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FINAL SAMPLING AND ANALYSIS PLAN (FIELD SAMPLING PLAN AND QUALITY
ASSURANCE PROJECT PLAN OPERABLE UNIT 1 (OU1) SITE 83 SOIL DELINEATION
SAMPLING MCAS CHERRY POINT NC
7/1/2009
RHEA ENGINEERS & CONSULTANTS, INC

FINAL

**SAMPLING AND ANALYSIS PLAN
(FIELD SAMPLING PLAN AND
QUALITY ASSURANCE PROJECT PLAN)**

**OPERABLE UNIT 1, SITE 83
SOIL DELINEATION SAMPLING
MCAS CHERRY POINT, NORTH CAROLINA**



**CONTRACT NO. N40085-08-D-1409
CTO: 0002**

RHĒA PROJECT NO. 389

JULY 2009

PREPARED FOR:



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SAP Worksheet #1 -- Title and Approval Page

**FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
Operable Unit 1, Site 83 Soil Delineation Sampling
Marine Corps Air Station Cherry Point, North Carolina**

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N40085-08-D-1409
CTO: 0002

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EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) provides the procedures and requirements to be implemented for collecting the proposed soil samples at Operable Unit 1 (OU1) Site 83 at Marine Corps Air Station (MCAS) Cherry Point, North Carolina, and was prepared in accordance with the requirements of the Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP) (United States Environmental Protection Agency [USEPA 2005]) and USEPA Guidance for Quality Assurance Project Plans, USEPA QA/G-5, QAMS (USEPA 2002). The Navy (Naval Facilities Engineering Command [NAVFAC] Mid-Atlantic Division) and MCAS Cherry Point Environmental Affairs Department [EAD]) is conducting this sampling under Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). USEPA Region 4 is the federal regulatory agency and North Carolina Department of Environment and Natural Resources (NCDENR) is the state regulatory agency.

The objective of the soil sampling is to confirm residual contamination of polycyclic aromatic hydrocarbons (PAHs), pesticides, and lead at the site, characterize portions of the site where historical information is limited or suspect, and delineate the vertical and horizontal extent of impacted site soils. This information will be incorporated into future site documents and will be used to develop feasible remedial alternatives. These investigative soil samples will be utilized as pre-confirmatory samples in the event that an excavation remedial alternative is selected.

Soil samples will be collected from three areas (i.e., Areas A, B, and C) at Site 83, including the former area of Building 96 and the adjacent lot, the area west of Building 96, and the area southwest of Building 96. Samples will be collected at a minimum of 29 locations and analyzed for specific PAHs and pesticides. A select grouping of samples will also be analyzed for lead.

Environmental Chemistry Consulting Services, Inc. (ECCS), a National Environmental Laboratory Accreditation Conference (NELAC) and NCDENR certified mobile laboratory, will provide analytical services for this project. TestAmerica Laboratories, Inc. (TestAmerica) will provide fix-based analytical services for lead analysis.

This SAP serves to guide the sampling effort so that the analytical data generated from the soil sampling will be of the quantity and quality necessary to provide technically sound and defensible assessment of the vertical and lateral extent of the Site 83 soil contamination.

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Acronyms

CA	Corrective Action
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
COC	Chain of Custody
CTO	Contract Task Order
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethylene
DDT	dichlorodiphenyltrichloroethane
DQI	Data Quality Indicator
EAD	Environmental Affairs Department
ECCS	Environmental Chemistry Consulting Services, Inc.
ECD	Electron Capture Detector
EPA	Environmental Protection Agency
FS	Feasibility Study
GC-MS	Gas Chromatograph-Mass Spectrometer
GPS	Global Positioning System
HAZWOPER	Hazardous Waste Operations and Emergency Response
ICP	Inductively Coupled Plasma
LCS	Laboratory Control Sample
LFB	Laboratory Fortified Blank
MCAS	Marine Corps Air Station
MDL	Method Detection Limit
MPC	Measurement Performance Criteria
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NAVFAC	Naval Facilities Engineering Command
NCDENR	North Carolina Department of Environment and Natural Resources
NELAC	National Environmental Laboratory Accreditation Conference
NIRIS	Naval Installation Restoration Information Solution
OU1	Operable Unit 1
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PQO	Project Quality Objective
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RI	Remedial Investigation
ROD	Record of Decision
RPD	Relative Percent Difference

RPM	Remedial Project Manager
RSD	Relative Standard Deviation
RSL	Regional Screening Levels
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
TBD	To Be Determined
UFP	Uniform Federal Policy
USEPA	United States Environmental Protection Agency
VOA	Volatile Organic Analyte
VOC	Volatile Organic Compound

SAP Worksheet #2 -- SAP Identifying Information

Site Name/Number: Site 83

Operable Unit: OU1

Contractor Name: Rhēa Engineers & Consultants, Inc.

Contract Number: N40085-08-D-1409, CTO 0002

Contract Title: Environmental Remediation Services

1. This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (USEPA 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (USEPA 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
<u>Partnering Meeting</u>	<u>November 2008</u>
<u>Partnering Meeting</u>	<u>February 2009</u>
<u> </u>	<u> </u>

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

Title	Date
<u>Final Remedial Investigation</u>	<u>November 2002</u>
<u> </u>	<u> </u>

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

Lead Organization: U.S. Navy (NAVFAC, Mid-Atlantic and MCAS Cherry Point EAD);
Federal Regulatory Agency: USEPA Region 4; State Regulatory Agency: NCDENR.

- 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:

The required SAP elements are included in this document.

SAP Worksheet #3 -- Distribution List

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Janice Nielsen	Remedial Project Manager	NAVFAC Mid-Atlantic	757-322-8339	Email: Janice.nielsen@navy.mil <u>Mailing/FedEx address:</u> Commander NAVFAC MIDLANT LRA, Building C, NC IPT 6506 Hampton Blvd Norfolk, VA 23508-1278	
Jeff Christopher	Installation Restoration Program Manager	MCAS Cherry Point Environmental Affairs Department	252-466-4421	Email: Jeffrey.christopher@usmc.mil <u>Mailing address:</u> MCAS Cherry Point PSC Box 8006 Cherry Point, NC 28533-0006 <u>FedEx address:</u> MCAS Cherry Point Building 4223, Access Road Cherry Point, NC 28533-0006	
Gena Townsend	Remedial Project Manager	USEPA Region 4	404-562-8538	Email: townsend.gena@epa.gov <u>Mailing/FedEx address:</u> USEPA Region 4 Atlanta Federal Center Superfund Division Federal Facilities Branch 61 Forsyth St. SW Atlanta, GA 30303-3104	

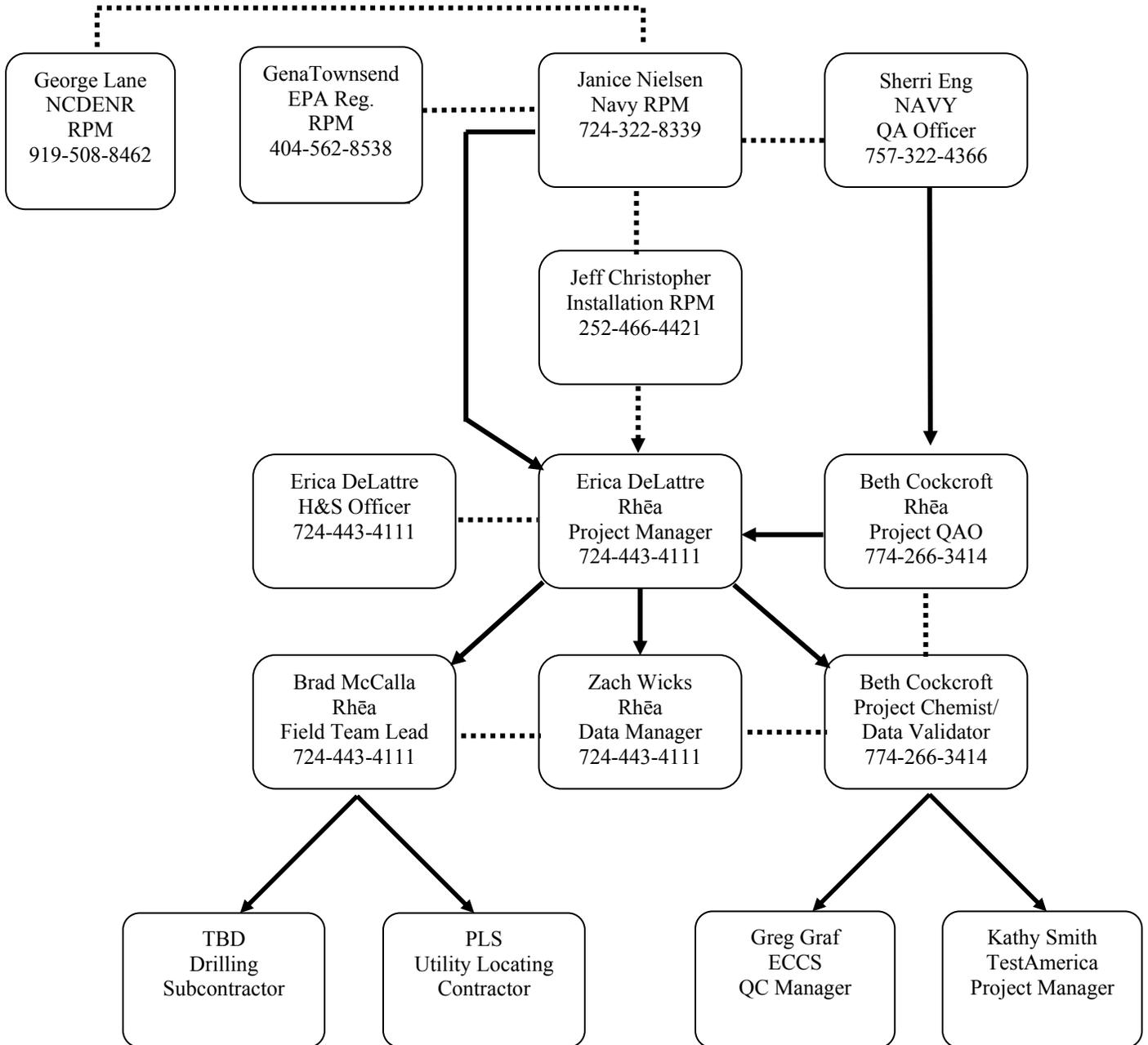
Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
George Lane	Remedial Project Manager	NCDENR	919-508-8462	Email: george.lane@ncmail.net Mailing address: NC Department of Environmental and Natural Resources, Superfund Section 401 Oberlin Road, Suite 150 1646 Mail Service Center Raleigh, NC 27699-1646 FedEx address: NC Department of Environmental and Natural Resources, Superfund Section 401 Oberlin Road, Suite 150 Raleigh, NC 27605	
Erica DeLattre	Project Manager / Health & Safety Officer	Rhēa	724-443-4111 724-316-6593 (cell)	erica@rhea.us	
Beth Cockcroft	Project Chemist / Data Validation	Rhēa	774-266-3414 (cell)	Email: beth@rhea.us Mailing address: 510 Bird Road Mansfield, MA 02048	
Brad McCalla	Field Team Leader	Rhēa	724-443-4111	brad@rhea.us	
Doug Bitterman	Activity Manager	CH2M HILL	757-671-6209	Doug.bitterman@ch2m.com	
Bill Hannah	Project Manager	CH2M HILL	757-671-6277	Bill.hannah@ch2m.com	
Bonnie Capito	Librarian	NAVFAC Atlantic	757-322-4785	bonnie.capito@navy.mil	
Nick Nigro	President	ECCS	608-221-8700	nkn@eccsmobilelab.com	
Kathy Smith	Project Manager	TestAmerica /	912-354-7858	Kathye.smith@testamericainc.com	

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Beth Cockcroft	Rhēa / Data Validation	774-266-3414 (cell)			
Brad McCalla	Rhēa / Field Team Leader	724-443-4111			
Zach Wicks	Rhēa / Data Manager	724-443-4111			
TBD	Rhēa / Field Team Members				
Nick Nigro	ECCS / President	608-221-8700			
Greg Graf	ECCS / QA Manager	608-221-8700			
Kathy Smith	TestAmerica / Project Manager	912-354-7858			

SAP Worksheet #5 -- Project Organizational Chart

Lines of Authority ————— Lines of Communication



SAP Worksheet #6 -- Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Point of Contact with Partnering Team	Navy Mid-Atlantic / Remedial Project Manager	Janice Nielsen	757-322-8339	Primary point of contact for Navy; materials and information will be forwarded to the Partnering Team following review.
Point of Contact with MCAS Cherry Point	MCAS Cherry Point IR Program Manager	Jeff Christopher	252-466-4421	Oversees remedial activities at MCAS Cherry Point. Issues that may impact the MCAS Cherry Point operations are to be reported to him immediately.
Primary contact for Rhēa activities	Rhēa Project Manager	Erica DeLattre	724-443-4111 724-316-6593 (cell)	Primary point of contact for the Navy and MCAS Program Manager; Reports directly to the Navy Remedial Project Manager (RPM); Implements changes to the SAP.
SAP changes in the field	Rhēa Field Team Leader	Brad McCalla	724-443-4111	Notify the Rhēa Project Manager by phone and or email of changes to the SAP made in the field within 24 hours.
Reporting Lab Data Quality Issues (mobile lab)	Lead Site Chemist and/or QA Manager	Bob Osmundson Greg Graf	608-221-8700	All QA/QC issues with the project samples will be reported by the lab to the Field Team Leader within 2 business days.
Reporting Lab Data Quality Issues (fixed lab)	Project Manager	Kathy Smith	912-354-7858	All QA/QC issues with the project samples will be reported by the lab to the Project Chemist and Scientist (i.e., data manager) within 2 business days.
Release of Analytical Data	Project Chemist	Beth Cockcroft	774-266-3414	No analytical data can be released until validation is completed and the Project Chemist has approved the release.

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Janice Nielsen	Remedial Project Manager	NAVFAC	Coordinates Environmental Restoration activities at MCAS Cherry Point	
Jeff Christopher	Installation Restoration Manager	MCAS Environmental Affairs Department	Oversight of remedial activities at MCAS Cherry Point	
Erica DeLattre	Project Manager / Health & Safety Officer	Rhēa	Directs and oversees staff and subcontractors. Presents the findings of the investigation in a report for the Partnering Team. Oversees health and safety for field activities.	B.S. Civil Engineering Professional Engineer: Pennsylvania & North Carolina 17 years experience
Beth Cockcroft	Project Chemist / Data Validation	Rhēa	Performs oversight of laboratory, validates data, releases analytical data, data usability evaluation	B.S. Civil Engineering M.S. Environmental Engineering Professional Engineer 28 years experience
Brad McCalla	Field Manager	Rhēa	Supervises field sampling and coordinates all field activities	B.S. Environmental Systems Engineering 9 years experience
Zach Wicks	Data Management	Rhēa	Manages sample tracking, coordinates with laboratory and data validator, data management	B.S. Geo-Environmental Studies 1 year experience

TBD	Driller	TBD	Operates equipment used to collect soil samples	Will be certified driller in the state of North Carolina
Greg Graf	Laboratory Quality Assurance	ECCS	Manages laboratory effort (mobile lab), releases reports	
Kathy Smith	Laboratory Project Manager	TestAmerica	Manages laboratory effort (fixed based lab), releases reports	

SAP Worksheet #8 -- Special Personnel Training Requirements Table

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates
Field Team Leader	HAZWOPER Supervisor Training	C.E.S.T., Inc.	--	Brad McCalla	Rhēa	Rhēa office in Gibsonia, Pennsylvania, copy on-site during field work
Field Team Leader	First Aid / Blood Borne Pathogens	American Red Cross	8/22/07	Brad McCalla	Rhēa	Rhēa office in Gibsonia, Pennsylvania, copy on-site during field work

SAP Worksheet #9-1 -- Project Scoping Session Participants Sheet

Project Name: Operable Unit 1 Projected Date(s) of Sampling: Summer 2009 Project Manager: Erica DeLattre		Site Name: Site 83 Site Location: MCAS Cherry Point, North Carolina			
Date of Session: November 6, 2008					
Scoping Session Purpose: Partnering Team Meeting					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Janice Nielsen	Remedial Project Manager	NAVFAC	757-322-8339	Janice.nielsen@navy.mil	Manages ER, N (CERCAL/MRP) activities for MCAS Cherry Point
Jeff Christopher	Installation Restoration Manager	MCAS EAD	252-466-4421	Jeffrey.christopher@usmc.mil	Manages IRP Environmental activities at MCAS Cherry Point
Gena Townsend	Project Manager	USEPA Region 4	404-562-8538	townsend.gena@epa.gov	USEPA Regulator
George Lane	Remedial Project Manager	NCDENR	919-508-8462	george.lane@ncmail.net	State Regulator
Doug Bitterman	Activity Manager	CH2M HILL	757-671-6209	Doug.Bitterman@CH2M.com	User of data
Bill Hannah	Project Manager	CH2M HILL	757-671-6277	Bill.Hannah@CH2M.com	User of data
Erica DeLattre	Project Manager	Rhēa	724-316-6593	erica@rhea.us	Project Manager

Comments: Discussion of Site 83 Feasibility Study (FS).

Consensus Decisions: The Team agreed that additional soil sampling was required at Site 83 to supplement the FS.

