total Endrin peak area}} \times 100$$

where:
Total Endrin degradation peak area = Endrin aldehyde + Endrin ketone.
Total Endrin peak area = Endrin + Endrin aldehyde + Endrin ketone.

15.0 DOCUMENTATION

15.1 The Analytical Logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

15.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in this log. For consistency, if surrogates are high or low indicate it as (↑) for high and (↓) for low.

15.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed, the page must be signed and dated by the respective person.

15.2.1 The Accutest Lot Number must be cross-referenced on the standard vial.

15.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

15.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

15.5 Unused blocks of any form must be x'ed or z'ed by the analyst before submitting the data for review.

15.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

16.0 DATA REVIEW AND REPORTING

16.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

16.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

16.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action before proceeding with analysis.

16.2.1 In some situation, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

16.3 Chromatogram Review. The chromatogram of each sample is evaluated for target compounds.

16.3.1 Check specific retention time windows for each target compound for the presence of the target compound in each chromatogram.

16.3.1.1 Each sample may require the reporting of different target compounds. Review the login to assure that the correct target compounds are identified.

16.3.2 The compound must be identified on the primary and confirmatory column before assigning a qualitative identification.

16.3.3 Manual integration of chromatographic peaks must be identified by the analysts by initialing and dating the changes made to the report.

16.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

17.0 POLLUTION PREVENTION & WASTE MANAGEMENT

17.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 17.2.

17.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

17.2.1 Non hazardous aqueous wastes.

17.2.2 Hazardous aqueous wastes

17.2.3 Chlorinated organic solvents

17.2.4 Non-chlorinated organic solvents

17.2.5 Hazardous solid wastes

17.2.6 Non-hazardous solid wastes

Table 1. Target Compound List and Reporting Limits			
Compound	**CAS No.**	**Water (µg/l)**	**Soil (µg/kg)**
alpha-BHC	319-84-6	0.02	1.18
beta-BHC	319-85-7	0.02	1.18
delta-BHC	319-86-8	0.02	1.18
Gamma-BHC (Lindane)	58-89-9	0.02	1.18
Heptachlor	76-44-8	0.02	1.18
Aldrin	309-00-2	0.02	1.18
Heptachlor epoxide	1024-57-3	0.02	1.18
Endosu1fan I	959-98-8	0.02	1.18
Dieldrin	60-57-1	0.02	1.18
4,4'-DDE	72-55-9	0.02	1.18
Endrin	72-20-8	0.02	1.18
Endosulfan II	33213-65-9	0.02	1.18
4,4'-DDD	72-54-8	0.02	1.18
Endosulfan sulfate	1031-07-8	0.02	1.18
4,4'-DDT	50-29-3	0.02	1.18
Methoxychlor	72-43-5	0.02	1.18
Endrin ketone	53494-70-5	0.02	1.18
Endrin aldehyde	7421-93-4	0.02	1.18
α-Chlordane	5103-71-9	0.02	1.18
γ-Chlordane	5103-74-2	0.02	1.18
Mirex	2385-85-5	0.02	1.18
Chlordane (technical)	12789-03-6	0.50	29.4
Toxaphene	8001-35-2	0.25	14.7

Table 2. RECOMMENDED OPERATING CONDITION	
Gas Chromatograph/Electron Capture Detectors	
Carrier Gas	Helium
Make-up gas	5 % Methane/ 95 % Argon
Make-up gas flow	*40 ml/min
Injection port temperature	*280°C
Injection type	Splitless
Detector temperature	*320°C
Column flow	2 ml/min
Gas Chromatograph Temperature Program*	
Initial temperature	*160°C
Time 1	*0 minutes
Column temperature rate 1	*45 degrees/min
Temperature 1	*200°C
Column temperature rate 2	*7 degrees/min
Temperature 2	*260°C
Column temperature rate 3	*50 degrees/min
Final temperature	*305°C
Time 3	*0.8 minutes
Total run time	10-20 minutes

*Parameter modification allowed for performance optimization as long as QC criteria are achieved.

Table 3. Pesticides and Surrogates Working Solution and ICV	
Stock Solution	**Volume Added**
Pesticides Mixture (1,000 µg/ml)	0.1 ml
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	0.5ml
Mirex (1000ug/ml) (optional)	0.1ml
Hexane	9.4 ml (or 9.3 ml with Mirex)
Total	10.0 ml

Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution: Prepared by measuring 0.1 ml of 1,000 µg/ml of pesticides mixture, 0.5ml of 200 µg/ml pesticides surrogate std spiking solution and bringing to 10 ml with hexane. Note larger or smaller volumes of standards may be prepared, as needed using the same ratios.

ICV is prepared in the same way, but a second source is used.

Table 4A. Pesticides Calibration Standard Solutions					
Solution	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration(µg/l)**
Standard A	Pesticides Mixture	10	500	50	100
	Surrogates	10			100
Standard B	Pesticides Mixture	10	250	50	50
	Surrogates	10			50
Standard C	Pesticides Mixture	10	125	50	25
	Surrogates	10			25
Standard D	Pesticides Mixture	10	50	50	10
	Surrogates	10			10
Standard E	Pesticides Mixture	10	25	50	5
	Surrogates	10			5
Standard F	Pesticides Mixture	10	10	50	2
	Surrogates	10			2

Standard A: Prepared by measuring 500 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard B: Prepared by measuring 250 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard C: Prepared by measuring 125 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard D: Prepared by measuring 50 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard E: Prepared by measuring 25 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard F: Prepared by measuring 10 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Table 4B. Toxaphene Calibration Standard Solution (20µg/ml) & ICV	
Stock Solution	**Volume Added (µl)**
Toxaphene stock (4000 µg/ml)	125
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	100
Hexane	24775
Total	25000

Toxaphene (20 µg/ml) and Surrogates (0.80 µg/l) Calibration Solution: Prepared by measuring 125 µl of 4000 µg/ml of Toxaphene stock solution, 100 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 4C. Chlordane Calibration Standard Solution (20 µg/ml) & ICV	
Stock Solution	**Volume Added (µl)**
Chlordane stock (2000 µg/ml)	250
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	100
Hexane	24650
Total	25000