The following is the summary of the Site 83 discussion from the Final Meeting Minutes (November 2008):

“Bill presented the purpose and assumptions of the FS for Site 83:

- *Remedial action objective* – to protect industrial workers and prevent migration of contaminants to groundwater
- *Remedial alternatives* – no action, land use controls, capping, and excavation with off-site disposal. LTM is included in all alternatives.
 - Assumptions and issues associated with the alternatives include:
 - There will be soil left on site with constituents that exceed SSLs
 - The technical implementation of excavation/cap activities may be difficult due to:
 - The extent of Industrial RSL exceedances and the proposed cap/excavation areas are estimated due to significant distances between soil samples with industrial RSL exceedances and lesser concentrations
 - Confirmation sampling during excavation activities may indicate that the excavation would need to extend further laterally from what is currently estimated
 - Down-slope area excavation at Site 83 assumes hazardous waste disposal due to chlordane-impacted soil and, as a result, costs could increase significantly if the excavation extent increases
 - Chlordane was the only pesticide analyzed as part of the 1996 FMD spill response
 - Depth of excavation estimated to 1 foot bgs since there will be less risk to industrial workers below this depth. Significant vegetation clearance and the removal of former building concrete pad would be required
 - Land Use Controls assumed to be sufficient to prevent human exposure

After Bill presented these assumptions, Doug reiterated that there are a significant number of assumptions upon which the FS is based, such that it makes CH2M HILL uncomfortable with moving forward on the FS without team acknowledgement and concurrence with the high degree of uncertainty. He asked for the team to think about what is the goal for the site - Is it to remove soil above the industrial RSL? Is it to ensure that the site will be protective of groundwater?

Gena asked if the risks generated in the 2002 RI were being used as the risk-basis for this site. Bill replied that it was but the new RSLs are being applied to help define the removal areas. Doug added that from an ecological risk standpoint, the team had already decided that nothing needed to be done.

Gena asked Bill what is the boundary of the contamination at Site 83. She said the chlordane hits are in the FMD area and it appears that those conducting the earlier removal action just did not go far enough in removing soil. Bill told Gena that she was correct; they started excavating to remove petroleum-related compounds but encountered chlordane and stopped. He said there is also an area immediately around the Site 83 boundary and the areas identified as driving the risk are due to exceedances of the industrial RSL. Gena said it seemed to her that if we address the Site 83 area and the area where FMD quit, the risk could go away. Bill replied that may be the case but there are not very many samples immediately around that area so there is still some uncertainty.

Gena said there are two options at this time: the FS can continue with the data we have or there can be an additional investigation to go back out and get data. Doug agreed but said the lateral and vertical extents of the removal area will have to be refined at some point. He said he was

concerned that what we learn from this eventual sampling might change the team's opinion on the selected remedy. George said he thinks it is clear that additional samples are necessary, and we just need to decide when to collect the samples.

Bill asked the team if they are comfortable with proceeding with the FS submittal given the significant remedial assumptions/issues. Gena said that she was fine with moving forward with the FS and completing the ROD. She said if we proceed with an excavation remedy and the actual quantity exceeds what has been assumed, we need to stop and revisit everything. She added if nothing changes but the quantity the ROD is still acceptable as the changes would be fairly insignificant.

Given the location and use of the area near Site 83, Doug asked if it was even necessary to spend the money to clean the site up via a removal action. Gena said if chlordane is driving the risk and it is a listed waste, the contamination can not just be left there. Doug replied that the exposure pathway would be someone going into the woods and digging around in the dirt, so LUCs would seem to be sufficient. Gena said LUCs would be sufficient if the contamination was not within the first foot of soil.

Gena said regulations are likely to become more conservative over time. She said that she does not believe any additional removal volume would actually lead to a different alternative for the site. Jeff said once the ROD is complete, the removal action has to start within a year or so and he is concerned that the excavation area would be open for a long time while the answers to all of the questions about the site are figured out. Gena replied that you do not have to start digging right away; rather you just need to show continuous operations at the site. She said that will allow for collecting pre-excavation samples to delineate the extent. Gena said she does not see the need to collect samples at this point since it will not change the remedy. She just suggested that conservative assumptions be made when the costs are being developed.

Jan asked if the current plan for the FS is only to dig a certain amount and leave anything below that in place or will the excavation continue to chase the contamination and try to get all of it. Bill said the FS currently assumes that the excavation is only to go to a depth of one foot. Gena said based on previous investigations we know there are two areas with elevated concentrations at 3 feet below ground surface, so the FS needs to factor in an area with a depth of up to 3 feet to get additional quantity. Jan clarified that since they found the contamination at 3 feet, the excavation may have to go deeper still. Gena added that she does not think it is necessary to do anything where the previous removal action and backfill occurred.

Doug summarized that CH2M HILL will proceed with the FS using the current assumptions and a sampling/delineation approach will be developed at a later date."

Note: Subsequent to the November 2008 Partnering Team Meeting, it was decided that the SAP and subsequent field sampling will be completed prior to proceeding with the FS.

SAP Worksheet #9-2 -- Project Scoping Session Participants Sheet

Project Name: Operable Unit 1 Projected Date(s) of Sampling: Summer 2009 Project Manager: Erica DeLattre		Site Name: Site 83 Site Location: MCAS Cherry Point, North Carolina			
Date of Session: February 19, 2009 Scoping Session Purpose: Partnering Team Meeting					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Janice Nielsen	Remedial Project Manager	NAVFAC	757-322-8339	Janice.nielsen@navy.mil	Manages ER, N (CERCAL/ MRP) activities for MCAS Cherry Point
Jeff Christopher	Installation Restoration Manager	MCAS EAD	252-466-4421	Jeffrey.christopher@usmc.mil	Manages IRP Environmental activities at MCAS Cherry Point
Gena Townsend	Project Manager	USEPA Region 4	404-562-8538	townsend.gena@epa.gov	USEPA Regulator
George Lane	Remedial Project Manager	NCDENR	919-508-8462	george.lane@ncmail.net	State Regulator
Doug Bitterman	Activity Manager	CH2M HILL	757-671-6209	Doug.Bitterman@CH2M.com	User of Data
Erica DeLattre	Project Manager	Rhēa	724-316-6593	erica@rhea.us	Project Manager
Tim Wenk	Staff Consultant	CH2M HILL	757-671-6265	Tim.Wenk@CH2M.com	Recorder

Comments: Meeting to discuss potential sampling locations, sampling area designations, sampling rationale, and analytical constituents. SAP worksheets #10, # 11, # 14, and #17 were discussed. A site visit was conducted by the Team to better understand the topography and surface conditions of Site 83.

Action Items: Prepare SAP

Consensus Decisions: The Team agreed on the sample locations, sampling rationale, and analytical constituents. The Team also agreed that the soil samples may be used as pre-confirmatory samples in the event that an excavation remedy is performed.

The following is the summary of the Site 83 discussion from the Final Meeting Minutes (February 2009):

“Following the site visit, the team held the scoping session for the Site 83 UFP-SAP that will support the collection of additional samples. The goal of the scoping session was to establish how data will ultimately be used, the sampling rationale, the sample locations, and the sample constituents. To support this, the team briefly reviewed the site history and previous investigations and removal actions.

Erica presented the proposed sample locations. She said a mobile laboratory will be used to analyze samples in the field, which will allow them to have a relatively dynamic sampling strategy. Samples will be collected starting at the surface down to 5 feet. The lab will analyze samples from 0-1 ft and 2-3 ft first and then analyze the deeper samples if the standards are exceeded (the 3-4 ft interval will be analyzed and, if the standards are exceeded in that sample, the 4-5 ft interval will be analyzed). The 1-2 ft interval will not be taken in order to reduce the number of samples.

The sample locations are based on a grid; where the rows and columns intersect, samples will be collected. Initially, only a small set of samples near the center of the investigation areas of the site will be analyzed. If the samples come back with results that exceed the standards, samples further away from the center of the site will be analyzed; samples will be collected radiating away from the center of the investigation areas. Doug suggested that Erica assign letters and numbers to the grids to allow them to readily identify where the samples originated.

Jan also suggested that the deeper samples collected from the area of the previous removal actions should be analyzed as well. Gena added that anywhere they did a removal action you need to be sure that you do not sample the backfill material.

Jan asked what would happen if the outermost samples come back with exceedances of the standards. Erica said they will take more samples further out. Doug added that we need to allow for collecting more samples using a “finer” grid (smaller grid spacing) if necessary.

Doug asked if Rhēa would be analyzing saturated samples, stating that it is effectively a groundwater sample and you cannot sort out the soil from the groundwater. Erica said the saturated samples will not be analyzed.

The team discussed the sample analysis suite for the sample areas. The site was divided into sub-areas A, B, and C, based on the results from the RI sampling, topography, and the historical removal actions at the site; the analysis suite for the area was based on the exceedances of the industrial RSLs found during previous sample events. Gena suggested that the constituents called out in the RI should all be included in the sample analysis, including metals. Doug stated that if we add metals to the analysis suite, using the mobile lab will not be as beneficial since metals require a completely different extraction technique. Bill added there are no single locations with just metals exceedances; the metals are all found in locations with occurrences of pesticides and PAHs.

Gena asked what created the risk at the site. Doug responded that PAHs and pesticides were the risk drivers. Bill reminded the team that the 2002 RI combined Sites 16 and 83 into one soil group. Gena asked why metals are shown in the figures if they aren't driving a risk. She said in the FS you are looking to manage the area with the COCs but the metals are not the COCs.

Gena also noted that there are several SSL exceedances at the site. Bill responded that the areas with SSL exceedances will be managed by LUCs and the areas with industrial RSL exceedances will be addressed through removal or capping. Gena replied that LUCs cannot be used to control a SSL exceedance; LUCs will not prevent leaching to groundwater, they are designed to maintain a remedy that has been implemented to prevent leaching. Jan said that a well is being installed at the site because there is not much groundwater data from the site. The well should help identify if there is a problem with constituents leaching from soil to groundwater. George stated that if a monitoring well is installed and there is a SSL exceedance, you will need to monitor for the constituents with a SSL exceedance, unless it is below two times the background concentration. Jeff agreed but added that if an area with a SSL exceedance is excavated we should not have to monitor for that constituent.

Initially, each area had a different proposed analysis suite, but that was changed following the team's discussion. All of the samples will be analyzed for:

- **Pesticides:** DDE, heptachlor, DDD, heptachlor epoxide, dieldrine, chlordane, DDT
- **PAHs** – benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, and indeno(1,2,3-cd)Pyrene

It was also noted that lead will be analyzed in the samples near the area that there were two historical elevated lead results. This is near the top of the slope on the east side of Area B.

Jeff asked why samples are being collected for pesticides that are near the industrial RSL but not exceeding it. Gena said it is just to confirm the previous samples and see if the constituents are there. Jeff disagreed with that rationale stating that the RI was supposed to identify the contamination. Bill said the rationale behind adding those constituents is that each investigation out there had a specific suite of analyses that did not necessarily include all of the COCs, and this will allow us to better determine the nature and extent of each COC. He added that the constituents may have been there but simply not included in the analytical suite for that event.

Erica added that performing the same analysis on all of the samples will also help with ease of analysis.

The sample data collected from the site will be validated.

Doug suggested that the sample locations should be collected with a GPS unit that is very accurate since there will not be confirmation samples following the removal action. He suggested that it may be worthwhile to drive rebar or survey pins into the ground at the sample locations. Erica said they will use an accurate GPS unit Rhēa recently purchased to record the sample locations.

Following the discussion, the team completed the UFP-SAP worksheets.”

SAP Worksheet #10 -- Problem Definition

10.1 SITE BACKGROUND

OU1 is an industrial area approximately 565 acres in size, located in the southwestern portion of MCAS Cherry Point (Figure 2). OU1 is bound by C Street and Sandy Branch to the northwest, portions of the MCAS Cherry Point flight line and runway to the northeast and southeast, and East Prong Slocum Creek to the southwest.

Site 83 (Figure 2) is a former pesticide mixing area, approximately one acre in size, located in the southwest portion of OU1, near Site 16 and East Prong Slocum Creek. The former pesticide shop (Building 96) was constructed before 1948 and was used as a pesticide mixing and storage area from 1965 to 1981 until a new pesticide shop was built at another location. Building 96 was used for storage of equipment and hazardous materials until 1997, when it was subsequently demolished. In early 2006 the Building 96 concrete foundation was removed during a non-CERCLA demolition project. Geotextile fabric was placed over the removed foundation area and covered with stone to eliminate potential exposure pathways.

The area around the Building 96 footprint is relatively flat and is covered by asphalt and concrete. A grassy area is located west of former Building 96, adjacent to the site boundary. The western edge of this grassy area contains a steep slope which leads to the wetland adjacent to East Prong Slocum Creek.

Site 83 was first identified by MCAS Cherry Point in 1997. A Solid Waste Management Unit (SWMU) Assessment Report (SAR) was conducted in 1998 that included the collection of soil, groundwater, and sediment samples. Groundwater and soil contamination was identified and an additional investigation of Site 83 was recommended as part of the comprehensive evaluation of OU1 (B&R, 1998). Soil contamination was also identified outside of the Site 83 boundary in the down slope wetland area. This area of contamination is considered to be associated with Site 83.

10.2 SUMMARY OF PREVIOUS INVESTIGATIONS AND SITE ACTIONS

The results of the environmental investigations conducted from 1983 to 2000 were presented in the 2002 Remedial Investigation (RI) (TetraTech, 2002). The results of the additional investigations conducted to further characterize the chlorinated Volatile Organic Compounds (VOCs) groundwater plume beneath OU1 were presented in the OU1 RI Addendum (CH2M HILL, 2008).

The following sections briefly summarize the various site investigations and other site actions completed to date at Site 83 (from oldest to most recent).

10.2.1 Facility Maintenance Department Spill Response

In February and April 1996, remedial activities were conducted for the cleanup of an oil spill near the Facility Maintenance Department (FMD) oil-water separator (OWS). The OWS was located south of Site 83, and the spill extended into the southern portion of Site 16. Petroleum-contaminated soil was excavated to depths ranging from 2 to 4 feet below ground surface (bgs) and confirmation samples were collected from the sides and bottom of the excavation. Excavated soil was disposed of offsite.

During the excavation activities, pesticide contamination was observed in the soil based on visual and olfactory observations. Ten soil samples collected from the sides and bottom of the excavation were analyzed for chlordane. Since pesticides were detected in the soil, the remedial action for the oil spill was stopped. The details of the investigation are presented in the FMD Spill Response Summary Report, Operable Unit 1, Site 16 (FMD Spill Response Summary Report) (OHM, 1996).

There is uncertainty regarding the locations of the soil samples collected during this spill response, as the 2002 RI (TetraTech, 2002) locations do not corroborate the locations identified on the FMD Spill Response Report (OHM, 1996). Similarly, the 2002 RI reported that the excavation bottom confirmation samples 16-FMD-CP63CS070, 16-FMD-CP63CS071, 16-FMD-CP63CS073, and 16-FMD-CP63CS075 were collected at a depth of 0 to 1 feet bgs; however, it is also documented that the samples were collected at a depth of 3 feet bgs (OHM, 1996).

10.2.2 Solid Waste Management Unit Assessment

In 1997, MCAS Cherry Point notified NCDENR and USEPA that a new SWMU had been discovered at Building 96, and the area was subsequently designated as Site 83. Multiple soil, sediment, and groundwater samples were collected and three monitoring wells were installed in the vicinity of the former Building 96. Details of the investigation are documented in the SWMU Assessment Report for Site 83, Building 96 Former Pesticide Mixing Area, Marine Corps Air Station Cherry Point, North Carolina (B&R, 1998). Pesticides and PAHs were detected in the surface soil at concentrations that pose an unacceptable risk to industrial workers; however, these concentrations were detected beneath the building concrete pad and did not provide a complete exposure pathway.

Fewer pesticides were detected with depth in the soil. No PAHs or pesticides were observed in groundwater.

10.2.3 Debris Piles Removal Action

A CERCLA Time-Critical Removal Action was conducted southwest of Building 96 in 1997 related to the numerous debris piles, tanks, empty storage vessels, and other construction debris on the site. Asbestos-containing material, debris, and soil contaminated with petroleum hydrocarbons, asbestos, and lead were removed for offsite disposal. All visible surface debris was removed. Confirmation soil samples were collected to verify that action levels had been attained. The details are presented in the CERCLA Time-Critical Removal for Operable Unit 1, Site 16 Debris Pile (OHM, 1998).

10.2.4 Remedial Investigation for Operable Unit 1

Data from historical site investigations were used in conjunction with additional soil, sediment, groundwater, and surface water samples collected for the RI. Details are presented in the 2002 RI (TetraTech, 2002).

Soil samples from Site 83 (and Site 16) had the most frequent detections of PAHs. Pesticides were detected in the area around the former pesticide shop (Site 83 and Site 16 FMD Spill Area) and in surface water bodies within OU1. The 2002 RI (TetraTech, 2002) determined that in the Site 83 area, the presence of pesticides in soil were associated with the former pesticide shop.

Additional soil samples were collected as part of the 2002 RI (TetraTech, 2002) to define the extent of the pesticide contamination. Chlordane immunoassay field test kits were used to minimize the number of laboratory soil sampling locations, since chlordane was previously detected during the 1996 FMD spill response. The highest concentrations detected from the soil samples were found in area where surface debris (old metal containers, etc.) was identified. In addition, the soil sampling also detected elevated concentrations of other pesticides, including 4,4'-dichlorodiphenyldichloroethane (4,4'-DDD), 4-4' dichlorodiphenyl-dichloroethylene (4-4'-DDE), and 4-4'-dichlorodiphenyltrichloroethane (4-4'-DDT).

10.2.5 Baseline Ecological Risk Assessment

As part of the Baseline Ecological Risk Assessment (BERA) for OU1, additional soil samples and toxicity samples from small insects were collected at Site 83 to fill data gaps and address areas of uncertainty. Samples were collected and analyzed for SVOCs,

pesticides, PCBs, (Polychlorinated Biphenyls) metals, and cyanide. Other pesticides, including endosulfan II, endosulfan II sulfate, heptachlor, and heptachlor epoxide were detected in soil. Details are presented in the Baseline Ecological Risk Assessment for Operable Unit 1, Marine Corps Air Station Cherry Point, North Carolina (OU1 BERA) (CH2M HILL, 2005a).

10.3 PHYSICAL CHARACTERISTICS

Site 83 consists of the former Building 96 concrete pad and is relatively flat; however, further to the west, the ground surface slopes significantly downward in a westerly direction towards East Prong Slocum Creek. The area west of Site 83 consists of dense woods.

The depths to the groundwater table (i.e., thickness of the vadose zone) beneath Site 83 is generally seven feet which flows west towards East Prong Slocum Creek. The uppermost soils consist predominantly of fill material composed of sands, silts, and clays mixed with wood fragments that may extend to a depth of 10 feet below ground surface (bgs). Figure 3 presents the hydrogeologic conceptual model beneath OU1.