Chlordane (20 µg/ml) and Surrogates (0.80 µg/ml) Calibration Solution: Prepared by measuring 250 µl of 2000 µg/ml of Chlordane stock solution, 100 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 4D. Multi-point Toxaphene Calibration Standards (optional)					
Solution	**Stock Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration(µg/l)**
Standard A	Toxaphene	20	3750	25	3000
	Surrogate Spiking	0.8	3750		120
Standard B	Toxaphene	20	2500	25	2000
	Surrogate Spiking	0.8	2500		80
Standard C	Toxaphene	20	1250	25	1000
	Surrogate Spiking	0.8	1250		40
Standard D	Toxaphene	20	625	25	500
	Surrogate Spiking	0.8	625		20
Standard E	Toxaphene	20	312.5	25	250
	Surrogate Spiking	0.8	312.5		10
Standard F	Toxaphene	20	62.5	25	50
	Surrogate Spiking	0.8	62.5		2

Standard A: Prepared by measuring 3750 µl of 20 µg/ml of Toxaphene stock solution, 3750 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard B: Prepared by measuring 2500 µl of 20 µg/ml of Toxaphene stock solution, 2500 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard C: Prepared by measuring 1000 µl of 20 µg/ml of Toxaphene stock solution, 1000 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard D: Prepared by measuring 625 µl of 20 µg/ml of Toxaphene stock solution, 625 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard E: Prepared by measuring 312.5 µl of 20 µg/ml of Toxaphene stock solution, 312.5 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard F: Prepared by measuring 62.5 µl of 20 µg/ml of Toxaphene stock solution, 32.5 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 4E. Multi-point Chlordane Calibration Standards (optional)					
Solution	**Stock Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration(µg/l)**
Standard A	Chlordane	20	3750	25	3000
	Surrogate Spiking	0.8	3750		120
Standard B	Chlordane	20	2500	25	2000
	Surrogate Spiking	0.8	2500		80
Standard C	Chlordane	20	1250	25	1000
	Surrogate Spiking	0.8	1250		40
Standard D	Chlordane	20	625	25	500
	Surrogate Spiking	0.8	625		20
Standard E	Chlordane	20	312.5	25	250
	Surrogate Spiking	0.8	312.5		10
Standard F	Chlordane	20	62.5	25	50
	Surrogate Spiking	0.8	62.5		2

Standard A: Prepared by measuring 3750 µl of 20 µg/ml of Chlordane stock solution, 3750 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard B: Prepared by measuring 2500 µl of 20 µg/ml of Chlordane stock solution, 2500 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard C: Prepared by measuring 1000 µl of 20 µg/ml of Chlordane stock solution, 1000 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard D: Prepared by measuring 625 µl of 20 µg/ml of Chlordane stock solution, 625 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard E: Prepared by measuring 312.5 µl of 20 µg/ml of Chlordane stock solution, 312.5 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard F: Prepared by measuring 62.5 µl of 20 µg/ml of Chlordane stock solution, 32.5 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 5. Continuing Calibration Check Solutions					
Checks	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration(µg/l)**
Solution 1	Pesticides Mixture	10	250	50	50
	Surrogates	10			50
Solution 2	Pesticides Mixture	10	50	50	10
	Surrogates	10			10
Solution 3	Pesticides Mixture	10	125	50	25
	Surrogates	10			25

Solution 1: Prepared by measuring 250 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Solution 2: Prepared by measuring 50 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Solution 3: Prepared by measuring 125 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Table 6. DDT and Endrin Breakdown Evaluation Standard	
Stock Solution	**Volume Added (µl)**
Pesticides Performance Evaluation Mixture (10-250 µg/ml)	50
Hexane	49950
Total	50000

DDT and Endrin Breakdown Evaluation Standard (10-250 µg/l): Prepared by measuring 50 µl of Pesticides Performance Evaluation Mixture (10-250 µg/ml) and diluting to 50 ml with hexane.

Table 7
Sample Dilution Table

All dilutions must be made using a 1ml calibrated syringe.

Dilution	*Intact Sample*	*Solvent*
1:2	500ul	500ul
1:5	200ul	800ul
1:10	100ul	900ul
1:20	50ul	950ul
1:25	40ul	960ul
1:50	20ul	980ul

Two Step dilution

Diution	**Step 1**		**Step 2**	
	Intact Sample	*Solvent*	*Sample Aliquot from Step 1*	*Solvent*
1:100	100ul	900ul	100ul	900ul
1:200	100ul	900ul	50ul	950ul
1:250	100ul	900ul	40ul	960ul
1:500	100ul	900ul	20ul	980ul

Three Step Dilution

Diution	**Step 1**		**Step 2**		**Step 3**	
	Intact Sample	*Solvent*	*Sample Aliquot from Step 1*	*Solvent*	*Sample Aliquot from Step 2*	*Solvent*
1:1000	100ul	900ul	100ul	900ul	100ul	900ul
1:2000	100ul	900ul	100ul	900ul	50ul	950ul
1:2500	100ul	900ul	100ul	900ul	40ul	960ul
1:5000	100ul	900ul	100ul	900ul	20ul	980ul

Four Step Dilution

Diution	**Step 1**		**Step 2**		**Step 3**		**Step 4**	
	Intact Sample	*Solvent*	*Sample Aliquot from Step 1*	*Solvent*	*Sample Aliquot from Step 2*	*Solvent*	*Sample Aliquot from Step 3*	*Solvent*
1:10,000	100ul	900ul	100ul	900ul	100ul	900ul	100ul	900ul
1:20,000	100ul	900ul	100ul	900ul	100ul	900ul	50ul	950ul
1:25,000	100ul	900ul	100ul	900ul	100ul	900ul	40ul	960ul
1:50,000	100ul	900ul	100ul	900ul	100ul	900ul	20ul	980ul

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE REVIEW

Date: 8/6/2010 Submitted by: Maria Ruschke

STANDARD OPERATING PROCEDURE NAME & NUMBER:

EGC8081-15 Determination of Organochlorine Pesticides Using GC System

DEPARTMENT: Organics

ANNUAL EVALUATION

NO REVISION: ___ Annual Evaluation

AMEND: ___ Reason for amendment of Standard Operating Procedure

REVISED: _ **X** _ Reason for revision of Standard Operating Procedure

1) Section 9.6.2 – An ICV for toxaphene and chordane must also be analyzed.
2) Section 10.2.1 - Added ICV for toxaphene and chlordane.
3) Table 2 – Adjust times and temps to match current useage.