10.4 RECEPTORS

Potential exposure at Site 83 may occur under current and future land use scenarios. Construction workers, base employees, and maintenance workers may be exposed to Site 83 soils. Since the quality of the soil and steep hillside are poor habitat for soil invertebrates, it was concluded by the Partnering Team that additional ecological investigations or related action at Site 83 was unnecessary.

10.5 NATURE AND EXTENT OF CONTAMINATION

Historical soil sampling analytical data indicates that PAH and pesticides concentrations exceed various screening criteria within the Site 83 boundary and in soils located west/southwest of the site adjacent to East Prong Slocum Creek.

Historical soil sampling locations and exceedances of applicable screening criteria are shown on Figure 4. This figure is copied with permission from CH2M HILL's Draft Focused Feasibility Study, Sites 14, 15, 16, 18, 40, and 83, Operable Unit 1 (January 2009). The exceedances shown in Figure 4 are the basis for the additional sampling detailed in this SAP.

10.6 PROBLEM DEFINITION

The objective of this soil sampling event is to confirm residual contamination of PAHs and pesticides at the site, characterize portions of the site where information is limited or suspect, and to delineate the vertical and horizontal extent of impacted site soils. This information will be incorporated into a document, which will be used to provide vital information for developing feasible remedial alternatives. It is intended that these additional investigative samples be utilized as pre-confirmatory samples in the event that an excavation remedial alternative is selected.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements

- Who will use the data?

The data will be used by the Cherry Point Partnering Team (i.e., USEPA, NCDENR, Rhēa, NAVFAC, MCAS EAD, and CH2M HILL) to characterize the site.

- What are the Project Action Limits (PAL's)?

Applicable regulatory standards include Regional Screening Levels (RSLs) for industrial soil. The specific PALs for each constituent are included in Worksheet #15. RSLs for industrial soil were chosen based on the future land use of Site 83.

- What will the data be used for?

The data will be used to further characterize and delineate the lateral and vertical extent of soil contamination at Site 83, which can be used to determine the final volume of soil exceeding the RSLs for industrial soil. The data generated from the sampling will ultimately be used to evaluate potential remedial alternatives and design the remediation strategy for the area of contamination.

- What types of data are needed (matrix, target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?
 - Pesticides and PAH soil analysis will be performed on-site by a state-certified mobile laboratory. Lead analysis will be performed by a state-certified fixed based laboratory.
 - Target analytes include the PAHs benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, and indeno (1,2,3-cd) pyrene.
 - Target pesticides include dieldrin, chlordane, 4-4' DDT, 4-4' DDD, 4-4' DDT, heptachlor epoxide, and heptachlor.
 - Target metals include lead (one area only).
 - Soil samples will be collected using Direct-Push Technology (DPT). Samples will be collected from areas where historical samples indicate contamination above relevant standards.
 - Pre-confirmatory samples are needed to provide the horizontal and vertical limits of contamination.

- A minimum of 80 samples will be collected and analyzed from 31 primary sample locations (see Figure 5) during the delineation. The number and locations of the sampling points were discussed and agreed upon with the Partnering Team.
 - Samples will initially be collected and analyzed from the 0-1 foot and 2-3 foot intervals at Areas A and B, and from the 0-1 foot, 2-3 foot, and 3-4 foot intervals from Area C. Additional samples will be collected as described in Worksheet 14.
- How “good” do the data need to be in order to support the environmental decision?

The data will be of the quantity and quality necessary to provide technically sound and defensible assessment of the vertical and lateral extent of the contamination. Analytical results from each sample will be validated using procedures established by the *National Functional Guidelines for Organic* (USEPA, 1994) and *Inorganic Analyses* (USEPA, 1993).

- How much data should be collected (number of samples for each analytical group, matrix, and concentration)?

A minimum of 80 samples will be collected from 31 locations. Additional samples will be collected as needed to delineate the vertical and horizontal extent of contamination. Figure 5 shows the proposed sampling locations.

- Where, when, and how should the data be collected/generated?

DPT soil samples will be collected from Site 83 during one mobilization event. Field work is planned for the summer or fall of 2009. Samples will be collected in accordance with this UFP-SAP and Standard Operating Procedures (SOPs). SOPs are provided in Appendix A. Specific details of the sampling are provided in Worksheet #14.

- Who will collect and generate the data? How will the data be reported?

Rhēa personnel will collect the samples with the assistance of a TBD North Carolina certified driller.

ECCS, a NELAC and NCDENR certified mobile laboratory, will provide analytical services for the pesticide and PAH samples collected during this project. TestAmerica will provide fix-based analytical services for lead analysis. In addition, three split samples from Area A and two split samples from Area B will be submitted to TestAmerica for pesticide and PAH analysis to confirm the results with the mobile laboratory.

Hard-copy, Level IV data deliverables and corresponding Electronic Data Deliverables (EDDs) will be provided. EDDs will be in Microsoft Excel format to be uploaded into Naval Installation Restoration Information Solution (NIRIS).

- How will the data be archived?

Data will be archived electronically in NIRIS. Hard copy data will be archived in Rhēa's Gibsonia, Pennsylvania office.

- List the Project Quality Objectives (PQOs) in the form of if/then qualitative and quantitative statements.
 - The decision tree to be used for the data evaluation during this investigation is presented in Figure 6.
 - If analytical results from a particular location are above the RSL for Industrial Soils, then the adjacent (horizontal and vertical) sample will be analyzed. If analytical results are below the RSL for Industrial Soils, then it will be concluded that the site is delineated in that direction.

SAP Worksheet #12-1 -- Measurement Performance Criteria Table

Measurement Performance Criteria Table – Field QC Samples

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)
Equipment Blank	PAHs Soil	One per day (per equipment type)	contamination	No specific Analytical MPC	S&A
Ambient Field Blank	PAHs Soil	One per week	contamination	No specific Analytical MPC	S&A
Field Duplicate	PAHs Soil	One per 10 field samples	precision	No specific Analytical MPC	S&A
Matrix Spike/Matrix Spike Duplicate,	PAHs Soil	One every batch of 20 samples or less	precision/accuracy	MS recovery 70-130% MSD recovery 70-130% MS/MSD RPD <20%	A

SAP Worksheet #12-2 -- Measurement Performance Criteria Table

Measurement Performance Criteria Table – Field QC Samples

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)
Equipment Blank	Pesticides Soil	One per day (per equipment type)	contamination	No specific Analytical MPC	S&A
Ambient Field Blank	Pesticides Soil	One per week	contamination	No specific Analytical MPC	S&A
Field Duplicate	Pesticides Soil	One per 10 field samples	precision	No specific Analytical MPC	S&A
Matrix Spike/Matrix Spike Duplicate,	Pesticides Soil	One every batch of 20 samples or less	precision/accuracy	MS recovery 70-130% MSD recovery 70-130% MS/MSD RPD <20%	A

SAP Worksheet #12-3 -- Measurement Performance Criteria Table

Measurement Performance Criteria Table – Field QC Samples

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)
Equipment Blank	Lead	One per day (per equipment type)	contamination	<RL RL - 5.0ug/L	S&A
Ambient Field Blank	Lead	One per week	contamination	<RL RL - 5.0ug/L	S&A
Field Duplicate	Lead	One per 10 field samples	precision	MLG Limits Relative Standard Deviation (RSD)<20%	S&A
Instrument Blank	Lead	One every batch of 20 samples or less	contamination/bias	<RL RL - 5.0ug/L	A
Matrix Spike/Matrix Spike Duplicate, LCS	Lead	One every batch of 20 samples or less	precision/accuracy	MLG Limits RSD<20% Accuracy 75-125%	A
Method Blank	Lead	One every batch of 20 samples or less	contamination/bias	<RL RL - 5.0ug/L	A

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table

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
MCAS Cherry Point Remedial Investigation	Tetrattech NUS, Inc., Final Remedial Investigation Operable Unit 1, MCAS Cherry Point, North Carolina, November 2002	Tetrattech NUS, Inc. Soil and groundwater samples	Data will be used to determine the general vicinity of proposed sampling locations.	This data is limited to uncertain location, age, sampling and analytical methods.

SAP Worksheet #14 -- Summary of Project Tasks

14.1 Site Tasks

The technical approach for the proposed field activities is summarized below. Primary tasks associated with the investigation include mobilization and site setup, utility clearance, site clearing, direct push technology (DPT) soil sampling, and site restoration.

Rhēa will procure the following subcontractors to support the site investigation activities:

- Utility locator;
- Land clearing services;
- Driller;
- Mobile analytical laboratory; and
- Fixed analytical laboratory.

Rhēa will coordinate the site activities with NAVFAC and the MCAS EAD.

14.2 Mobilization and Site Setup

This task includes the mobilization of personnel and equipment to the work site, setup of the mobile laboratory, and marking areas for utility surveying and site clearing.

The ECCS mobile laboratory contains equipment with a radiological source that is used in the analysis of samples; therefore, in accordance with MCAS Cherry Point policy, a approval will be required to enter the base. Mobile laboratory personnel will obtain this approval prior to mobilization.

14.3 Utility Clearance

Prior to initiating intrusive activities, a Level B underground utility survey will be performed by a professional locating service to mark the lateral extent of existing underground utilities within the removal areas.

14.4 Clearing

Approximately 0.5 acres of trees and underbrush will be cleared prior to sampling activities. The vegetative material will be chipped/grinded in-place using heavy duty rubber tracked brush grinders or excavators mounted with saw and/or grinder heads.

14.5 Soil Sampling Procedures

The soil sample locations will be located with survey-grade Global Positioning System (GPS) equipment and the locations will be marked with pinflags prior to sampling.

Soil samples will be collected using DPT sampling methods. At each location, continuous soil cores will be collected to the required depth. Five soil samples will be collected at 1-foot depth intervals (0-1 foot (ft), 1-2 ft, 2-3 ft, 3-4 ft, and 4-5 ft) at each location. Three distinct areas were identified based on location and topography. These areas are identified as Area A -Site 83 (the former building 96 location) and adjacent lot, Area B - west of Site 83 (on the steep slope), and Area C - southwest of Site 83 (area of previous soil removals), and analyses will be as follows:

- Area A (Site 83 and adjacent lot) – benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, indeno (1,2,3-cd) pyrene, dieldrin, heptachlor epoxide, heptachlor, 4-4' DDE, 4-4' DDD, 4-4' DDT, and chlordane
- Area B (West of Site 83) – benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, indeno (1,2,3-cd) pyrene, dieldrin, heptachlor epoxide, heptachlor, 4-4' DDE, 4-4' DDD, 4-4' DDT, chlordane, and lead
- Area C (Southwest of Site 83) – benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, indeno (1,2,3-cd) pyrene, dieldrin, heptachlor epoxide, heptachlor, 4-4' DDE, 4-4' DDD, 4-4' DDT, and chlordane

The 0-1 ft and 2-3 ft depth intervals will initially be analyzed from each location at Areas A and B. If the 2-3 ft depth interval sample does not meet the applicable (RSL) (for industrial soil) for a particular compound, the 3-4 ft sample interval will be analyzed for the constituents that did not meet the RSL (for industrial soil) in the 2-3 ft depth interval. Likewise, if the 3-4 ft depth interval sample does not meet the applicable industrial standard for a particular compound, the 4-5 ft sample interval will be analyzed for the constituents that did not meet RSL (for industrial soil) in the 3-4 ft depth interval. For Area C, the 0-1 ft, 2-3 ft, and 3-4 ft depth intervals will be analyzed. Subsequent depth intervals will be analyzed, if necessary, as described above.

If the 0-1 ft analytical result is above the RSL, AND the 2-3 ft analytical result is below the RSL, the 1-2 ft depth interval will be analyzed. The 1-2 ft depth interval is considered a secondary sample. This additional analysis is being performed to better delineate the vertical extent of contamination.

In the event that the soil is not delineated in a specific direction, secondary soil samples will be collected. Anticipated secondary locations are shown on Figure 5.

Once an area is delineated in a particular direction, it is anticipated that additional soil samples will be collected between the exceedance and non-exceedance borings. These samples serve to tighten the delineation around a “hot” sample.

A Rhēa field team member will characterize the lithology (i.e., physical characteristics, soil type, cohesiveness, color, grain size, and relative moisture content) of the soil at each boring location and record the data into a field book. Upon completion of sampling at each location, equipment will be removed and excess sample material will be returned to the borehole. Boring locations will be backfilled with bentonite chips.

The Rhēa Field Team Leader can re-locate soil boring locations when site conditions warrant and with approval from the Rhēa Project Manager. Examples of scenarios where boring locations may be re-located include conditions where refusal is encountered or adequate sample material is not retrieved, or the presence of a physical obstacle.

Additional details associated with DPT soil sampling are provided in Appendix A (i.e., SOPs).

14.6 Surveying

Sample locations that were moved in the field and any other sample locations not identified in Figure 5 will be surveyed to provide northing and easting coordinates.

14.7 Quality Assurance/Quality Control

Quality control (QC) samples, including duplicates, matrix spike/matrix spike duplicate, trip blank, field blank, and equipment blank samples, will be collected in accordance with the Master Quality Assurance Plan (AGVIQ/CH2M HILL) dated November 2004. The quality control sample frequencies are as follows:

- Duplicates: 1 per 10 samples;
- MS/MSD: 1 per 20 samples;
- Field Blanks: 1 per week; and
- Equipment Blanks: 1 per day.

The quality control sampling frequency is also provided in Worksheet #12.

14.8 Equipment Decontamination

Non-disposable sampling equipment will be decontaminated prior to use and between sampling locations in accordance with applicable SOPs. Macro-core samplers and associated equipment used for soil sample collection will be decontaminated by scrubbing and washing with a Liquinox®-water solution and rinsing with potable water.

14.9 Investigative Derived Wastes

Investigative Derived Waste (IDW) is expected to consist of DPT soil cores, decontamination fluids, and personal protective equipment. Soil cores generated by sampling efforts will be containerized in 55-gallon steel drums, labeled, and subsequently sampled to determine disposal requirements. Disposal will be performed in accordance with MCAS Cherry Point guidance. IDW soil will be characterized and properly disposed of within 90 days of generation. Decontamination fluids generated by sampling efforts will be containerized and disposed of at the Industrial Wastewater Treatment Plant (IWTP). A chit signed by the MCAS Environmental Affairs Department (EAD) will accompany each delivery of water to the IWTP. Personal protective equipment and other wastes (i.e., gloves, disposable sampling equipment) will be rinsed and disposed of as regular trash.

14.10 Site Restoration

Cleared areas will be seeded and mulched upon completion of sampling activities.

14.11 Data Management

Samples will be tracked from collection through analysis by Rhēa's data manager. The analytical laboratories will provide Level IV data packages and Electronic Data Deliverables (EDDs) to Rhēa within 28 days from the completion of the field work.

14.12 Data Validation

Data will be validated as described in the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, and SW-846. Data qualifiers and data validation reason codes (as appropriate) will be added to EDD files. Rhēa's data manager will check the EDDs for completeness and accuracy, and will compare the EDD to the hard copy version of the reports. Final validated data will be incorporated into an

excel table once it is cleared by both the data validator/project chemist and data manager.
Final data will be uploaded into NIRIS.

SAP Worksheet #15-1 -- Reference Limits and Evaluation Table

Matrix: Soil
Analytical Group: PAHs

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal ² (µg/kg)	Mobile Laboratory ³		Fixed Laboratory ³	
					QLs	MDLs	QLs	MDLs
Benzo(a)pyrene	50-32-8	210	RSLs Industrial Soil	7.3 µg/kg	7.3 µg/kg	2.2 µg/kg	0.2 µg/kg	0.017 µg/kg
Benzo(a)anthracene	56-55-3	2,100	RSLs Industrial Soil	17 µg/kg	17 µg/kg	5.2 µg/kg	0.2 µg/kg	0.05 µg/kg
Benzo(b)fluoranthene	205-99-2	2,100	RSLs Industrial Soil	12 µg/kg	12 µg/kg	3.5 µg/kg	0.2 µg/kg	0.021 µg/kg
Dibenzo(a,h)anthracene	53-70-3	210	RSLs Industrial Soil	12 µg/kg	12 µg/kg	3.7 µg/kg	0.2 µg/kg	0.022 µg/kg
Indeno(1,2,3-cd)anthracene	193-39-5	2,100	RSLs Industrial Soil	11 µg/kg	11 µg/kg	3.4 µg/kg	0.2 µg/kg	0.050 µg/kg

¹Soil results will be compared to the RSLs for Industrial Soil.

²Project Quantitation Limit Goals were determined based on the Laboratory's achievable Quantitation Limit.

³Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

SAP Worksheet #15-2 -- Reference Limits and Evaluation Table

Matrix: Soil
Analytical Group: Pesticides

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal ² (µg/kg)	Mobile Laboratory ³		Fixed Laboratory ³	
					QLs	MDLs	QLs	MDLs
Dieldrin	60-57-1	110	RSLs Industrial Soil	40 µg/kg	40 µg/kg	0.47 µg/kg	3.3 µg/kg	0.31 µg/kg
Chlordane	12789-03-6	6,500	RSLs Industrial Soil	80 µg/kg	80 µg/kg	12 µg/kg	17 µg/kg	1.4 µg/kg
4,4' - DDT	50-29-3	7,000	RSLs Industrial Soil	40 µg/kg	40 µg/kg	0.47 µg/kg	3.3 µg/kg	0.52 µg/kg
Heptachlor epoxide	1024-57-3	190	RSLs Industrial Soil	20 µg/kg	20 µg/kg	0.26 µg/kg	1.7 µg/kg	0.10 µg/kg
Heptachlor	76-44-8	380	RSLs Industrial Soil	20 µg/kg	20 µg/kg	0.18 µg/kg	1.7 µg/kg	0.23 µg/kg
4-4' DDE	72-55-9	5,100	RSLs Industrial Soil	40 µg/kg	40 µg/kg	0.48 µg/kg	3.3 µg/kg	0.32 µg/kg
4-4' DDD	72-54-8	7,200	RSLs Industrial Soil	40 µg/kg	40 µg/kg	0.65 µg/kg	3.3 µg/kg	0.36 µg/kg

¹Soil results will be compared to the RSLs for Industrial Soil.

²Project Quantitation Limit Goals were determined based on the Laboratory's achievable Quantitation Limit.

³Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

SAP Worksheet #15-3 -- Reference Limits and Evaluation Table

Matrix: Soil
 Analytical Group: Metals

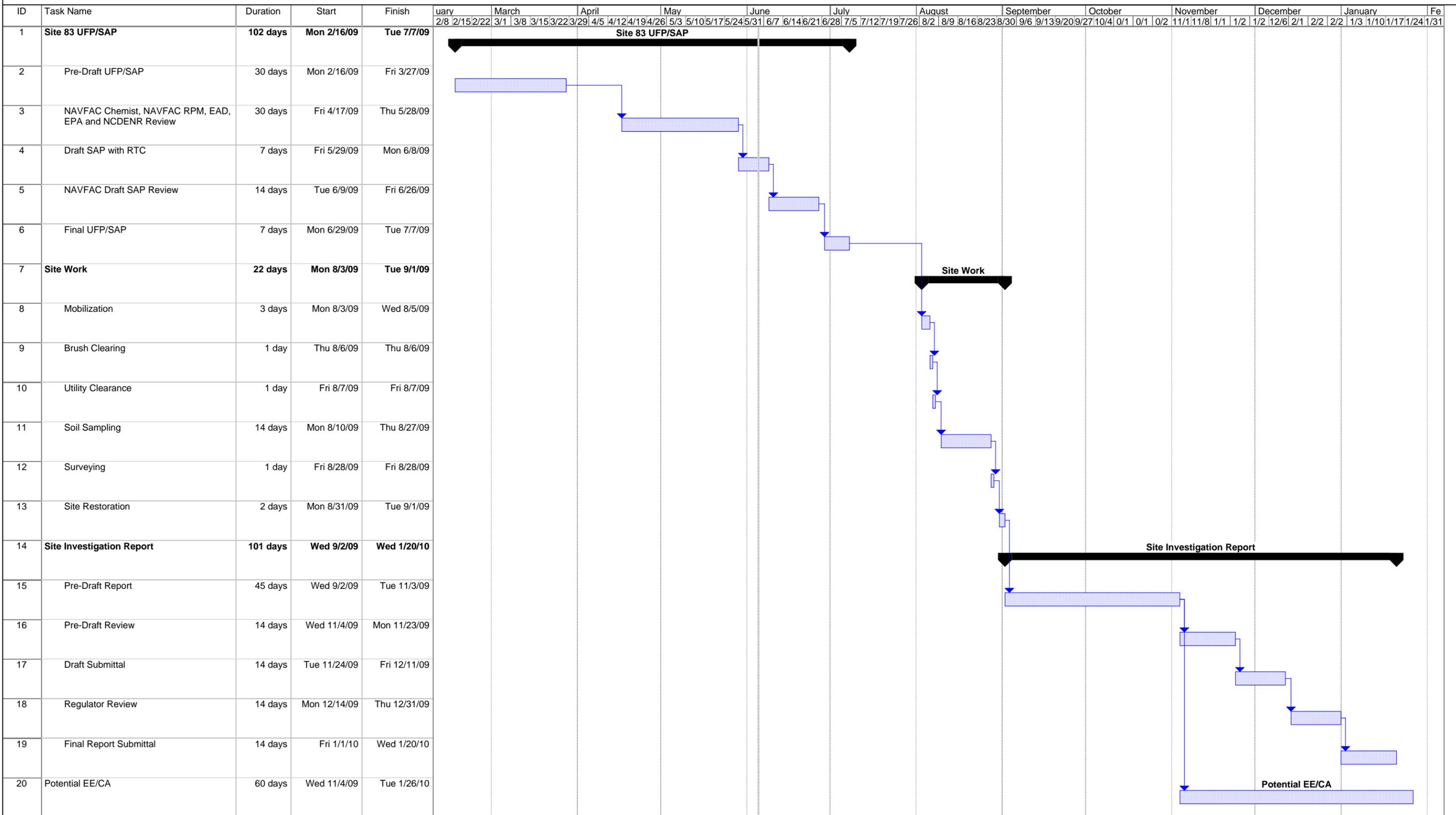
Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal ² (mg/kg)	Laboratory-specific ³	
					QLs	MDLs
Lead	7439-92-1	800	RSLs Industrial Soil	0.5 mg/kg	0.5 mg/kg	0.19 mg/kg

¹Soil results will be compared to the RSLs for Industrial Soil.

²Project Quantitation Limit Goals were determined based on the Laboratory's achievable Quantitation Limit.

³Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

SAP Worksheet #16 - Project Schedule
 OU1 Site 83 Soil Delineation Sampling
 MCAS Cherry Point, North Carolina



Project: Schedule.mpp Date: Fri 6/5/09

Task Progress Summary External Tasks Deadline

Split Milestone Project Summary External Milestone

SAP Worksheet #17 -- Sampling Design and Rationale

The sampling design and rationale was developed using the Guidance for Performing Site Inspections Under CERCLA (Interim Final, USEPA/540-R-92-021, PB92-963375, September 1992) as a reference. The sampling locations were selected to confirm elevated concentrations from previous investigative activities, to characterize portions of the site that were not previously investigated, and to delineate the vertical and horizontal extent of impacted site soils. The number and locations of the sampling points were discussed and agreed upon with the Partnering Team (February 2009). The sampling locations are shown in Figure 5.

As described in Worksheet #14, soil samples will be collected using DPT sampling methods. At each location, continuous soil cores will be collected to the required depth. Five soil samples will be collected at 1-foot depth intervals (0-1 foot (ft), 1-2 ft, 2-3 ft, 3-4 ft, and 4-5 ft) at each primary location. Three distinct areas were identified based on location and topography. These areas are identified as Area A -Site 83 (the former building 96 location) and adjacent lot, Area B - west of Site 83 (on the steep slope), and Area C - southwest of Site 83 (area of previous soil removals), and analyses will be as follows:

- Area A (Site 83 and adjacent lot) – benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, indeno (1,2,3-cd) pyrene, dieldrin, heptachlor epoxide, heptachlor, 4-4' DDE, 4-4' DDD, 4-4' DDT, and chlordane
- Area B (West of Site 83) – benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, indeno (1,2,3-cd) pyrene, dieldrin, heptachlor epoxide, heptachlor, 4-4' DDE, 4-4' DDD, 4-4' DDT, chlordane, and lead
- Area C (Southwest of Site 83) – benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, indeno (1,2,3-cd) pyrene, dieldrin, heptachlor epoxide, heptachlor, 4-4' DDE, 4-4' DDD, 4-4' DDT, and chlordane

The 0-1 ft and 2-3 ft depth intervals will initially be analyzed from each location at Areas A and B. If the 2-3 ft depth interval sample does not meet the applicable RSL (for industrial soil) for a particular compound, the 3-4 ft sample interval will be analyzed for the constituents that did not meet the RSL (for industrial soil) in the 2-3 ft depth interval. Likewise, if the 3-4 ft depth interval sample does not meet the applicable industrial standard for a particular compound, the 4-5 ft sample interval will be analyzed for the constituents that did not meet RSL (for industrial soil) in the 3-4' depth interval. For Area C, the 0-1 ft, 2-3 ft, and 3-4 ft depth intervals will be analyzed. Subsequent depth intervals will be analyzed, if necessary, as described above.

If the 0-1 ft analytical result is above the RSL, AND the 2-3 ft analytical result is below the RSL, the 1-2 ft depth interval will be analyzed. The 1-2 ft depth interval is considered a secondary

sample. This additional analysis is being performed to better delineate the vertical extent of contamination.

If a sample from the 4-5 ft sample interval does not meet the applicable RSL (for industrial soil) for a particular compound, additional samples will be collected and analyzed at subsequent 1-foot depth intervals. In the event that the soil is not delineated in a specific direction, secondary soil samples will be collected. Anticipated secondary locations are also shown on Figure 5.

Once an area is delineated in a particular direction, it is anticipated that additional soil samples will be collected between the exceedance and non-exceedance borings. These samples serve to tighten the delineation around a “hot” sample.

The Rhēa Field Team Leader can re-locate soil boring locations when site conditions warrant and with approval from the Rhēa Project Manager. Examples of scenarios where boring locations may be re-located include conditions where refusal is encountered or adequate sample material is not retrieved, or the presence of a physical obstacle.

Sampling methods and quality control sample types and frequencies are included in Worksheet #14.

SAP Worksheet #18-1 – Area A Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
11E / OU1-83-11E-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
11E / OU1-83-11E-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
11E / OU1-83-11E-S-3-4	Soil	3-4	TBD	
11E / OU1-83-11E-S-4-5	Soil	4-5	TBD	
11G / OU1-83-11G-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
11G / OU1-83-11G-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
11G / OU1-83-11G-S-3-4	Soil	3-4	TBD	
11G / OU1-83-11G-S-4-5	Soil	4-5	TBD	
11I / OU1-83-11I-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
11I / OU1-83-11I-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	OU1-83-11I-S-2-3-P
11I / OU1-83-11I-S-3-4	Soil	3-4	TBD	
11I / OU1-83-11I-S-4-5	Soil	4-5	TBD	
11K / OU1-83-11K-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
11K / OU1-83-11K-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
11K / OU1-83-11K-S-3-4	Soil	3-4	TBD	
11K / OU1-83-11K-S-4-5	Soil	4-5	TBD	

SAP Worksheet #18-1 (cont'd) – Area A Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
13E / OU1-83-13E-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
13E / OU1-83-13E-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
13E / OU1-83-13E-S-3-4	Soil	3-4	TBD	
13E / OU1-83-13E-S-4-5	Soil	4-5	TBD	
13G / OU1-83-13G-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	OU1-83-13G-S-0-1-P
13G / OU1-83-13G-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
13G / OU1-83-13G-S-3-4	Soil	3-4	TBD	
13G / OU1-83-13G-S-4-5	Soil	4-5	TBD	
14E / OU1-83-14E-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
14E / OU1-83-14E-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
14E / OU1-83-14E-S-3-4	Soil	3-4	TBD	
14E / OU1-83-14E-S-4-5	Soil	4-5	TBD	

SAP Worksheet #18-1 (cont'd) – Area A Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
14E / OU1-83-14G-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
14E / OU1-83-14G-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
14E / OU1-83-14G-S-3-4	Soil	3-4	TBD	
14E / OU1-83-14G-S-4-5	Soil	4-5	TBD	

All soil samples will be collected in accordance with Field SOP 001 – Direct Push Soil Sampling.
 Sample locations will be field adjusted if no sample material is recovered or if refusal is encountered.

SAP Worksheet #18-2 – Area B Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
8H / OU1-83-8H-S-0-1	Soil	0-1	See Worksheets 15-1, 15-2, and 15-3	OU1-83-8H-S-0-1-P
8H / OU1-83-8H-S-2-3	Soil	2-3	See Worksheets 15-1, 15-2, and 15-3	
8H / OU1-83-8H-S-3-4	Soil	3-4	TBD	
8H / OU1-83-8H-S-4-5	Soil	4-5	TBD	
9G / OU1-83-9G-S-0-1	Soil	0-1	See Worksheets 15-1, 15-2, and 15-3	
9G / OU1-83-9G-S-2-3	Soil	2-3	See Worksheets 15-1, 15-2, and 15-3	
9G / OU1-83-9G-S-3-4	Soil	3-4	TBD	
9G / OU1-83-9G-S-4-5	Soil	4-5	TBD	
9I / OU1-83-9I-S-0-1	Soil	0-1	See Worksheets 15-1, 15-2, and 15-3	
9I / OU1-83-9I-S-2-3	Soil	2-3	See Worksheets 15-1, 15-2, and 15-3	
9I / OU1-83-9I-S-3-4	Soil	3-4	TBD	
9I / OU1-83-9I-S-4-5	Soil	4-5	TBD	
9K / OU1-83-9K-S-0-1	Soil	0-1	See Worksheets 15-1, 15-2, and 15-3	
9K / OU1-83-9K-S-2-3	Soil	2-3	See Worksheets 15-1, 15-2, and 15-3	OU1-83-9K-S-2-3-P
9K / OU1-83-9K-S-3-4	Soil	3-4	TBD	
9K / OU1-83-9K-S-4-5	Soil	4-5	TBD	

SAP Worksheet #18-2 – Area B Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
8H / OU1-83-8k-S-0-1	Soil	0-1	See Worksheets 15-1, 15-2, and 15-3	OU1-83-8H-S-0-1-P
8H / OU1-83-8k-S-2-3	Soil	2-3	See Worksheets 15-1, 15-2, and 15-3	
8H / OU1-83-8k-S-3-4	Soil	3-4	TBD	
8H / OU1-83-8k-S-4-5	Soil	4-5	TBD	

All soil samples will be collected in accordance with Field SOP 001 – Direct Push Soil Sampling.
Sample locations will be field adjusted if no sample material is recovered or if refusal is encountered.

SAP Worksheet #18-3 – Area C Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
6N / OU1-83-6N-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
6N / OU1-83-6N-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
6N / OU1-83-6N-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
6N / OU1-83-6N-S-4-5	Soil	4-5	TBD	
6O / OU1-83-6O-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	OU1-83-6O-S-0-1-P
6O / OU1-83-6O-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
6O / OU1-83-6O-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
6O / OU1-83-6O-S-4-5	Soil	4-5	TBD	
6P / OU1-83-6P-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
6P / OU1-83-6P-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
6P / OU1-83-6P-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
6P / OU1-83-6P-S-4-5	Soil	4-5	TBD	
6Q / OU1-83-6Q-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
6Q / OU1-83-6Q-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
6Q / OU1-83-6Q-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	OU1-83-6Q-S-3-4-P
6Q / OU1-83-6Q-S-4-5	Soil	4-5	TBD	

SAP Worksheet #18-3 (cont'd) – Area C Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
6R / OU1-83-6R-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
6R / OU1-83-6R-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
6R / OU1-83-6R-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
6R / OU1-83-6R-S-4-5	Soil	4-5	TBD	
7N / OU1-83-7N-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
7N / OU1-83-7N-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
7N / OU1-83-7N-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
7N / OU1-83-7N-S-4-5	Soil	4-5	TBD	
7O / OU1-83-7O-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
7O / OU1-83-7O-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	OU1-83-7O-S-2-3-P
7O / OU1-83-7O-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
7O / OU1-83-7O-S-4-5	Soil	4-5	TBD	
7P / OU1-83-7P-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
7P / OU1-83-7P-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
7P / OU1-83-7P-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
7P / OU1-83-7P-S-4-5	Soil	4-5	TBD	

SAP Worksheet #18-3 (cont'd) – Area C Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
7Q / OU1-83-7Q-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
7Q / OU1-83-7Q-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
7Q / OU1-83-7Q-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
7Q / OU1-83-7Q-S-4-5	Soil	4-5	TBD	
7R / OU1-83-7R-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
7R / OU1-83-7R-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
7R / OU1-83-7R-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
7R / OU1-83-7R-S-4-5	Soil	4-5	TBD	
8O / OU1-83-8O-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
8O / OU1-83-8O-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
8O / OU1-83-8O-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
8O / OU1-83-8O-S-4-5	Soil	4-5	TBD	
8P / OU1-83-8P-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
8P / OU1-83-8P-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
8P / OU1-83-8P-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
8P / OU1-83-8P-S-4-5	Soil	4-5	TBD	

SAP Worksheet #18-3 (cont'd) – Area C Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
8Q / OU1-83-8Q-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
8Q / OU1-83-8Q-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	OU1-83-8Q-S-2-3-P
8Q / OU1-83-8Q-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
8Q / OU1-83-8Q-S-4-5	Soil	4-5	TBD	
8R / OU1-83-8R-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
8R / OU1-83-8R-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
8R / OU1-83-8R-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
8R / OU1-83-8R-S-4-5	Soil	4-5	TBD	
9O / OU1-83-9O-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
9O / OU1-83-9O-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
9O / OU1-83-9O-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
9O / OU1-83-9O-S-4-5	Soil	4-5	TBD	
9P / OU1-83-9P-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
9P / OU1-83-9P-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
9P / OU1-83-9P-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
9P / OU1-83-9P-S-4-5	Soil	4-5	TBD	

SAP Worksheet #18-3 (cont'd) – Area C Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (feet)	Analytical Group	Field Duplicate
9Q / OU1-83-9Q-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
9Q / OU1-83-9Q-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
9Q / OU1-83-9Q-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
9Q / OU1-83-9Q-S-4-5	Soil	4-5	TBD	
9R / OU1-83-9R-S-0-1	Soil	0-1	See Worksheets 15-1 and 15-2	
9R / OU1-83-9R-S-2-3	Soil	2-3	See Worksheets 15-1 and 15-2	
9R / OU1-83-9R-S-3-4	Soil	3-4	See Worksheets 15-1 and 15-2	
9R / OU1-83-9R-S-4-5	Soil	4-5	TBD	

All soil samples will be collected in accordance with Field SOP 001 – Direct Push Soil Sampling.
Sample locations will be field adjusted if no sample material is recovered or if refusal is encountered.

SAP Worksheet #19 -- Analytical SOP Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference¹	Containers (number, size, and type)	Sample volume (units)	Preservation Requirements² (chemical, temperature, light protected)	Maximum Holding Time³ (preparation / analysis)
Soil	PAHs	8270C / LAM-008	4 oz wide-mouth jar with Teflon-lined cap	4 oz wide-mouth jar with Teflon-lined cap	none	14 days / 40 days
Soil	Metals	6010B / SA-ME-70 rev. 10	4 oz wide-mouth jar with Teflon-lined cap	4 oz wide-mouth jar with Teflon-lined cap	4-degrees Celcius	6 months
Soil	Pesticides	8081A / LAM-003	4 oz wide-mouth jar with Teflon-lined cap	4 oz wide-mouth jar with Teflon-lined cap	none	14 days / 40 days

¹ Analytical SOP References are summarized in Worksheet #23.