RETIRE/REMOVE: _ Reason for retirement of Standard Operating Procedure

REINSTATED __ Reason for reinstatement of Standard Operating Procedure

NEW: __ Reason for new Standard Operating Procedure:

MANAGEMENT REVIEW AND APPROVAL

FIELD SERVICE MANAGEMENT NAME:_____ DATE:_____

INORGANIC MANAGEMENT NAME:_____ DATE:_____

X ORGANIC MANAGEMENT NAME: Dave Dervice DATE 8/5/10

LABORATORY MANAGEMENT NAME: _____ DATE:_____

QUALITY ASSURANCE MANAG. NAME: Tracy M. Wiley DATE: 8/6/10

COMMENT: _____

QUALITY ASSURANCE APPROVAL NAME: Maria Ruschke DATE: 8-6-10

Form: QA45-02
Rev. Date 5/1/02

ACCUTEST LABORATORIES
Standard Operating Procedures

Lab Manager
QA Manager

Effective Date: 5/24/10

TEST NAME **SW846 8082: DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs) USING GC SYSTEM**

METHOD REFERENCE SW846 8082 (Revision 0, December 1996), SW846 8000C, EQA044 (Manual Integration)

Revised Sections: ***9.3.1, 13.5.2, 13.5.3, 13.6.2, 13.7.4, 13.8.2, Table 1***

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the analytical procedures, which are utilized by Accutest to acquire samples for analysis of polychlorinated biphenyls (PCBs) as Aroclors, using dual open-tubular, capillary columns with electron capture detectors (ECD).

1.2 This gas chromatographic (GC) method applicable to the determination of the PCB Aroclors listed in Table 1 in extracts from solid and aqueous matrices.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 15 g for solids) is extracted using the appropriate matrix-specific sample extraction technique. Petroleum Products and organic wastes are diluted with an organic solvent and follow SW 846 Method 3580A. Aqueous samples are extracted at neutral pH with methylene chloride using Method 3510 (separatory funnel). Solid samples are extracted with using Method 3545, Pressurized Fluid Extraction.

2.2 Extracts for PCB analysis may be subjected to a sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides.

2.3 After cleanup, the extract is analyzed by injecting a 1 or 2-µL aliquot into a gas chromatograph with dual narrow bore fused silica capillary columns and electron capture detectors (GC/ECD). The chromatographic data may be used to determine the seven Aroclors in Table 1.

2.4 The peaks detected are qualitatively identified by comparison to retention times specific to the known target list of PCBs on two different column types (primary and confirmation).

2.5 Once identified, the Aroclor is quantitated by external standard techniques with an average calibration factor generated from a calibration curve.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. RL's may vary depending on matrix difficulties and sample volumes or weight and percent moisture. Refer to Table 1 for current reporting limits.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. The types of blanks are Method Blank; Instrument Blank, Storage Blank, and Sulfur Blank.

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTINUING CALIBRATION - analytical standard run every 12 hours and at the end of analytical sequence to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the electron capture detector to the target compounds.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is <u>not</u> synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD DETECTION LIMITS (MDLs) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs should be determined approximately once per year for frequently analyzed parameters.

PERCENT DIFFERENCE (%D) - As used in this SOP and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

RELATIVE RESPONSE FACTOR (RRF) - a measure of the instrument response of an analyte. Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RETENTION TIME (RT) - the time required (in minutes) for a standard compound to elute from a chromatographic column.

SURROGATES - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recoveries. Surrogate are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

INSTRUMENT BLANK - a system evaluation sample containing solvent and surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with

plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. 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 data handling sheets must be made available to all personnel involved in these analyses.

5.3 Polychlorinated biphenyls have been classified as known or suspected human or mammalian carcinogens. Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/Mass approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 INTERFERENCES

6.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

6.2 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned. Refer to "The Preparation of Glassware for Extraction of organic contaminants" SOP for practices utilized in the extraction department.

6.3 Interferences may be caused by contaminants that are co-extracted from the sample. The extent of the interferences will vary from source to source, which can be grouped into three broad categories.

6.3.1 Contaminated solvents, reagents, or sample processing hardware.

6.3.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

6.3.3 Compounds extracted from the sample matrix to which the detector will respond.

6.4 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determination.

6.4.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interference from phthalate esters.

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

6.4.3 These materials can be removed through the use of Method 3665 (sulfuric acid/permanganate cleanup).

6.5 Elemental sulfur is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Method 3660 is suggested for removal of sulfur.

6.6 To reduce carryover when high-concentration samples are sequentially analyzed, the syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of an instrument blank to check for cross contamination.

7.0 SAMPLE PRESERVATION AND HOLDING TIME

7.1 PRESERVATION

7.1.1 Water Samples

7.1.1.1 Collect samples in 1 liter glass amber bottles without preservatives.

7.1.1.2 A liter of an unpreserved sample is required for extraction. Additional sample volume is necessary for any samples used for matrix spike and matrix spike duplicates. Therefore, 3 liters of at least one sample in every group of 20 field samples are required for analysis to accommodate all quality control requirements.

7.1.2 Soil Samples

7.1.2.1 Samples are collected in a 300-ml amber glass sample bottle. No preservative is required.

7.1.3 Sample should be taken with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing possible phthalate contamination.

7.1.4 The samples must be protected from light and refrigerated at 4 °C (± 2 °C) from the time of receipt until extraction and analysis.

7.2 HOLDING TIME

7.2.1 Aqueous sample must be extracted within 7 days of sampling.

7.2.2 Soil sample must be extracted within 14 days of sampling.

7.2.3 Extracts must be analyzed within 40 days following extraction.

8.0 APPARATUS AND MATERIALS

8.1 GAS CHROMATOGRAPH SYSTEM

8.1.1 Gas Chromatograph-Agilent or Hewlett Packard Model 5890 and 6890. The analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for splitless injection with capillary columns. The capillary columns are directly coupled to the detectors.

8.1.2 Columns

8.1.2.1 Column pair 1

8.1.2.1.1 30 m x 0.32 mm fused silica (0.5 µm film thickness) DB-1701 narrow-bore capillary column or equivalent.

8.1.2.1.2 30 m x 0.32 mm fused silica (0,5 µm film thickness) DB-5 narrow-bore capillary column or equivalent.

8.1.2.2 Column pair 2

8.1.2.2.1 30 m x 0.32 mm fused silica (0.5 µm film thickness) RTX CLPI narrow-bore capillary column or equivalent.

8.1.2.2.2 30 m x 0.32 mm fused silica (0.25 µm film thickness) RTX CLPII narrow-bore capillary column or equivalent.