² It is assumed that the mobile laboratory will be extracting PAH and pesticide samples within hours of collection; therefore the preservation of samples to 4-degrees Celcius will not be required. If there is a delay in sample extraction, then the samples will be put on ice or refrigerated.

³ 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

Matrix	Analytical Group	No. of Samples¹	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks²	No. of Equip. Blanks	No. of Volatile Organic Analytes (VOA) Trip Blanks	No. of PT Samples³	Total No. of Samples to Lab
Soil	PAHs	76	8	4	3	15	0	0	106
Soil	Pesticides	76	8	4	3	15	0	0	106
Soil	Metals	8	1	1	1	1	0	0	12

¹ The number of samples identified only includes samples collected from the primary sample locations identified in Worksheets #18-1, #18-2, and #18-3. An unknown number of additional samples will be collected as part of this investigation. Additional quality control samples will be collected at the frequencies identified in Worksheet #14.

² One field blank sample will be collected per week of sampling. The total number provided is estimated based on the assumed duration of the project.

³ One equipment blank sample will be collected per day of sampling. The total number provided is estimated based on the assumed duration of the project.

SAP Worksheet #21 -- Project Sampling SOP References Table

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
001	Direct Push Technology Soil Sampling	Rhēa	Geoprobe sampling rods	N	
002	Decontamination of Soil Sampling Equipment	Rhēa	DI Water, Etc.	N	

¹ Field SOPs are provided in Appendix A.

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

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
NA ¹	NA	NA	NA	NA	NA	NA	NA

¹Not Applicable. No field monitoring and/or measurement equipment will be used during the field sampling event.

SAP Worksheet #23 -- Analytical SOP References Table

Lab SOP Number¹	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
<u>Mobile Laboratory</u>						
LAM-008	Alkylated PAHs, Revision No: 2, 07/01/08	Definitive	Soil, PAHs	GC-MS	ECCS, Inc.	N
LAM-003	OC pesticides by 8081A, Revision No: 4, 02/21/08	Definitive	Soil, OC Pesticides	GC-ECD	ECCS, Inc.	N
<u>Fixed Based Laboratory</u>						
ME51:09.20.07:4	Digestion Procedures for Solids for ICP and ICPMS (EPA 3050B)	Definitive	Soil	ICP	TA Savannah	N
SA-ME-70 rev. 10	Elements by ICP (EPA 6010B)	Definitive	Soil	ICP	TA Savannah	N

¹ Laboratory SOPs are provided in Appendix B.

SAP Worksheet #24 -- Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
<u>Mobile Laboratory</u>						
GC-MS	Internal Standard Average Response Factor, Quadratic	Initial and as needed	Average Response Factor % RSD <20, Correlation Coefficient >0.995	Instrument maintenance, Instrument recalibration	Analytical SOP requirements	LAM-008
GC-ECD	External Standard Calibration	Initial and as needed	Correlation Coefficient >0.995	Instrument maintenance, Instrument recalibration	Analytical SOP requirements	LAM-003
<u>Fixed Based Laboratory</u>						
ICP-D (Thermo Jarrell Ash 61E Trace)	Single standard and a blank	Daily	+/-10% (6010)	Recalibrate	Analyst	SA-ME-70
ICP-E (Varian 730-ES)	Single standard and a blank	Daily	+/-10% (6010)	Recalibrate	Analyst	SA-ME-70

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
<u>Mobile Laboratory</u>								
GC-MS	Setum change	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-008
GC-MS	Injection port liner change	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-008
GC-MS	Injection port gold seal change	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-008
GC-MS	Analytical column cutting	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-008
GC-MS	Analytical column replacement	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-008
GC-MS	MSD Source Cleaning	MS Tuning	Inspect Tune File	As needed	Acceptable Mass Spectra	Re-maintenance	Chemist	LAM-008
GC-MS	Gas cylinder replacement	View gas cylinder guage	View gas cylinder guage	As needed	200 psi	Re-maintenance	Chemist	LAM-008

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC-ECD	Septum change	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-003
GC-ECD	Injection port liner change	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-003
GC-ECD	Injection port gold seal change	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-003
GC-ECD	Analytical column cutting	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-003
GC-ECD	Analytical column replacement	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-003
GC-ECD	Analytical column ferrel replacement	Analytical injection	Inspect chromatogram	As needed	Acceptable chromatography	Re-maintenance	Chemist	LAM-003
GC-ECD	Electron Capture Detector (ECD) replacement	Analytical injection	Inspect chromatogram	As needed	Acceptable Electron Capture detector signal	Re-maintenance	Chemist	LAM-003

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC-ECD	Gas cylinder replacement	View gas cylinder gauge	View gas cylinder gauge	As needed	200 psi	Re-maintenance	Chemist	LAM-003
<u>Fixed Based Laboratory</u>								
ICP-D (Thermo Jarrell Ash 61E Trace)	Pump tubing changed as needed.	Wavelength profile checked daily.	Torch, injector tip, transfer tubing inspected and cleaned as needed	See individual items	NA	Re-maintenance	Analyst	SA-ME-70
ICP-E (Varian 730-ES)	Pump tubing changed as needed.	Wavelength alignment and torch alignment minimum of weekly.	Torch, injector tip, transfer tubing inspected and cleaned as needed	See individual items	NA	Re-maintenance	Analyst	SA-ME-70

SAP Worksheet #26 -- Sample Handling System

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Rhēa. Field SOPs are provided in Appendix A.
Sample Packaging (Personnel/Organization): Rhēa
Coordination of Shipment (Personnel/Organization): Rhēa
Type of Shipment/Carrier: Hand delivery for PAH and pesticide samples (mobile laboratory), FedEx Priority Overnight for lead samples (fixed based laboratory)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): ECCS chemist/TestAmerica receiving department
Sample Custody and Storage (Personnel/Organization): ECCS chemist/TestAmerica receiving department
Sample Preparation (Personnel/Organization): ECCS chemist/TestAmerica receiving department
Sample Determinative Analysis (Personnel/Organization): ECCS chemist/TestAmerica receiving department
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): As determined by the Rhēa Field Team Leader for on-site samples / 30 days after the final report is issued
Sample Extract/Digestate Storage (No. of days from extraction/digestion): As determined by the Rhēa Field Team Leader for on-site samples / 30 days after the final report is issued
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Rhēa for on-site samples/TestAmerica for lead samples
Number of Days from Analysis: As determined by the Rhēa Field Team Leader / 30 days after the final report is issued

SAP Worksheet #27 – Sample Custody Requirements Table

27.1 Sample Handling

Soil samples will be collected by the field team under the direction and supervision of the field team leader. Pesticides and PAH analysis will be performed by a mobile laboratory. Lead analysis will be performed by a fixed based laboratory.

27.1.1 Mobile Laboratory

Samples will be hand delivered to the on-site mobile laboratory for preparation and subsequent analysis. Since samples will be analyzed within hours of sample collection, samples will not be preserved with wet ice. Chain of custody (COC) documentation will be completed for each group of samples delivered to the mobile laboratory, as described in Section 27.4.

27.1.2 Fixed Based Laboratory

Sample containers will be placed into shipping containers as soon as possible. Packing will be provided between containers to avoid breakage. Shipping containers will be secured with strapping tape to avoid tampering during transport to the laboratory, and a custody seal will be placed on each container. Documentation, i.e., COC, will be placed in a sealed plastic bag within one of the shipping containers.

Chemical samples will be maintained at approximately four degrees Celsius during shipping. Shipping containers will be insulated coolers and packed with wet ice (dry ice, blue ice, or chemical cooling packs will not be used). Chemical samples will typically be delivered or shipped to allow for receipt at the laboratory within 24 hours of packaging.

Samples will typically be considered non-hazardous substances and will be transported to the laboratory by commercial shippers (i.e., Federal Express). Shipping labels for non-hazardous substances will identify the samples as “soil/water/air samples.”

Samples are not expected to be hazardous substances. If the sample is known, or expected to be a hazardous substance, a sample specific protocol will be developed prior to sample transport. The specific protocol will be in accordance with the requirements of Department of Transportation regulations for hazardous materials per 49 (Code of Federal Regulations (CFR) Parts 110-119. 3.4.

27.2 Field Sampling Quality Control

The Field Team Leader will conduct a preparatory meeting with sampling personnel prior to the field event to ensure that each team member understands his or her responsibilities. At minimum, sample collection procedures, sample labeling, quality assurance/quality control, sampling handling, and proper documentation will be discussed during this meeting. Applicable SOPs and SAP Worksheets will be reviewed by sampling team members prior to the field event.

Sample labeling, field documentation, and collection procedures will be observed and reviewed by the Field Team Leader during the field event. Field books and forms will be reviewed on a daily basis for accuracy and completeness.

27.3 Sample Labels

All sample containers will be labeled in advance of sampling activities. The sample label will include a unique sample identification number. The sample label will also include the date and time the sample was collected, the name (or initials) of the sampler, the sample location, and the required analysis. The label will also identify the container preservative, if any, as completed by the laboratory.

The sample identification number will be of the following form:

Operable Unit - Site name - Sample Grid Location - Matrix (liquid(L) or solid/soil(S) or gas(G)) – Top of Sample Interval – Bottom of Sample Interval

For example, a soil sample collected from the 0-1 foot depth interval from Grid Location 6I would have the following identification number:

OU1-83-6I-S-0-1

27.4 Sample Custody and Handling

A COC record will be completed for each shipping container of samples. The COC record will typically be completed on a carbon-copy form provided by the laboratory. The record will, at a minimum, contain the following:

- Site name;
- Full name of sampler;
- Sample identification number for each sample;
- Date and time of collection for each sample;
- Sample matrix (liquid or solid);
- Number of containers for each sample;
- Description of sample location for each sample;
- Required analyses for each sample;
- Preservation for each sample, if required;
- Notation whether samples shipped on ice or not;
- Notation if sample is expected to be highly contaminated;
- Signature of person(s) involved in chain of possession; and
- Transfer date(s) and time(s) in chain of possession.

The preparer of the COC form, i.e., sampler, will retain a copy of the form and attach the form to daily field logs for the project.

If the samples are shipped by common carrier, the COC form will be placed in a sealed plastic bag inside the shipping container and the shipping container secured with strapping tape and a custody seal. Thus, in the case of the common carrier, two signatures will occur on the final COC; one signature by the preparer of the form, and one signature of the sample custodian assigned by the laboratory. The sample custodian assigned by the laboratory will open the shipping container and will denote any breaks to the custody seal of the shipping container and/or damage to the shipping container or sample containers on the COC form.

27.5 Laboratory Management of Samples

27.5.1 Mobile Laboratory

The laboratory chemist will assign a laboratory number to each sample (to be denoted on the COC), log in the sample in a field logbook, and store the sample for subsequent

analysis. Any issues with samples and/or analysis will be communicated with the Rhēa Field Team Leader so that appropriate action can be taken.

27.5.2 Fixed-Based Laboratory

The laboratory sample custodian will assign a laboratory number to each sample (to be denoted on the COC), log in the sample in the laboratory logbook, and store the sample in a secured storage room or cabinet until assigned to an analyst for analysis. The sample shall be stored at conditions (i.e. four degrees Celsius if appropriate, etc.) and for maximum holding times identified by 40 CFR 136, as appropriate (USEPA “Guidelines Establishing Test Procedures For The Analysis Of Pollutants”).

The custodian will immediately contact the person completing the COC in the event the seal on the shipping container is broken, any discrepancies exist between the COC and sample labels, or any sample container is damaged. Problems noted by the sample custodian will be resolved with the sampler before the sample is assigned for analysis. Once the sample is received by the analyst, that person is responsible for its care and custody and that person should be prepared to testify that the sample was in his/her possession, or secured in the laboratory at all times until the analysis was performed.

27.6 Sample Disposal

27.6.1 Mobile Laboratory

Rhēa personnel will collect sample material from the mobile laboratory and store it with other solid IDW (i.e., soil cores) generated from the sampling activities. This material will be analyzed to determine disposal requirements as described in Worksheet #14.

27.6.2 Fixed Based Laboratory

The fixed based laboratory will dispose of all samples in accordance with the requirements the USEPA. The fixed base laboratory will be responsible for determination of whether each individual sample is “hazardous” or “non-hazardous” based upon guidelines established in 40 CFR 260. If deemed a hazardous waste by the laboratory, the sample and sample container will be disposed of at a facility permitted in accordance with the requirements of 40 CFR 264. If deemed a non-hazardous waste by the laboratory, the sample and sample container will be disposed of as a solid waste at a facility permitted in accordance with 40 CFR 257.

SAP Worksheet #28-1 -- Laboratory QC Samples Table

Matrix	Soil					
Analytical Group	PAH's					
Analytical Method / SOP Reference	LAM-008					
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	1 per 20 or less basis with a minimum of 1 per day	Method Blanks must be free of target compounds.	Reanalysis of Method Blank, Reextraction of Method Blank and associated samples, Qualification of data	Chemist	Contamination / Bias	Method Blanks must be free of target compounds. Surrogate recoveries of p-Terphenyl-d ₁₄ : 70 – 130 %
LCS	1 per 20 or less basis with a minimum of 1 per day	LCS recovery 70 – 130 %	Reanalysis of LCS, Reextraction of LCS and associated samples, Qualification of data	Chemist	Accuracy	LCS recovery 70 – 130 % Surrogate recoveries of p-Terphenyl-d ₁₄ : 70 – 130 %
MS/MSD	1 per 20 or less	MS recovery 70 – 130 % MSD recovery 70 – 130 % MS/MSD RPD < 20 %	Reanalysis of MS / MSD, Qualification of data	Chemist	Precision / Accuracy	MS recovery 70 – 130 % MSD recovery 70 – 130 % MS/MSD RPD < 20 % Surrogate recoveries of p-Terphenyl-d ₁₄ : 70 – 130 %
DFTPP Tune Standard	Prior to daily sample analysis, Beginning of each 12 hour analysis period	See Method LAM-008 for the 12 criteria of Key Ions and Ion Abundance Criteria	Perform instrument maintenance, Reanalysis of samples	Chemist	Instrument Performance	See Method LAM-008 for the 12 criteria of Key Ions and Ion Abundance Criteria

Matrix	Soil					
Analytical Group	PAH's					
Analytical Method / SOP Reference	LAM-008					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standard	3 Internal Standards Acenaphthene-d ₁₀ , Chrysene-d ₁₂ , Perylene-d ₁₂ added to each standard and sample prior to analysis	The response of the internal standards must not vary by <50% or >200% from their response in the mid-point standard (200 ng/ml) of the initial calibration.	Perform instrument maintenance, Reanalysis of standards, samples.	Chemist	Instrument Performance	The response of the internal standards must not vary by <50% or >200% from their response in the mid-point standard (200 ng/ml) of the initial calibration.

SAP Worksheet #28-2 -- Laboratory QC Samples Table

Matrix	Soil					
Analytical Group	Pesticides					
Analytical Method / SOP Reference	LAM-003					
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	1 per 20 or less basis with a minimum of 1 per day	Method Blank must be free of target compounds, but if present, must be < 50% of low standard.	Reanalysis of Method Blank, Reextraction of Method Blank and associated samples, Qualification of data	Chemist	Contamination / Bias	Method Blank must be free of target compounds, but if present, must be < 50% of low standard. Surrogate recoveries of Tetrachloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP): 60 – 140 %
LCS	1 per 20 or less basis with a minimum of 1 per day	LCS recovery 70 – 130 %	Reanalysis of LCS, Reextraction of LCS and associated samples, Qualification of data	Chemist	Accuracy	LCS recovery 70 – 130 % Surrogate recoveries of Tetrachloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP): 60 – 140 %

Matrix	Soil					
Analytical Group	Pesticides					
Analytical Method / SOP Reference	LAM-003					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	1 per 20 or less	MS recovery 70 – 130 % MSD recovery 70 – 130 % MS/MSD RPD < 20 %	Reanalysis of MS / MSD, Qualification of data	Chemist	Precision / Accuracy	MS recovery 70 – 130 % MSD recovery 70 – 130 % MS/MSD RPD < 20 % Surrogate recoveries of Tetrachloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP): 60 – 140 %
Breakdown Standard	Prior to daily sample analysis, Beginning of each 12 hour analysis period, End of sample analysis	Breakdown of 4,4'-DDT and Endrin must be < 15 %	Perform instrument maintenance, Reanalysis of samples	Chemist	Instrument Performance	Breakdown of 4,4'-DDT and Endrin must be < 15 %
Instrument Blank	1 per 20 or less basis with a minimum of 1 per day	Instrument Blank must be free of target compounds, but if present, must be < 50% of low standard.	Reanalysis of instrument Blank, Reanalysis of effected samples, Instrument maintenance	Chemist	Contamination	Instrument Blank must be free of target compounds, but if present, must be < 50% of low standard.

SAP Worksheet #28-3 -- Laboratory QC Samples Table

Matrix	Surface Soil					
Analytical Group	Lead					
Analytical Method / SOP Reference	6010					
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	1/batch (20 samples)	Less than RL	- Re-analyze - Re-prep/re-analyze	Primary Analyst / Department Manager	Accuracy/Bias-Contamination	No Target Compounds>RL
LCS	1/batch (20 samples)	MLG Limits 75-125%	- Re-analyze - Re-prep/re-analyze	Primary Analyst / Department Manager	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1 per 20 or less	MS recovery 75 – 125 % MSD recovery 75 – 125 % MS/MSD RPD < 20 %	Reanalysis of MS / MSD, Qualification of data	Primary Analyst / Department Manager	Precision / Accuracy	MS recovery 75 – 125 % MSD recovery 75 – 125 % MS/MSD RPD < 20 %
LFB	1 pair/batch (20 samples)	MLG Limits 75-125%, <20% RSD	- Re-analyze - Report and flag data	Primary Analyst / Department Manager	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits

SAP Worksheet #29 -- Project Documents and Records Table

Document	Where Maintained
Field Notebooks, Field Data Collection Sheets	Rhēa office
Chain-of Custody Records	Rhēa office
Airbills	Rhēa office
Communication Logs	Rhēa office
Corrective Action Forms	Rhēa office
Documentation of corrective action results	Rhēa office
Documentation of deviation from methods	Rhēa office
Documentation of internal QA review	Rhēa office
Electronic data deliverables	Lab data will be uploaded to NIRIS. EDD's will be saved on Rhēa's network server. Hardcopies will be stored in Rhēa's office.
Identification of QC samples	Rhēa office
Meteorological data from field (e.g., temperature)	Rhēa office
Sampling instrument decontamination records	Rhēa office
Sampling Instrument calibration logs	Rhēa office
Sampling location and sampling plan	Rhēa office
Field Investigative Report	Rhēa office, distribution list
IDW Disposal Chit	Rhēa's office, copy to MCAS EAD

Document	Where Maintained
Field Investigative Report	Rhēa office
Laboratory Reports	Lab data will be uploaded to NIRIS. EDD's will be saved on Rhēa's network server. Hardcopies will be stored in Rhēa's office.
Data Validation Reports	Rhēa's server

SAP Worksheet #30 -- Analytical Services Table

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)
Soil	PAHs	See Figure 5 and Worksheet #18	LAM-008	28 days from end of field activities	Environmental Chemistry Consulting Services, Inc. (ECCS) 2525 Advance Road, Madison WI, 53718 Contact: Nick Nigro 608-221-8700	TestAmerica
Soil	Pesticides	See Figure 5 and Worksheet #18	LAM-003	28 days from end of field activities	Environmental Chemistry Consulting Services, Inc. (ECCS) 2525 Advance Road, Madison WI, 53718 Contact: Nick Nigro 608-221-8700	TestAmerica
Soil	Lead	See Figure 5 and Worksheet #18	6010B	28 days from receipt of samples	TestAmerica Laboratories, Inc. 5102 LaRoche Avenue, Savannah, GA 31404 Contact: Kathy Smith 912-354-7858	TBD

TestAmerica (fixed based laboratory) will be utilized as the backup laboratory if ECCS (mobile laboratory) is no longer able to provide analytical services.
 A backup fixed based laboratory has not been determined at this time.

SAP Worksheet #31 -- Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	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)
Laboratory Audit	Maintain current NELAC and NCDENR certifications	Internal	Rhēa	Project QA Officer (Beth Cockcroft) will verify that laboratories are NELAC/NCDENR certified for appropriate analysis	TestAmerica-Kathy Smith/Project Manager ECCS-Nick Nigro / President	TBD TBD	Project QA Officer - Rhēa – Beth Cockcroft
Field Audit ¹	Minimum of one field audit every six months	Internal	Rhēa	Project QA Officer will verify that field team members are following sampling protocols and applicable SOPs	Field Team Leader and/or Project Manager	Project Manager	Project Manager
NFESC Laboratory Assessment	Laboratory must have current NFESC evaluation letter	External	U.S. Navy (NFESC)		Subcontracted Laboratory's QA Officer	Subcontracted Laboratory's QA Officer	Project QA Officer - Rhēa – Beth Cockcroft

¹ Field audits are generally performed every 6 months on a representative sampling project in accordance with Rhēa's quality control program. A field audit may be conducted on this project.

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

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory Audit ¹	Phone Log or Email	TestAmerica-Kathy Smith/Project Manager ECCS-Nick Nigro / President	Prior to project	Appropriate certification documents	Project Manager - Rhēa – Erica DeLattre	Prior to project
Field Audit ²	Written Audit Report	Rhēa Project Manager	verbal notification within 48 hours, audit report within 2 weeks	Memorandum	Rhēa QA officer and Field Team Members	Implement Corrective Action measures as soon as possible
NFESC Audit	Written Audit Report	TestAmerica-Kathy Smith/Project Manager	Within 2 months of audit	Memorandum	NFESC Auditor, TBD	Within two months of receipt of initial notification

¹ Laboratories will be required to have current NELAC and NCDENR certifications. Laboratories without these certifications will not be permitted to perform analyses.

² Field audits are generally performed every 6 months on a representative sampling project in accordance with Rhēa’s quality control program.

SAP Worksheet #33 -- QA Management Reports Table

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)
Site Investigation Report	After field sampling event	November 2009	Erica DeLattre/Rhēa	Stakeholders, see Worksheet #4

The Site Investigation Report will include the following:

- Summary of project QA/QC requirements and procedures
- Conformance of project activities to UFP-SAP requirements and procedures
- Status of project schedule
- Deviations from the approved UFP-SAP and approved amendments
- Results of data review activities (how much usable data was generated)
- Corrective actions implemented and their effectiveness (if needed)
- Data usability in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity
- Limitations on the use of data
- Additional data concerns (including, but not limited to those identified on page 104 of the the UFP-QAPP Manual, V.1., March 2005)

SAP Worksheet #34 -- Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain of custody & Sample management (Mobile Laboratory)	Chain of custody forms will be reviewed internally upon their completion and verified against the samples they represent. Rhēa personnel will sign the samples over to the on-site laboratory by signing the COC. The laboratory chemist will review the COC against the samples they represent prior to accepting samples. Laboratory samples will be logged in immediately upon receipt.	Internal	Rhēa team members/ECCS employees
Chain of custody & Sample management (Fixed Laboratory)	Chain of custody forms will be reviewed internally upon their completion and verified against the packed sample coolers they represent. Once review is complete, the COC will be signed. A copy of the form will be retained and the remaining copies will be placed inside the sample cooler for shipment.	Internal	Rhēa team members
Sample receipt and management (Fixed Laboratory)	Samples will be cross-referenced against the COC upon arrival at the laboratory. The laboratory project manager will communicate any discrepancies between the COC and samples to Rhēa.	Internal	Test America employees Test America Project Manager – Kathy Smith
Analytical data package	All analytical data packages will be verified internally by the laboratory performing the work for completeness prior to submittal. The laboratory shall complete the appropriate form documenting the organization and complete contents of each data package.	Internal	ECCS QA Officer – Greg Graf Test America Project Manager – Kathy Smith
Field Notebooks and forms	Field notes will be reviewed for accuracy and completeness	Internal	Rhēa Field Team Leader – Brad McCalla
Review of Analytical Reports	Analytical reports will be compared with the chain of custody for completeness and accuracy (i.e., correct sample identifications, sample dates, and analyses, etc.). Quality control samples will be verified for completeness.	External	Rhēa Data Manager – Zach Wicks

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

Step IIa / IIb¹	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Sampling Methods & Procedures / SOPs	Confirm that required sample handling, receipt, and storage procedures were followed, and that deviations were documented.	Rhēa Field Manager – Brad McCalla
IIa	Analytical Methods & Procedures	Establish that required analytical methods were used and that any deviations were noted.	Rhēa Field Manager – Brad McCalla Rhēa Data Validator/Chemist – Beth Cockcroft
IIa	Holding Times	Confirm that samples were analyzed within the required holding time.	Rhēa Field Manager – Brad McCalla Rhea Data Validator/Chemist– Beth Cockcroft
IIa	Analytes	Confirm that the required lists of analytes were reported as specified as per the chain of custody.	Rhēa Field Manager – Brad McCalla
IIa	Data Qualifiers	Determine that the laboratory data qualifiers were defined and applied as specified in methods, procedures, or contracts	Rhēa Data Validator/Chemist – Beth Cockcroft
IIa	Data Validation Report	Summarize deviations from methods, procedures, or contracts	Rhēa Data Validator/Chemist – Beth Cockcroft
IIb	Sampling Plan & Procedures	Determine whether the sampling plan was executed as specified, and evaluate whether the sampling procedures were followed with respect to equipment and proper sampling support	Rhēa Project Manager - Erica DeLattre
IIb	Co-located Field	Compare results of co-located field duplicates	Rhēa Data Validator/Chemist –

Step IIa / IIb¹	Validation Input	Description	Responsible for Validation (name, organization)
	Duplicates		Beth Cockcroft
IIb	Project Quantification Limits	Determine sample results met the project quantification and action limits specified in Worksheet #15	Rhēa Field Manager – Brad McCalla Rhēa Data Validator/Chemist – Beth Cockcroft
IIb	Performance Criteria	Evaluate QC data against project-specific performance criteria	Rhēa Data Validator/Chemist – Beth Cockcroft
IIb	Data Validation Report	Summarize outcome of comparison of analytical data to measurement performance criteria	Rhēa Data Validator/Chemist – Beth Cockcroft

¹ IIa=compliance with methods, procedures, and contracts

IIb=comparison with measurement performance criteria in the SAP

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

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa	Soil	PAHs/Pesticides/ Lead	Analytical methods and laboratory SOPs as presented in this SAP will be used to evaluate compliance with QA/QC criteria.	Rhēa Data Validator /Chemist – Beth Cockcroft
IIb	Soil	PAHs/Pesticides/ Lead	Comparison to Project Action Limits and method performance criteria	Rhēa Data Validator /Chemist – Beth Cockcroft

Data validation will be conducted as described in the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Review, and SW-846. Guidelines will be used to provide flagging guidance, but are not being used for quality control limit criteria.

Worksheets #12-1, 12-2, and 12-3 provide the measurement performance criteria.

SAP Worksheet #37 -- Usability Assessment

Describe the evaluative procedures used to assess overall measurement error associated with the project. Identify the personnel responsible for performing the usability assessment:

Individual analytes will be checked to confirm they are within acceptable quantitation and qualification limits (see Worksheet #15). Non-detected analytes will be evaluated to confirm that the required project quantitation limits were achieved. If project quantitation limits were achieved and the verification and validation yielded acceptable data, then the data is considered useable.

Laboratory qualifiers will be added to data when deficiencies or uncertainties arise. The data qualifiers J, UJ, K, L, and UL will be added to denote minor deficiencies or uncertainties. An R qualifier will denote a major QC deficiency. R qualified data is typically not considered useable.

Analytical data will be checked to ensure that the numerical values and qualifiers are appropriately transferred to the EDD. Deviations from the SAP will be reviewed to assess whether corrective action is warranted and to assess impacts to achievement of project objectives. Beth Cockroft, (Rhēa Data Validator) and Erica DeLattre (Rhēa Project Manager) will be responsible for performing the usability assessment.

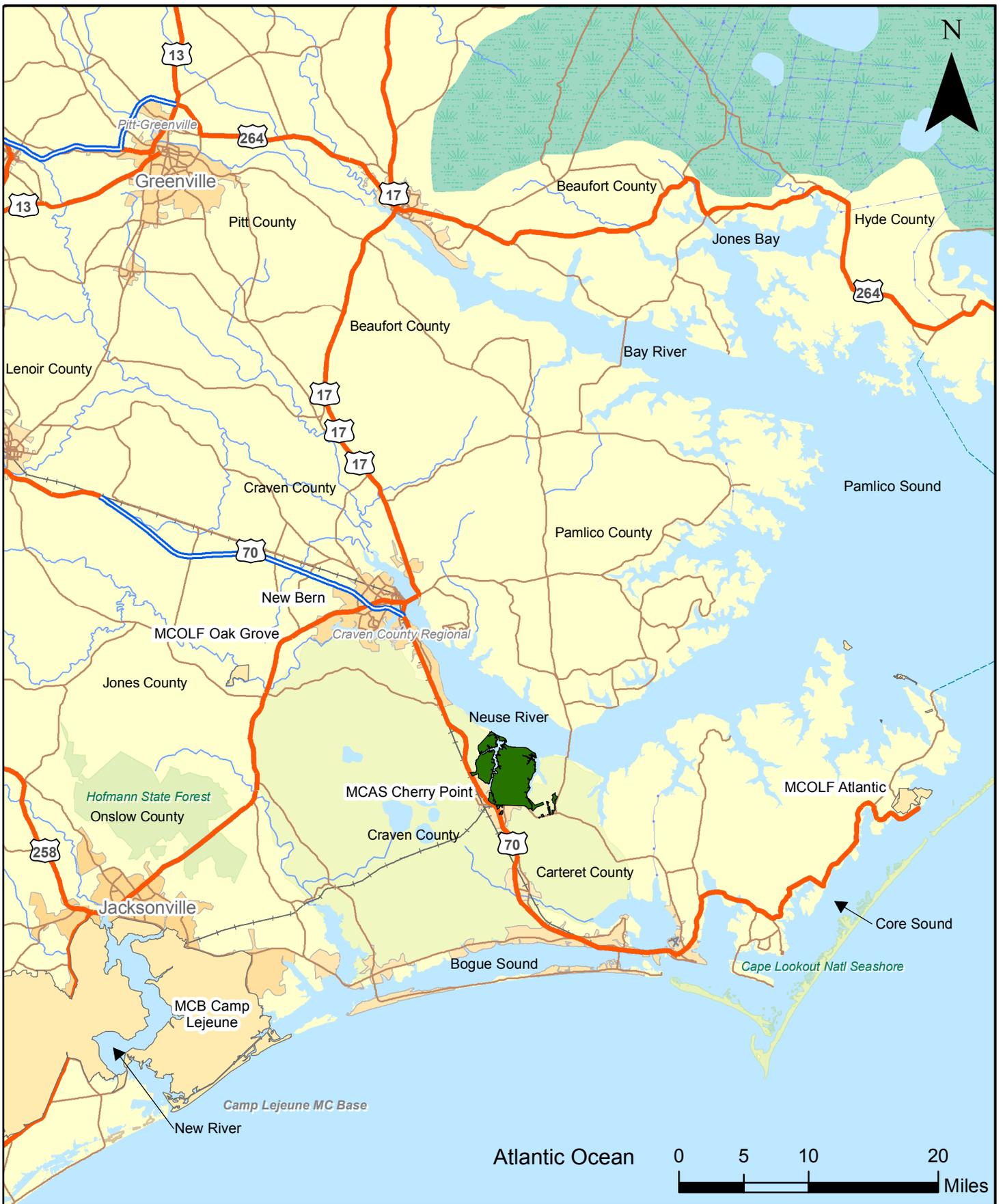
Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The Site Investigation Report will present the data, identify trends and relationships, and document anomalous results. Analytical data summary tables and data validation narratives will be included in the Site Investigation Report. Analytical data will be compared against the applicable screening criteria (i.e., screening levels for industrial soils). Data qualifiers will be included in the tables and discussed in the Site Investigation Report. The data validation narratives will include text evaluating data in regards to completeness, chain of custody documentation, holding times, calibrations, blanks, surrogate recoveries, field duplicates, matrix spike and matrix spike duplicated, LCS analyses, compound identifications, and quantitation/reporting limits.

Discuss how the entire project team should reconvene to perform the usability assessment to ensure that the PQOs are understood and the full scope is considered. Describe how data quality issues will be addressed and how limitations on the use of the data will be handled:

Data quality issues and limitations will be identified in the Site Investigative Report. The results of the sampling, including a discussion of any data gaps and/or usability, will be presented to the Partnering Team during one of the scheduled meetings. The Partnering Team will determine a course of action if it is determined that any of the PQOs were not achieved.

FIGURES



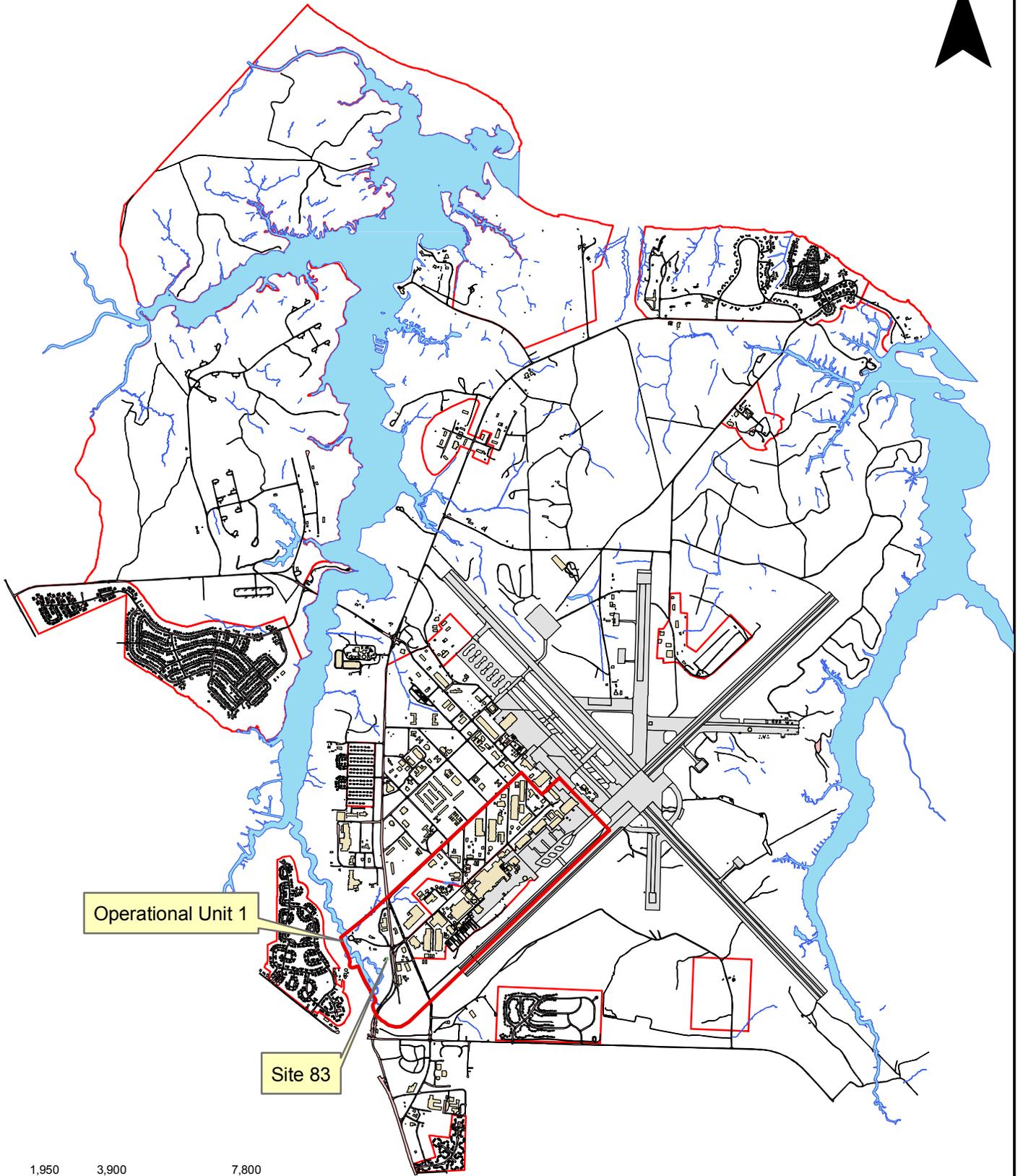
- Legend**
- MCAS Cherry Point
 - County Boundaries
 - Highway
 - Major Road
 - Local Road
 - Major Railroad Lines
 - Stream
 - Water Bodies



RHEA
Engineers & Consultants, Inc.