8.1.3 Detectors

8.1.3.1 Electron Capture Detectors (HP).

8.1.3.2 Micro Electron Capture Detectors (HP).

8.2 AUTOSAMPLER

8.2.1 Agilent or Hewlett Packard Models: 7673A, 7683 7 7643A ,capable of holding 100 of 2-ml crimp vials.

8.3 DATA SYSTEM

8.3.1 MSD interfaced to the gas chromatograph which allows the continuous acquisition and storage on machine readable media (disc) of all chromatographic data obtained throughout the duration of the analysis.

8.3.2 The ENVIROQUANT data system is capable of quantitation using multi-point calibration.

8.3.3 Lagato Networker with lookup database on 4mm DAT tape for long term, off line magnetic storage of data.

8.4 SYRINGE

8.4.1 Manually held ul-syringes, various volumes (Hamilton or equiv.).

8.4.2 10 µl graduated, auto sampler (Hamilton or equiv.).

9.0 REAGENTS AND STANDARDS

9.1 Refer to Accutest Sample Preparation SOPs EOP001 and EOP040 for reagents and standards used for sample extraction.

9.2 Solvents - Ultra pure, chromatography grade Hexane.

9.3 Stock standard solutions.

9.3.1 Two separate sources of commercially prepared standards with traceability documentation are used. The standards contain Aroclors 1016, 1221, 1232, 1242, 1248, 1254 and 1260 1262 and 1268.

9.4 Working Solutions

9.4.1 Prepare working solutions, using stock solution, in hexane, as needed, that contain the compounds of interest, either singly or mixed together. Refer to Table 3A, 3B for details.

9.5 Calibration Standards

9.5.1 Initial Calibration Standards

9.5.1.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks in the other five Aroclor mixtures. As a result, a multi-point calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibration for each of the seven Aroclors. Prepare a minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260, including surrogates, by dilution of the above working solutions (Section 9.4) with hexane. Suggested levels and preparations are shown in Table 4A.

9.5.1.2 Separate calibration standards are required for the other five Aroclors. Unless otherwise necessary for a specific project, a single calibration standard near the mid-point of the expected calibration range of each remaining Aroclor is employed to determine its calibration factor. Refer to Table 4B for preparation scheme. Optional curves as shown on Table 4C may also be used for a multi-point calibration per project's specification.

9.5.2 Continuing Calibration Verification (CCV)

9.5.2.1 For Aroclor analyses, the continuing calibration checks should be a mixture of Aroclor 1016 and Aroclor 1260. Two standards at 500 µg/l and 1,000 µg/l are prepared as described in Table 5A. During the analysis, these two solutions are alternated to check the initial calibration.

9.5.2.2 In situations where only a few Aroclors are of interest for a specific project, the calibration checks of each Aroclor of interest may be prepared (Table 5B) and analyzed as the 1016/1260 mixture throughout the analytical sequence.

9.6 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

9.6.1 Prepare the ICV check standards from separate sources of stock standards from the calibration curve following the procedures in Table 6A, and 6B.

9.6.2 The ICV is prepared at 1,000 µg/l for each Aroclor and is analyzed immediately after and initial calibration.

9.7 Surrogates

9.7.1 Tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are used as surrogate standards for this method.

9.7.2 A calibration range must be constructed for the surrogate compounds. Accordingly, appropriate amounts of surrogates are mixed with each calibration solution to define a range similar to the target compounds.

9.7.3 Surrogate compounds are also contained in continuing calibration checks, and second source calibration check standard.

9.7.4 Spike each sample, QC sample and blank with an appropriate amount of corresponding surrogate spiking solution, prior to extraction, for a final concentration in the extract of 40 µg/l of each surrogate compound.

9.8 Storage of Standards

9.8.1 Store unopened stock standard solutions according to the manufacturer's documented holding time and storage temperature recommendations. Protect from light.

9.8.2 Store all other working standard solutions in glass vials with Teflon lined screw caps at 4°C (± 2°C) in the dark.

9.8.3 Opened stock standard solutions must be replaced after 6 months or sooner if manufacturer's expiration date comes first or comparison with quality control check samples indicates a problem.

9.8.4 All other standards must be replaced after six months or sooner if routine QC indicates a problem or manufacturer's expiration date comes first.

10.0 CALIBRATION

10.1 Initial Calibration

10.1.1 The method reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Lower concentration standard may be needed to meet the reporting limit requirements of state specific regulatory program. The linear range covered by this calibration is the highest concentration standard.

10.1.2 The initial calibration for this method consists of two parts, described below.

10.1.2.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detectors and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. The calibration range covered for Aroclor 1016 and Aroclor 1260 employs standards of 50, 250, 500, 1,000, 2,000, and 3,000 µg/l.

10.1.2.2 Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixtures in Section 10.1.2.1 has been used to describe the detector response. The concentration of each Aroclor standard is near the mid-point of the linear range of the detector, usually at 1,000 µg/l. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of those 1016/1260 standards.

10.1.2.3 In situations where only a few Aroclors are of interest for a specific project, an initial calibration of a minimum of five standards of each Aroclors of interest instead of the 1016/1260 mixture may be performed.

10.1.3 A calibration range must be constructed for each surrogate compound. Accordingly, add appropriate amounts of each surrogate compound to the calibration solution to define a range similar to the target compounds.

10.1.4 Aliquot proper amount of each calibration standard into a 2 ml crimp top vial.

10.1.5 PCBs are quantitatively determined as Aroclors by the external standard technique. The Calibration Factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards is calculated using the equation in Section 14.1.

10.1.5.1 Use at least five peaks for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors. At least five sets of calibration factors will be generated, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture.

10.1.5.2 A minimum of 3 characteristic peaks must be chosen for each of the other Aroclors, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Thus, each single standard will generate at least three calibration factors, one for each selected peak.

10.1.5.3 Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 6 peaks should include at least one peak that is unique to that Aroclor.

10.1.5.4 The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. When the Aroclor 1016/1260 mixture is used

to demonstrate the detector response, the calibration model chosen for this mixture must be applied to the other five Aroclors for which only single standards are analyzed. If multi-point calibration is performed for individual Aroclors, use the calibration factors from those standards to evaluate linearity.

10.1.6 For the initial calibration to be valid, the percent relative standard deviation (% RSD) (see Section 14.2) must be less than 20 % for each Aroclor of interest on each column. If any analyte exceeds the 20% acceptance limit for a given calibration, corrective action must be taken.