Figure 1
MCAS Cherry Point Location Map
MCAS Cherry Point, North Carolina

N



0 1,950 3,900 7,800 Feet

Legend

-  Site 83
-  Operable Unit 1
-  Water Bodies
-  Buildings
-  Base Area



Figure 2
OU 1 Location Map
MCAS Cherry Point, North Carolina

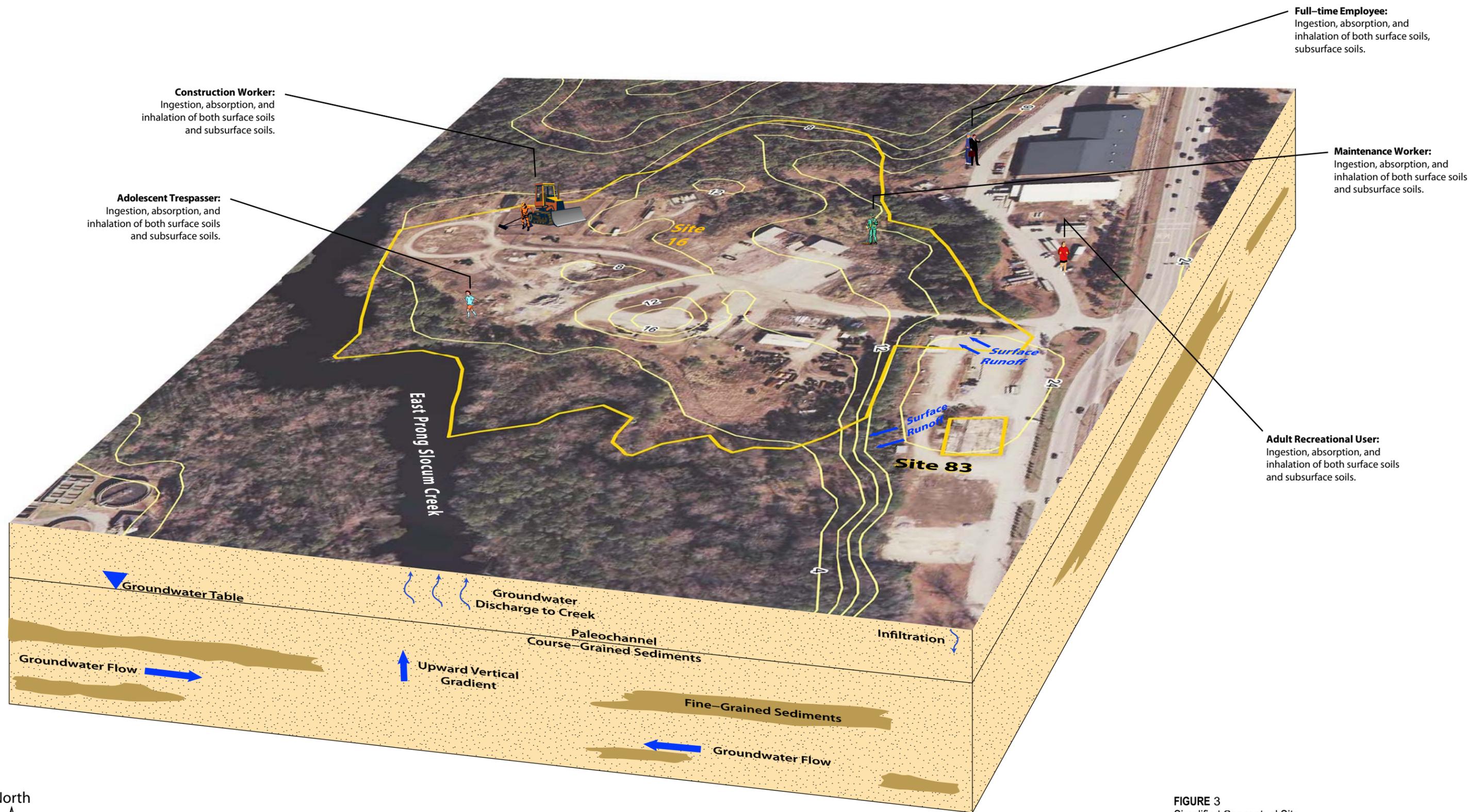
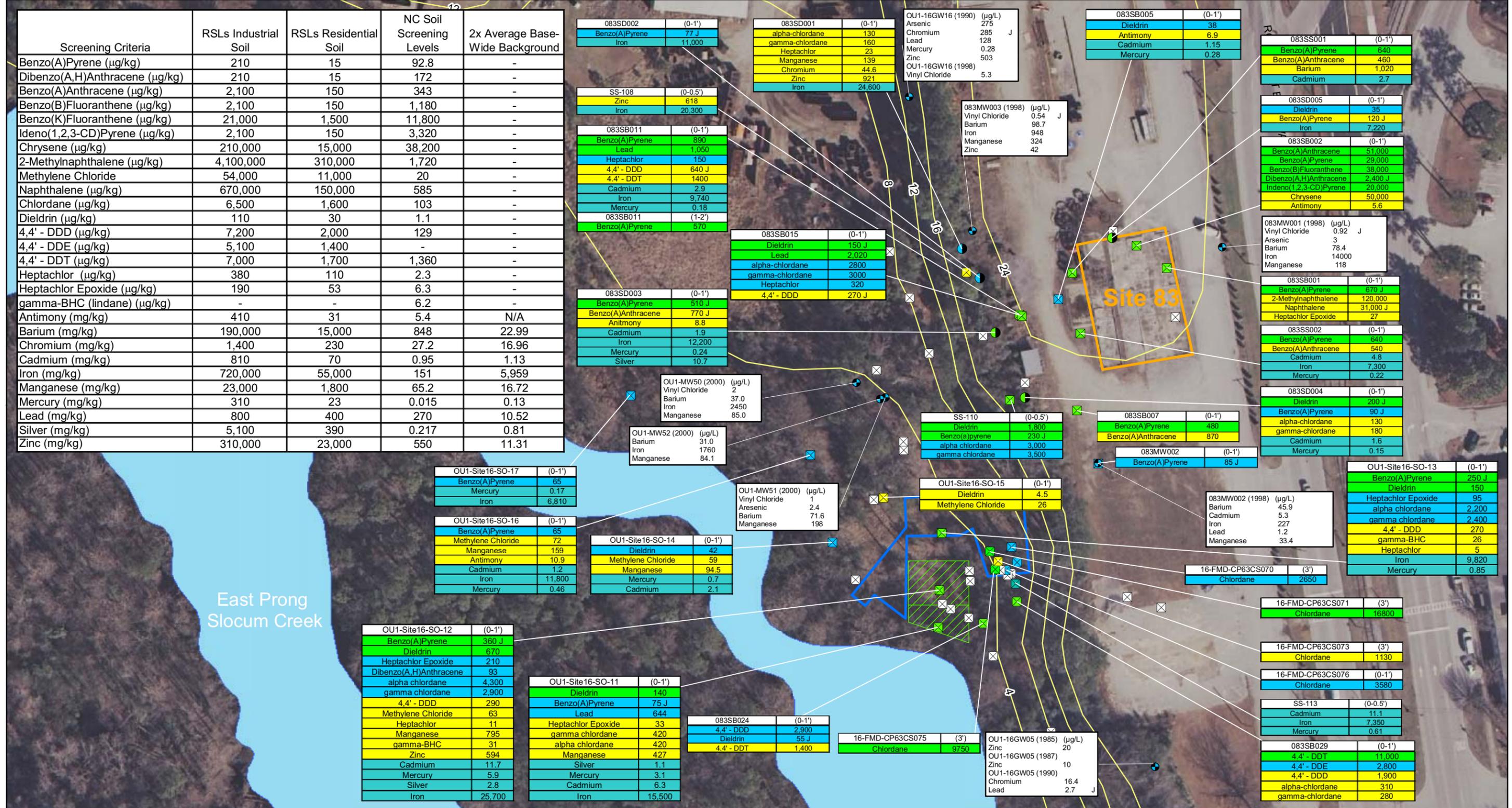


FIGURE 3
Simplified Conceptual Site
Model of Sites 16 and 83
Marine Corps Air Station Cherry Point
Cherry Point, North Carolina



Legend

- Sediment Sample Location with RSL Industrial Exceedance
- Sediment Location with RSL Residential Exceedance
- ⊠ Soil Boring Location with NC SSL Exceedance
- ⊠ Soil Boring Location with RSL Residential Exceedance
- ⊠ Soil Boring Location with RSL Industrial Exceedance
- ⊠ Soil Boring Location with Exceedance of Screening Criteria
- Monitoring Well
- Ground Surface Contour (4 feet interval)
- ⊠ 1996 FMD Soil Response Excavation Area
- ⊠ 1997 Excavated Area (Debris Piles Removal Action)
- ⊠ Site 83 Boundary

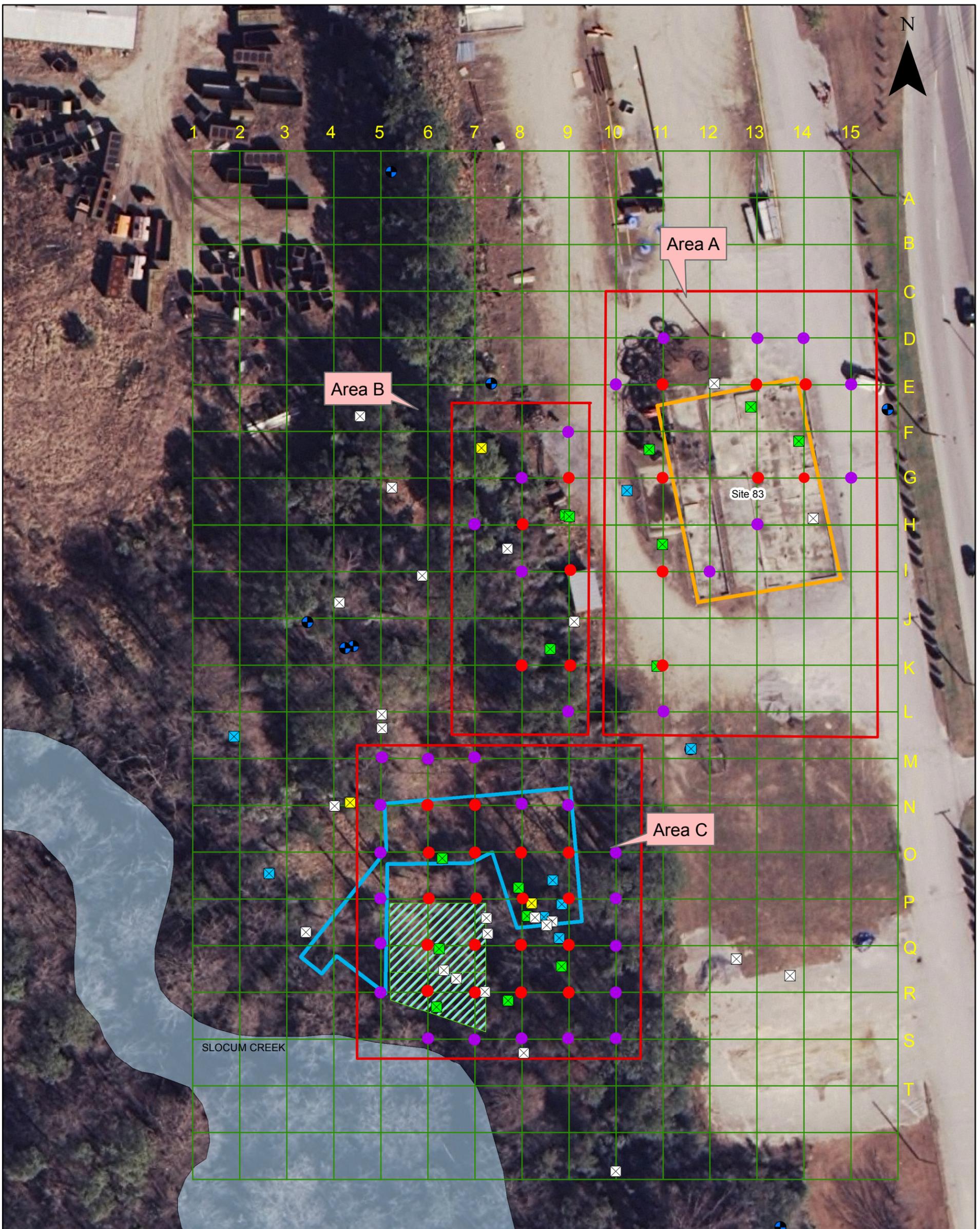
Notes:
 J - Estimated Value
 Shading exceeds RSL Industrial
 Shading exceeds RSL Residential
 Shading exceeds NC SSLs
 Shading exceeds 2x Base Background Average

NC SSL - North Carolina Soil Screening Levels
 FMD - Facility Maintenance Department
 µg/kg - micrograms per kilogram
 mg/kg - milligrams per kilogram

Soil Sample Locations and Analytical Results are provided from Tetra Tech, 2002

For samples exceeding more than one screening criteria for an analyte, the most conservative value is shown.

Figure 4
 Exceedances of Screening Criteria in Soil
 Operable Unit 1, Site 83
 MCAS Cherry Point, NC



0 40 80 Feet

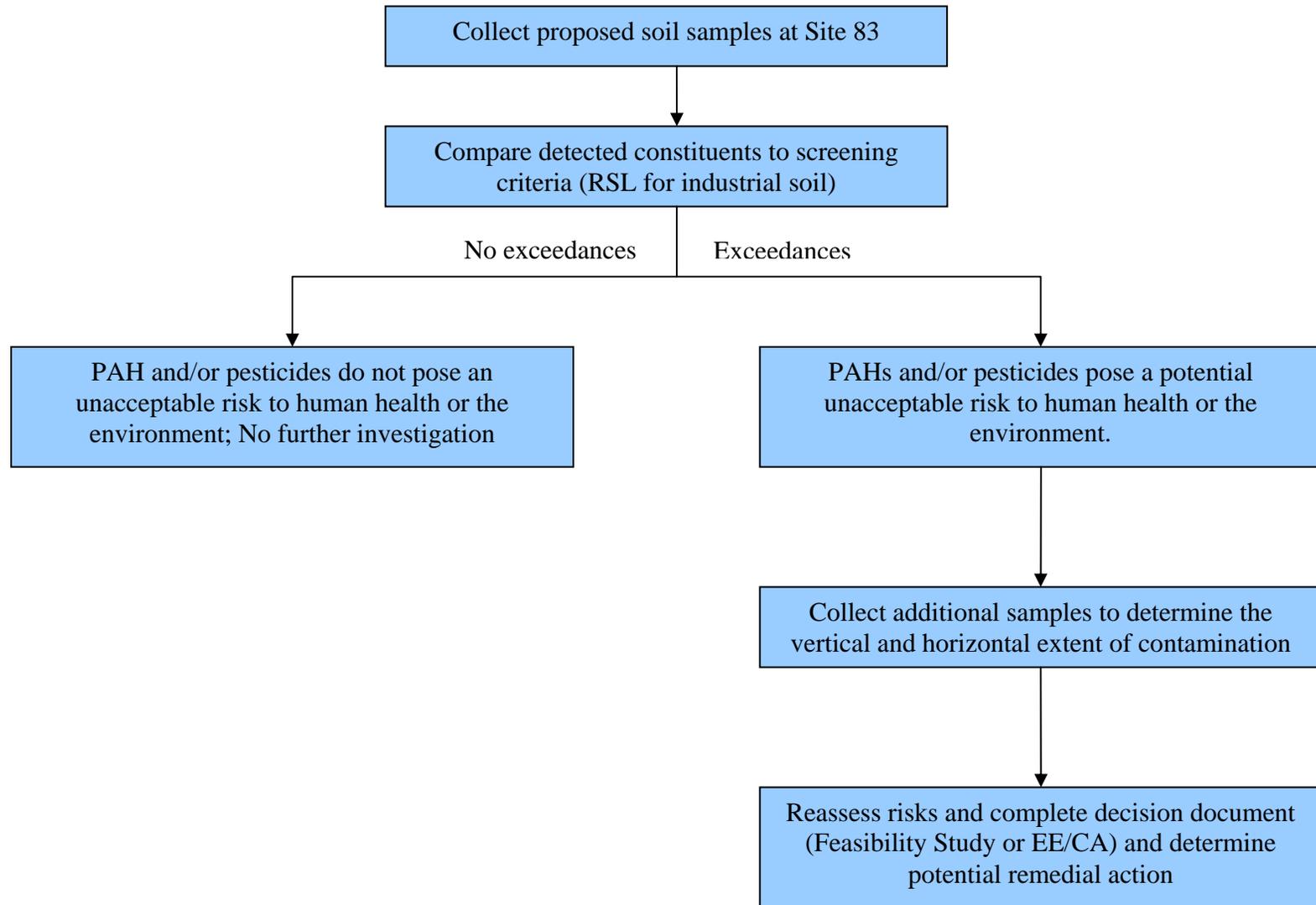
Legend

- Proposed Primary Soil Sample Location - 2009
- Proposed Secondary Soil Sample Location - 2009
- ⊠ Historical Soil Boring Location with No Exceedances
- ⊠ Historical Soil Boring Location with NC SSL Exceedance
- ⊠ Historical Soil Boring Location with RSL Residential Exceedance
- ⊠ Historical Soil Boring Location with RSL Industrial Exceedance
- Groundwater Sample Collection Points
- Site 83 Areas of Concern
- Site 83 25 ft. Grid Sampling
- Streams (Major)
- Site 83
- ▨ 1997 Excavation Area (Debris Piles Removal Action)
- 1996 FMD Spill Response Excavation Area



Historical soil sample locations are provided by TetraTech, 2002

Figure 5
Proposed Sample Locations
OU1, Site 83 Soil Delineation
MCAS Cherry Point, North Carolina



RSL – Regional Screening Level
 PAH – Polycyclic Aromatic Hydrocarbon
 EE/CA – Engineering Evaluation / Cost Analysis

Figure 6
Decision Tree
Site 83
MCAS Cherry Point, North Carolina

APPENDIX A

FIELD SOPs

Standard Operating Procedures

Direct-Push Soil Sample Collection

I PURPOSE

To provide a general guideline for the collection of soil samples using direct-push (e.g., Geoprobe®) sampling methods.