10.1.6.1 If the problem is associated with specific standards, reanalyze the standard and recalculate the RSD.

10.1.6.2 Alternatively, narrow the calibration range by replacing one or more of the calibration standards that cover a narrow range.

10.1.6.2.1 The changes to the upper end of the calibration range will affect the need to dilute samples above the range. If the instrument response indicates signs of detector saturation, the concentration of the standard at the upper limit will be reduced. The changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

10.2 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

10.2.1 The initial calibration is verified with an ICV, a second source calibration check standard from an external source (Section 9.6). It must be performed right after the initial calibration.

10.2.2 The percent difference (%D) (Section 14.3) for this standard must meet the %D criteria of 15% used for calibration verification on each column.

10.2.2.1 If %D is greater than 15%, reanalyze the second source check. If the limit cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

10.2.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, prepare a third source and repeat the process. Make fresh calibration standards using one of the two standard sources that matches each other.

10.3 Continuing Calibration Verification (CCV)

10.3.1 Continuing calibration verificatiion (CCV) standards (Section 9.5.2) must be acquired at the beginning of each 12-hour shift, after every 10 injections not to exceed 12 hours and at the end of the analysis sequence. The 500 µg/l check standard is alternated with 1,000 µg/l standard for calibration verification. Each CCV, including the end check, must be followed by the analysis of an Instrument Blank.

10.3.2 For Aroclor analyses, the calibration verification standard should be a mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclors after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

10.3.3 The percent difference (%D) (see section 14.3) must be less than 15 % for each Aroclor of interest on each column.

10.3.4 Each sample analysis must be bracketed by periodic analyses of acceptable calibration verification standards every 10 injections not to exceed 12 hours. If %D criteria fails during a mid sequence calibration check or at the end of the analysis sequence, a continuing calibration check is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed. In situations where the first check fails to meet the criteria, the instrument logbook should have clear documented notations as to what the problem was and what corrective action was implemented to enable the second check to pass.

10.3.5 A continuing calibration standard is analyzed whenever the analyst suspects that the analytical system is out of calibration. If the calibration cannot be verified, corrective action is performed to bring the system into control. Analysis may not continue until the system is under control.

10.3.6 When a calibration verification standard fails to meet the QC criteria at the end of the analysis sequence, all samples injected after the last standard that met the QC criteria must be evaluated to prevent mis-quantitations, and re-injection of the sample extracts may be required.

10.3.6.1 The analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed when the calibration standard response is above the initial calibration response.

10.3.6.2 Whether the analyte was detected in the specific samples analyzed during the analytical shift, or the calibration standard response is below the initial calibration response, then the extracts for those samples need to be reanalyzed.

10.3.7 Each subsequent injection of a continuing calibration standard during the 12-hour analytical shift must be checked against the retention time windows established in Section 11.0. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

11.0 RETENTION TIME WINDOWS

11.1 Absolute retention times are used for the identification of PCBs as Aroclors. Retention time windows must be calculated for each surrogate and at least 3 to 5 characteristic peaks of each Aroclor on each GC column, when a new initial calibration is run and whenever a new chromatographic column is installed, or when there are significant changes in the operating conditions. The retention time windows must be reported with the analysis results in support of the identifications made.

11.2 Employ the following approach to establish retention time windows:

11.2.1 Make three injections of each Aroclor at approximately equal intervals during the 72-hr period.

11.2.2 For each Aroclor, choose three or five major peaks and calculate the mean and standard deviation of the three retention times for that peak. The peak chosen should be fairly immune to losses due to degradation and weathering in the samples. Record the retention time to three decimal places (e.g. 10.015 min) for each Aroclor.

11.2.3 In those cases where the standard deviations of the retention time window for a particular Aroclor is 0.01 minutes or less, the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

11.2.4 Apply plus or minus three times the standard deviations to retention time of each Aroclor standard (continuing calibration or middle level of initial calibration). This will be used to define the retention time window for the sample.

11.2.4.1 If default standard deviation of 0.01 minutes is employed, the width of the window will be 0.03 minutes.

11.2.5 Establish the center of the retention time window for each Aroclor and surrogate by using the absolute retention time for each Aroclor and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

12.0 PROCEDURE

12.1 Sample Extraction

12.1.1 In general, water samples are extracted at a neutral pH with methylene chloride using a separate funnel (Method 3510) (Refer to SOP: EOP001). Solid samples are extracted using Method 3545, Pressurized Fluid Extraction (Refer to SOP: EOP040).

12.2 Sample Cleanup

12.2.1 Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. Refer to appropriate SOPs for details.

12.2.1.1 Interferences by phthalate esters can be removed through the use of a sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for PCBs. This method should be used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs.

12.2.1.2 Element sulfur, which may be present in certain sediments and industrial wastes, interfere with the electron capture gas chromatography of certain Aroclors. Sulfur should be removed by the technique described in Method 3660.

12.3 Instrument conditions.

12.3.1 Recommended instrument conditions are listed in Table 2. Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification should be approved by team leader/manager.

12.4 Initial calibration

12.4.1 Refer to Section 10.1.

12.5 Initial calibration Verification (ICV) -Second source calibration check standard

12.5.1 Refer to Section 10.2.

12.6 Continuing calibration Verifications (CCV)

12.6.1 Refer to Section 10.3.

12.7 Sample analysis (Primary)

12.7.1 All samples and quality control samples are injected into the Gas Chromatograph using the autosampler. Program the sampler for an appropriate number of syringe rinses and a 1ul or 2 µl injection size. A splitless injection technology is used.

12.7.2 Sample concentrations are calculated by comparing sample responses with the initial calibration of the system (Section 14.4). If sample response exceeds the limits of the initial calibration range, dilute the extract and reanalyze. Extracts should be diluted so that all peaks are on scale, as overlapping peaks are not always evident when peaks are off scale.

12.7.3 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the sample meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

12.7.4 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

12.7.5 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix.

12.8 Confirmation analysis.

12.8.1 Confirmation analysis is to confirm the presence of Aroclors tentatively identified in the primary analysis.

12.8.1.1 All instrument performance quality control criteria for calibration and retention time must be satisfied on the confirmation analysis.

12.8.2 Each tentative identification must be confirmed: using a second GC column of dissimilar stationary phase (as in the dual-column analysis), based on a clearly identifiable Aroclor pattern, or using another technique such as GC/MS.

12.8.2.1 The primary and secondary analysis is conducted simultaneously in the dual-column analysis.

12.8.2.2 GC/MS confirmation may be used in conjunction with dual-column analysis if the concentration is sufficient for detection in GC/MS, normally a concentration of approximately 10 ng/µl in the final extract for each Aroclor is required. Method 8270 is recommended as a confirmation technique when sensitivity permits.

12.8.3 Once the identification has been confirmed, the agreement between the quantitative results on both columns should be checked.

12.9 Sample Dilution

12.9.1 Establish dilution of sample in order to fall within calibration range or to minimize the matrix interference.

- Utilize screen data (specific project only).
- Utilize acquired sample data.
- Utilize the history program or approval from client/project.
- Sample characteristics (appearance, odor).

12.9.2 If no lower dilution has been reported, the dilution factor chosen should keep the response of the largest peak for a target analyte in the upper half of the initial calibration range of the instrument.

12.9.3 Preparing Dilutions.

12.9.3.1 Prepare sample dilutions quantitatively. Dilute a stored sample extract with hexane using logical volume to volume ratios, i.e., 1:5, 1:10, 1:50, etc.

12.9.3.2 Syringe Dilutions - A calibrated 1ml syringe must be used to prepare dilutions. Gently shake to disperse the extract throughout the solvent prior to loading on the auto-sampler tray for further analysis.

12.9.3.3 Volumetric Flask Dilutions – Dilutions can also be made with a Class A volumetric flask. Measure appropriate sample extract volume in a calibrated syringe and bring to a final volume with dilution solvent in a Class A volumetric flask. Gently shake to disperse the extract throughout the solvent prior to loading on the auto-sampler tray for further analysis.

12.10 Data interpretation

12.10.1 Qualitative identification

12.10.1.1 Analyst shall identify the target analytes with competent knowledge interpreting retention time and/or chromatographic pattern by comparison of the sample to the standard of the suspected Aroclor. The criteria required for a positive identification are:

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

12.10.1.1.2 The target analytes must elute within the daily absolute retention time window on both primary and confirmation column.

12.10.1.1.3 For PCBs, at least five major peaks are selected. The retention time window for each peak is determined from the initial calibration analysis. This identification of PCBs as Aroclors is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of multi-component target analytes. Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte.

12.10.1.1.4 Be aware of matrix interfering effects on peak shape and relative peak ratios which could distort the pattern. Interpretation of this phenomenon may require a highly experienced chromatographer or at least a second opinion.

12.10.2 Quantitative analysis

12.10.2.1 Once the Aroclor pattern has been identified, compare the responses of at least 3 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each corresponding peak and the linear calibration established from the multi-point calibration of the 1016/1260 mixture. A concentration (see section 14.4) based on the

integrated area/or height of each of the characteristic peaks is determined and then those resulting concentrations are averaged to provide the final result for the sample.

12.10.2.2 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. The quantitation may then be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times should be subtracted from the total area. When quantitation is performed in this manner, the problems should be fully described for the data user and the specific procedures employed by the analyst should be thoroughly documented.

12.10.2.3 When sample results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results must be evaluated after the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula in Section 14.6. The lower result is reported.

12.10.2.3.1 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

12.10.2.3.2 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the lower result with the footnote (remark) indicating "More than 40% RPD for detected concentrations between two GC columns".

13.0 QUALITY CONTROL

13.1 QC Requirements Summary

Initial Calibration	Whenever needed
Initial Calibration Verification (ICV)	Following initial calibration
Continuing Calibration Verifications (CCV)	Every 12-hour shift , after every 10 samples and at the end of analysis sequence
Method blank	One per extraction batch*
Instrument Blank	After every CCV and end calibration check
Blank Spike	one per extraction batch*
Matrix Spike	one per extraction batch*
Matrix Spike Duplicate	one per extraction batch*
Surrogates	every sample and standard

*The maximum number of samples per batch is twenty or per project specification.

13.2 Initial Calibration.

13.2.1 Refer to Section 10.1.

13.3 Initial Calibration Verification (ICV) -Second Source Calibration Check.

13.3.1 Refer to Section 10.2.

13.4 Continuing Calibration Verifications (CCV)

13.4.1 Refer to Section 10.3.

13.5 Method Blank.

13.5.1 The method blank is either DI water or sodium sulfate (depending upon the sample matrix) which must be extracted with each set of 20 or less samples. For a running batch, a new method blank is required for each different extraction day. The method blank should be carried through all stages of the sample preparation and measurement.

13.5.2 If the method blank contains a target analyte above its MDL In LIMS, the entire batch must be re-extracted and reanalyzed.

13.5.3 Surrogate compounds are added to the method blank prior to extraction and analysis. If the surrogate accuracy in the blank does not meet criteria in LIMS, the entire batch must be re-extracted and reanalyzed.

13.6 Blank Spike.

13.6.1 A blank spike must be extracted with each set of 20 or less samples. For a running batch, a new blank spike is required for each different day. The blank spike consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. It is spiked with the same analyte at the same concentration as matrix spike. When the presence of specific Aroclors is not anticipated, the Aroclor 1016/1260 mixture may be appropriate choice for spiking. In situations where the other Aroclors are of interest for a specific project, the analyst may employ different spiking mixtures. The blank spike is prepared at a concentration of 2 µg/l or 66.7 µg/kg (on a dry weight basis) for each Aroclor.

13.6.2 The blank spike recoveries should be assessed using in house limits specified in LIMS.

13.6.3 If a blank spike is out of control, the following corrective actions must be taken. In the case where the blank spike recovery is high and no hits reported in associated samples and QC batch the sample results can be reported with footnote (remark) and no further action is required.

13.6.3.1 Check to be sure that there are no errors in the calculations, or spike solutions. If errors are found, recalculate the data accordingly.

13.6.3.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample batch.

13.6.3.3 If no problem is found, re-extract and reanalyze the sample batch.

13.7 Matrix Spike (MS) / Matrix Spike Duplicate (MSD).

13.7.1 One sample is randomly selected from each extraction batch and spiked in duplicate with select Aroclors to assess the performance of the method as applied to a particular matrix and to provide information on the homogeneity of the matrix. Both the MS and MSD are carried through the complete sample preparation, cleanup, and determinative procedures.

13.7.2 The MS and MSD should be spiked with the Aroclors of interest. If samples are not expected to contain target analytes, a matrix spike and matrix spike duplicate pair should be spiked with Aroclor 1016/1260 mixture. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclor should be used for spiking.

13.7.3 Matrix spikes are prepared by spiking an actual sample at a concentration 2 µg/l or 66.7µg/kg on a dry weight basis.

13.7.4 Assess the matrix spike recoveries and relative percent difference (RPD) against the control limits in LIMS.

13.7.5 If the matrix spike accuracy of any individual Aroclor is out of control, the accuracy for that Aroclor in the blank spike must be within control. Matrix interference is assumed and the data is reportable. No further corrective action is required.

13.8 Surrogates.

13.8.1 Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogate standards. All blanks, samples, matrix spikes, and calibration standards contain surrogate compounds which are used to monitor performance of the extraction, cleanup (when used), and analytical system.

13.8.2 The recoveries (Section 14.5) of the surrogates must be evaluated versus the surrogate control limits in LIMS developed by the laboratory annually.

13.8.3 If surrogate recoveries are not within established control limits, corrective action must be performed if surrogate recoveries indicate that a procedural error may have occurred during the analysis of the sample.

13.8.3.1 Check the surrogate calculations for calculation or integration errors and perform corrections if detected.

13.8.3.2 Reanalyze the extract if no calculation errors are detected. If the surrogate recoveries for the reanalyzed extract are in control, report the data from the reanalysis only.

13.8.3.3 If the data from the reanalysis is also out of control, re-extract and reanalyze the sample.

13.8.3.4 If, upon reanalysis, the surrogate recoveries are acceptable, report the reanalysis data. If the holding time has expired prior to the reanalysis, report both the original and reanalysis results and note the holding time problem.

13.8.3.5 If the recovery is again not within limits, the problem is considered to be matrix interference. Submit both data sets with the original analysis being reported.

13.8.4 The retention time shift for surrogate must be evaluated after the analysis of each sample. The sample must be reanalyzed when the retention times for both surrogates are outside the retention time window.

13.8.4.1 Reanalyses are not required for samples having visible matrix interference, defined as excessive signal levels from target or non-target interfering peaks. This judgment should be approved by a team leader or supervisor.

14.0 CALCULATION

14.1 Calibration Factor (CF).

$$CF = \frac{A_s}{C_s}$$

where:
A_s = Area of the peak for the compound being measured.
C_s = Concentration of the compound being measured (µg/l).

14.2 Percent Relative Standard Deviation (% RSD).

$$\%RSD = \frac{SD}{CF_{av}} \times 100$$

where:
SD = Standard Deviation.
CF_{av} = Average calibration factor from initial calibration.

14.3 Percent Difference (% D).

$$\% D = \frac{|CF_{av} - CF_c|}{CF_{av}} \times 100$$

where:
CF_c = CF from continuing calibration (CBCHK).

14.4 Concentration (Conc.).

For water:

$$\text{Conc. } (\mu g/l) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{V_i}$$

For soil/sediment (on a dry weight basis):

$$\text{Conc. } (\mu g/kg) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{W_s \times S}$$

where:
A_c = Area of peak for compound being measured.
V_f = Final Volume of total extract (ml).
D = Secondary dilution factor.
V_i = Initial volume of water extracted (ml).
W_s = Weight of sample extracted (g).
S = (100 - % moisture in sample) / 100 or % solid/100.
M = Multiplier.

14.5 Percent Recovery (% R).

$$\%\ R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

14.6 Relative Percent Difference (RPD).

$$RPD = \frac{|C_1 - C_2|}{(1/2)(C_1 + C_2)} \times 100$$

where:
C_1 = Matrix Spike Concentration or the result on column 1.
C_2 = Matrix Spike Duplicate Concentration or the result on column 2.

15.0 DOCUMENTATION

15.1 The Analytical Logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

15.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in this log. For consistency, if surrogates are high or low indicate it as (↑) for high and (↓) for low.

15.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed, the page must be signed and dated by the respective person.

15.2.1 The Accutest Lot Number must be cross-reference on the standard vial.

15.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

15.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

15.5 Unused blocks of any form must be x'ed and z'ed by the analyst before submitting the data for review.

15.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

16.0 DATA REVIEW AND REPORTING

16.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

16.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

16.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action before proceeding with analysis.

16.2.1 In some situation, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

16.3 Chromatogram Review. The chromatogram of each sample is evaluated for target analytes.

16.3.1 Check specific retention time windows for each target compound for the presence of the target compound in each chromatogram.

16.3.1.1 Each sample may require the reporting of different target analytes. Review the login to assure that the correct target compounds are identified.

16.3.2 The Aroclor must be identified on the primary and confirmatory column before assigning a qualitative identification.

16.3.3 Manual integration of chromatographic peaks must be identified by the analysts. An electronic signature is applied upon data review.

16.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

17.0 POLLUTION PREVENTION & WASTE MANAGEMENT

17.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 17.2.

17.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

17.2.1 Non hazardous aqueous wastes.

17.2.2 Hazardous aqueous wastes

17.2.3 Chlorinated organic solvents

17.2.4 Non-chlorinated organic solvents

17.2.5 Hazardous solid wastes

17.2.6 Non-hazardous solid wastes

Table 1. PCB Aroclors and Reporting Limits				
Compound	**CAS Number**	**Water (µg/l)**	**Soil (µg/kg)**	**Oil (µg/kg)**
Arochlor – 1016	12674-11-2	0.5	30	2000
Arochlor – 1221	11104-28-2	0.5	30	2000
Arochlor – 1232	11141-16-5	0.5	30	2000
Arochlor – 1242	53469-21-9	0.5	30	2000
Arochlor – 1248	12672-29-6	0.5	30	2000
Arochlor – 1254	11097-69-1	0.5	30	2000
Arochlor – 1260	11096-82-5	0.5	30	2000
Arochlor - 1262	37324-23-5	0.5	30	2000
Arochlor - 1268	11100-14-4	0.5	30	2000

Table 2. RECOMMENDED OPERATING CONDITION	
Gas Chromatograph/Electron Capture Detectors	
Carrier Gas	Helium
Make-up gas	5 % Methane/ 95 % Argon
Make-up gas flow	*30 ml/min
Injection port temperature	*235 °C
Injection type	Splitless
Detector temperature	*320 °C
Column flow	*5 ml/min
Gas Chromatograph Temperature Program*	
Initial temperature	*170 °C
Time 1	*2 min
Column temperature rate 1	*30 degrees/min
Temperature 1	*180 °C
Column temperature rate 2	*3.5 degrees/min
Temperature 2	*240 °C
Column temperature rate 3	*10 degrees/min
Final temperature	*280 °C
Time 3	*5 min
Total run time	30-40 min

* Parameter modification allowed for performance optimization as long as QC criteria are achieved.

Table 3A. Aroclors 1016/1260 Mixture and Surrogates Working Solution	
Stock Solution	**Volume Added**
Aroclor 1016 /1260(1,000 µg/ml)	500 µl
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	100 µl
Hexane	fill to volume
Total	25.0 ml

- Aroclors 1016/1260 (20 µg/ml) and Surrogates (0.8 µg/ml) Working Solution: Prepared by measuring 500 µl of 1,000 µg/ml Aroclor 1016 / 1260 and 100 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 3B. Individual Aroclor* and Surrogates Working Solution	
Stock Solution	**Volume Added**
Individual Aroclor* (1,000 µg/ml)	500 µl
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	100 µl
Hexane	24.4 ml
Total	25 ml

*Aroclor: 1221, 1232, 1242, 1248, 1254, 1262 & 1268

- Individual Aroclor (20 µg/ml) and Surrogates (0.8 µg/ml) Working Solution: Prepared by measuring 500 µl of 1,000 µg/ml each individual Aroclor, 100 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 10 ml with hexane.

Table 4A. Aroclors 1016/1260 Calibration Standard Solutions					
Standard	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration(µg/l)**
Standard A	Aroclors 1016/1260	20	62.5	25	50
	Surrogates	0.8			2
Standard B	Aroclors 1016/1260	20	312.5	25	250
	Surrogates	0.8			10
Standard C	Aroclors 1016/1260	20	625	25	500
	Surrogates	0.8			20
Standard D	Aroclors 1016/1260	20	1250	25	1,000
	Surrogates	0.8			40
Standard E	Aroclors 1016/1260	20	2,500	25	2,000
	Surrogates	0.8			80
Standard F	Aroclors 1016/1260	20	3,750	25	3,000
	Surrogates	0.8			120

- Standard A: Prepared by measuring 62.5 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.
- Standard B: Prepared by measuring 312.5 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.
- Standard C: Prepared by measuring 625 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.
- Standard D: Prepared by measuring 1,250 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.
- Standard E: Prepared by measuring 2,500 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.
- Standard F: Prepared by measuring 3,750 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

Table 4B. Single-Point Calibration Standard (1,000 µg/l) for Individual Aroclor*		
	Stock Solution	**Volume Added**
Individual Aroclor*/Surrogate Working Solution (20 µg/ml/0.80µg/ml) (Table 3B)		1,250 µl
Hexane		23.75 ml
Total		25 ml

* Aroclor: 1221, 1232, 1242, 1248, 1254, 1262 & 1268

- Individual Aroclor Calibration Standard (1,000 µg/l) and Surrogates (40 µg/l) Solution: Prepared by measuring 1,250 µl of individual Aroclor and surrogates working solution, containing 20 µg/ml of each corresponding Aroclor and 0.80 µg/ml of both surrogate compounds, and bringing to 25 ml with hexane.

Table 4C. Multi-point Calibration Standards for Individual Aroclor* (optional)					
Standard	**Stock Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration(µg/l)**
Standard A	Aroclor*	20	62.5	25	50
	Surrogates	0.8			2
Standard B	Aroclor*	20	312.5	25	250
	Surrogates	0.8			10
Standard C	Aroclor*	20	625	25	500
	Surrogates	0.8			20
Standard D	Aroclor*	20	1250	25	1,000
	Surrogates	0.8			40
Standard E	Aroclor*	20	2,500	25	2,000
	Surrogates	0.8			80
Standard F	Aroclor*	20	3,750	25	3,000
	Surrogates	0.8			120

*Aroclor: 1221, 1232, 1242, 1248, 1254, 1262 & 1268

- Standard A: Prepared by measuring 62.5 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.
- Standard B: Prepared by measuring 312.5 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.
- Standard C: Prepared by measuring 625 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.
- Standard D: Prepared by measuring 1,250 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.
- Standard E: Prepared by measuring 2,500 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.
- Standard F: Prepared by measuring 3,750 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

Table 5A. Continuing Calibration Check Solutions for Aroclors 1016/1260					
Checks	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration (µg/l)**
Solution 1	Aroclors 1016/1260	20	625	25	500
	Surrogates	0.8			20
Solution 2	Aroclors 1016/1260	20	1250	25	1,000
	Surrogates	0.8			40

- Solution 1: Prepared by measuring 625 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

- Solution 2: Prepared by measuring 1,250 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

Table 5B. Continuing Calibration Check Solutions for Individual Aroclor*					
Checks	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration (µg/l)**
Solution 1	Aroclor*	20	625	25	500
	Surrogates	0.8			20
Solution 2	Aroclor*	20	1250	25	1,000
	Surrogates	0.8			40

* Aroclor: 1221, 1232, 1242, 1248, 1254, 1262 & 1268

- Solution 1: Prepared by measuring 625 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

- Solution 2: Prepared by measuring 1,250 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

Table 6A. Second Source Calibration Check Standard for Aroclors 1016/1260 (1,000 µg/l)	
Stock Solution	**Volume Added**
Aroclors 1016/1260 (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution	1,000 µl
Hexane	24 ml
Total	25 ml

- Aroclors 1016/1260 (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution: Prepared by measuring 250 µl of 1,000 µg/ml Aroclors 1016/1260 mix solution (2nd source), 125 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 10 ml with hexane.

- Aroclors 1016/1260 (1,000 µg/l) and Surrogates (100 µg/l) Solution: Prepared by measuring 1,000 µl of Aroclors 1016/1260 (25 µg/ml) and surrogates (2.5 µg/ml) working solution and bringing to 25 ml with hexane.

Table 6B. Second Source Calibration Check Standard for Individual Aroclor* (1,000 µg/l)	
Stock Solution	**Volume Added**
Individual Aroclor* (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution	1,000 µl
Hexane	24 ml
Total	25 ml

*Aroclor: 1221, 1232, 1242, 1248, 1254, 1262 & 1268

- Individual Aroclor (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution: Prepared by measuring 250 µl of 1,000 µg/ml each individual Aroclor stock solution (2nd source), 125 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 10 ml with hexane.

- Individual Aroclor (1,000 µg/l) and Surrogates (100 µg/l) Solution: Prepared by measuring 1,000 µl of each individual Aroclor (25 µg/ml) and surrogates (2.5 µg/ml) working solution and bringing to 25 ml with hexane.