II SCOPE

Standard direct-push (e.g., Geoprobe®) soil sampling methods.

III EQUIPMENT AND MATERIALS

- Truck-mounted hydraulic percussion hammer;
- Sampling rods;
- Sampling tubes and acetate liners (if desired);
- Pre-cleaned sample containers and stainless-steel sampling implements; and
- Clean latex or surgical gloves.

IV PROCEDURES AND GUIDELINES

1. Decontaminate sampling tubes and other non-dedicated downhole equipment in accordance with SOP *Decontamination of Soil Sampling Equipment*.
2. Drive sampling tube to the desired sampling depth using the truck-mounted hydraulic percussion hammer. If soil above the desired depth is not to be sampled, first drive the lead rod, without a sampling tube, to the top of the desired depth.
3. Remove the rods and sampling tube from the borehole and remove the sample from the tube.
4. Fill all sample containers, beginning with the containers for VOC analysis, using a decontaminated or dedicated sampling implement.
5. Decontaminate all non-dedicated downhole equipment (rods, sampling tubes, etc.) in accordance with SOP *Decontamination of Personnel and Equipment*.
6. Backfill borehole at each sampling location with grout or bentonite and repair the surface with like material (bentonite, asphalt patch, concrete, etc.), as required.

V KEY CHECKS AND ITEMS

1. Verify that the hydraulic percussion hammer is clean and in proper working order.

2. Verify that the direct-push operator thoroughly completes the decontamination process between sampling locations.
3. Verify that the borehole made during sampling activities has been properly backfilled.

Standard Operating Procedures

Decontamination of Soil Sampling Equipment

1.0 Equipment Decontamination Procedures

Sample-taking equipment (i.e. shovel, spoon, probe, buckets, etc.) should be decontaminated prior to and after each sample collection. Tools used to handle soil will be brushed with a detergent solution and then flushed with a pressure washer containing potable water. A temporary decontamination pad constructed of a wooden frame or straw bales, and overlain by heavy-duty plastic will be used for this purpose.

Soil sampling equipment will be decontaminated by hand by the following method, unless alternate recommendations are provided by the equipment manufacturer:

- Wash with a solution of detergent;
- Rinse with tap water;
- Rinse twice with isopropanol or acetone; and
- Rinse twice with distilled water.

The wastewater collected should be contained and disposed of accordingly.

The decontamination area will be a temporary structure large enough to contain sampling equipment and the working end of the heavy equipment. The area should be used to collect, contain, and drain fluids to a central point so that the collected fluids can be pumped or drummed. The decontamination area will be constructed of materials that preclude puncturing or leakage caused by decontamination of activities. Racks should be built / provided to hold equipment and materials and keep them off the ground during decontamination. Equipment and materials should be covered kept on the racks when not in use. Decontaminated materials will be handled with unused latex or nitrile gloves to avoid further contamination prior to sampling.

APPENDIX B

LABORATORY SOPs



MODIFIED METHOD 8081A

ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS (PCB) AS AROCLORS BY GAS CHROMATOGRAPHY (GC)

1 SCOPE AND APPLICATION

- 1.1 This method is used for the determination of organochlorine pesticides, PCBs as Aroclors, and related compounds in aqueous and solid matrices. The following compounds can be determined by this method:

Compound	CAS Registry No.
Aldrin	309-00-2
α -BHC	319-84-6
β -BHC	319-85-7
γ -BHC (Lindane)	58-89-9
δ -BHC	319-86-8
α -Chlordane	5103-71-9
γ -Chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Methoxychlor	72-43-5
2,4'-DDE	3424-82-6
2,4'-DDD	53-19-0
2,4'-DDT	789-02-6
Hexachlorobenzene (HCB)	118-74-1
Pentachloronitrobenzene (PCNB)	82-68-8
Mirex	2385-85-5
trans-Nonachlor	39765-80-5
cis-Nonachlor	5103-73-1



Target Mixture	CAS Registry No.
Technical Chlordane (not otherwise specified)	57-74-9
Toxaphene	8001-35-2
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

- 1.2 Instrumentation: HP 5890 Gas Chromatograph (GC) equipped with an electron capture detector (ECD) and a LEAP Technologies CTC A200S autosampler and Agilent Chemstation Data Acquisition software
- 1.3 Compounds are all part of a class of compounds known as organochlorine pesticides. Historically included with this class is a class of compounds known as multi-responders (i.e. toxaphene, technical chlordane, and PCBs). While PCBs have their own method (8082), traditionally they can be analyzed from the same extract as the organochlorine pesticides.
- 1.4 When the method is used to analyze unknown samples, compound identification is achieved by retention times and supported by at least one additional qualitative technique. This method describes second column confirmation where two aliquots of each sample extract are injected on two GCs operating different capillary columns that result in different retention orders for a list of 22 analytes. Identification is considered absolute when both analytical columns have a peak at the expected retention time and the calculated results are within 50% difference. If both columns show presence of a compound, always report the lower number if the difference is greater than 50%. If one column has a positive result and the other one doesn't, the result is not confirmed and the compound is determined to be not present.
- 1.5 The estimated method detection limit (MDL) for each of the compounds in sand and water matrices is listed in Table 1. The detection limits given may vary based on sample size or sample matrix and/or regulatory requirements. Use of MDLs is still a requirement in Wisconsin, but many other states have adopted published reporting limits or using the low standard as the reporting limit.
- 1.6 Typical Initial Demonstration of Capability (IDC) studies for sand and water are listed in Table 2.
- 1.7 This method is restricted to use by, or under the supervision of, analysts experienced in the use of a gas chromatograph and skilled in the interpretation of chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this



method.

2 SUMMARY OF METHOD

- 2.1 The method provides for the extraction and GC conditions for the analysis of organochlorine pesticides in aqueous and solid matrices.
- 2.2 Soil samples are dried with sodium sulfate and extracted with a mixture of Iso-octane/Acetone. Water samples are extracted with dichloromethane, solvent evaporated, and made to volume with Iso-octane/Acetone.
- 2.3 The extract is transferred to injection vials and analyzed by GC-ECD.
- 2.4 The sensitivity and identification issues associated with this method depend on the level of matrix interferences in addition to instrumental limitations. If interferences are present, the resulting report limit may have to be elevated. Table 1 lists MDLs that can be obtained in sand and water in the absence of interferences.
- 2.5 Standard curves are generated for each analyte from a minimum five point standard curve using power regression as the regression analysis equation. Quantitation for each target analyte is done using reverse extrapolation.

3 INTERFERENCES

- 3.1 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents, or sample processing hardware; contaminated GC carrier gas, parts, column surfaces, or detector surfaces; and the presence of co-eluting compounds in the sample. Interferences co-extracted from the samples will vary considerably from sample to sample. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.
- 3.2 Interferences by phthalates introduced during sample preparation can pose a major problem in organochlorine pesticide determinations. Common flexible plastics contain varying amounts of phthalates, which are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interferences from phthalate esters. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.
- 3.3 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by detergent washing with hot water, three rinses with hot tap water, and three rinses with organic-free reagent water. Glassware/apparatus is then rinsed with dichloromethane or appropriate solvent just prior to use.



- 3.4 The presence of elemental sulfur will result in a broad peak that interferes with the detection of heptachlor epoxide through the DDT organochlorine pesticides on the RTX-35 and BHCs on the RTX-1701. Sulfur contamination should be expected with sediment samples. Liquid mercury cleanup is suggested for removal of sulfur. Since the recovery of Endrin aldehyde is drastically reduced, if possible this compound must be determined prior to sulfur cleanup, assuming it is a target analyte.

4 APPARATUS AND MATERIALS

4.1 Gas Chromatograph (GC)

- 4.1.1 Gas Chromatograph: HP5890
Autosampler: LEAP Technologies A200S
Detector: HP Electron Capture
Injector: Split/splitless Injector
Data system: Agilent Chemstation chromatography data system using HPIB capable of presenting chromatograms, retention time, peak integration data and calculating standard curves using regression analysis.

- 4.1.2 Dual column analysis: Columns are placed in two separate GCs set up for separation of as many of the analytes as possible.

4.1.2.1 Column 1: RTX-35, 30 m x 0.53 mm ID, 0.5 μ m film thickness or equivalent

4.1.2.2 Column 2: RTX-1701, 30 m x 0.53 mm ID, 0.25 μ m film thickness or equivalent

- 4.1.3 Injection hardware: Restek SILTEK 4 mm gooseneck liner and SILTEK cross seal

4.2 Balances:

4.2.1 Top loader capable of weighing to 0.01 g

4.2.2 Analytical capable of weighing to 0.0001 g

4.3 Vials:

4.3.1 3 dram (12 mL) amber glass vials with Teflon® lined caps

4.3.2 40 and/or 60 mL amber or clear VOA vials with Teflon® lined screw caps

4.3.3 2 mL amber gas chromatograph injection vials with Teflon® lined crimp seals

4.3.4 Scintillation glass vials with polypropylene caps

4.4 Syringes: Gas-tight, various sizes



- 4.5 Disposable glass transfer pipettes and 2 mL rubber bulbs
- 4.6 Separatory funnels: 2 L, 500 mL, and 250 mL with Teflon® stopcock
- 4.7 Glass funnel, 60-90 mm
- 4.8 Turbovap concentrator station, Zymark
 - 4.8.1 Turbovap tubes, 200 mL Zymark or custom
- 4.9 pH indicator paper – wide range pH 1 to 14
- 4.10 Graduated cylinders, various sizes
- 4.11 Drying oven capable of maintaining 105 °C
- 4.12 Muffle oven capable of maintaining 400 °C
- 4.13 Glass wool, VWR Scientific
- 4.14 Compressed Gas
 - 4.14.1 Helium, Grade 5
 - 4.14.2 Nitrogen, Grade 5
- 4.15 Refrigerator capable of maintaining 4 °C
- 4.16 Freezer capable of maintaining temperatures below -15 °C
- 4.17 Repipetter – solvent delivery Optifix
- 4.18 Repeater digital – Eppendorf with appropriate size Combitips

5 REAGENTS

- 5.1 Solvents
 - 5.1.1 Dichloromethane, CH_2Cl_2 – pesticide quality or equivalent
 - 5.1.2 Hexane, C_6H_{14} – pesticide quality or equivalent
 - 5.1.3 Acetone, CH_3COCH_3 – pesticide quality or equivalent
 - 5.1.4 Methanol, CH_3OH – pesticide quality or equivalent
 - 5.1.5 Iso-octane (2,2,4-trimethyl pentane), $\text{CH}_3\text{C}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_3$
 - 5.1.6 Tetradecane – Aldrich 99+%



5.1.7 Deionized water, organic free

5.1.8 Extraction/Final Solvent – 80% Iso-octane/20% Acetone: Combine 3200 mL of Iso-octane with 800 mL of Acetone in a 4 L solvent bottle. Label appropriately and assign a 6 month expiration date.

5.2 Solid Reagents

NOTE: Reagents should be stored in glass once heated.

5.2.1 Sodium sulfate – granular, anhydrous, Na_2SO_4 : Purify by heating to 400 °C for a minimum of 8 hours.

5.2.2 Silica sand: Purify at 400 °C for a minimum of 8 hours.

5.2.3 Sodium chloride, NaCl : Purify by heating to 400 °C for a minimum of 8 hours.

5.2.4 Pre-purified liquid mercury (Hg)

5.3 Acids and Bases

5.3.1 10 N Sodium Hydroxide, NaOH – purchased from VWR

5.3.2 Concentrated sulfuric acid (H_2SO_4), ACS reagent grade or better

5.3.3 12 N Sulfuric Acid: Carefully and slowly add 333 mL of concentrated sulfuric acid to approximately 500 mL of DI water in either a 1 L volumetric flask or graduated cylinder. Use of an ice bath is encouraged to help dissipate the heat generated from this dilution. Allow to cool and make to approximately 1 L with DI water, allowing the solution to cool again. Final room temperature volume is 1 L. Transfer to a 1 L container and store in the refrigerator.

5.4 Stock Standards

5.4.1 Primary stock standards for pesticides mixtures are purchased from Absolute Standards as certified solutions at the following concentrations: 80, 160 and 800 $\mu\text{g}/\text{mL}$. See Table 4 for specific concentrations.

5.4.2 Mix 2 (10/20 $\mu\text{g}/\text{mL}$) standard purchased from Absolute Standards at certified concentrations (see Table 4 for concentrations of individual components).

5.4.3 Toxaphene (1000 $\mu\text{g}/\text{mL}$) is purchased from Absolute Standards or Restek at a certified concentration.

5.4.4 Technical Chlordane (1000 $\mu\text{g}/\text{mL}$) is purchased from Absolute Standards or Restek at a certified concentration.

5.4.5 For Arochlors/PCBs refer to ECCS method 8082.



5.5 Intermediate Standards

5.5.1 A secondary combined stock standard for the individual compounds (Mix A and Mix B separate mixtures) is prepared by diluting 1.25 mL (Use a 1 mL and 250 μ L Gastight syringe) of each primary standard mix (Mix A and B) combined into a 100 mL volumetric flask and made to volume with 80% Iso-octane/20% Acetone. (See Table 5 for concentrations.) Transfer to 40 mL VOA vials that expire one year from preparation date.

5.6 Calibration Standards

5.6.1 Calibration standards are prepared at a minimum of five concentrations by dilution of the secondary stock standard (See Section 5.5.1) in 80% Iso-octane/20% Acetone. A calibration range was chosen to obtain satisfactory limits and a reasonable dynamic range. (See Table 6 Mix 1 for dilution and standard curve range.) Calibration standards expire one year from preparation date.

NOTE: See Figures 1 and 2 for acceptable chromatograms.

NOTE: Standards, once prepared and brought to volume, are transferred to 40 mL VOA vials and stored in a refrigerator when not in use.

5.6.2 Mix 2 Calibration Standards (See Table 6)

NOTE: See Figures 3 and 4 for acceptable chromatograms.

- 5.6.2.1 Level 7 (0.10/0.20 μ g/mL). Using a 1 mL gas tight syringe, aliquot 1 mL of stock (See Section 5.4.2) and using a second volumetric pipette, aliquot 2 mL of surrogate at 6.0 μ g/mL (See Section 5.7.4) into a 100 mL volumetric flask.
- 5.6.2.2 Level 6 (0.05/0.10 μ g/mL). Using a 500 mL gas tight syringe, aliquot 1 mL of stock (See Section 5.4.2) and using a 1 mL gas tight syringe, aliquot 1 mL of surrogate 6.0 μ g/mL (See Section 5.7.4) into a 100 mL volumetric flask.
- 5.6.2.3 Level 5 (0.02/0.04 μ g/mL). Using a 500 mL gas tight syringe, aliquot 400 mL of stock (See Table 4 Mix 2) and using a 1 mL gas tight syringe, aliquot 1 mL of surrogate 6.0 μ g/mL (See Section 5.7.4) into a 100 mL volumetric flask.
- 5.6.2.4 Make each of the above standards to 100 mL with 80% Iso-octane / 20% Acetone.
- 5.6.2.5 Level 4 (0.01/0.02 μ g/mL). Using a 10 mL volumetric pipette, aliquot 10 mL of Level 7 (See Section 5.6.2.1) into a 100 mL volumetric flask.



- 5.6.2.6 Level 3 (0.005/0.01 µg/mL). Using a 10 mL volumetric pipette, aliquot 10 mL of Level 6 (See Section 5.6.2.2) into a 100 mL volumetric flask.
- 5.6.2.7 Level 2 (0.002/0.004 µg/mL). Using a 10 mL volumetric pipette, aliquot 10 mL of Level 5 (See Section 5.6.2.3) into a 100 mL volumetric flask.
- 5.6.2.8 Level 1 (0.001/0.0002 µg/mL). Using a 5 mL volumetric pipette, aliquot 5 mL of the Level 5 (See Section 5.6.2.3) into a 100 mL volumetric flask.
- 5.6.2.9 Make all ICAL standards to 100 mL with 80% Iso-octane / 20% Acetone.
- 5.6.2.10 Transfer all standards to LIMS labeled 40 mL VOA vials, store refrigerated, and assign a one year expiration date.

5.6.3 Toxaphene ICAL

NOTE: See Figures 5 and 6 for acceptable chromatograms

- 5.6.3.1 Level 7 (8.0 µg/mL). Using a 1 mL gas tight syringe aliquot 800 mL of Toxaphene (See Section 5.4.3) and using a 4 mL volumetric pipette, aliquot 4 mL of surrogate mix (6.0 µg/mL, See Section 5.7.4) into a 100 mL volumetric flask.
- 5.6.3.2 Level 6 (4.0 µg/mL). Using a 500 mL gas tight syringe, aliquot 400 mL of Toxaphene (See Section 5.4.3) and using a 2 mL volumetric pipette, aliquot 2 mL of surrogate mix (6.0 µg/mL, See Section 5.7.4) into a 100 mL volumetric flask.
- 5.6.3.3 Level 5 (2.0 µg/mL). Using a 250 mL gas tight syringe, aliquot 200 mL of Toxaphene (See Section 5.4.3) and using a 1 mL gas tight syringe, aliquot 1.0 mL of surrogate mix (6.0 µg/mL, See Section 5.7.4) into a 100 mL volumetric flask.
- 5.6.3.4 Level 4 (1.0 µg/mL). Repeat step 5.6.3.3 except the final volume is 200 mL.
- 5.6.3.5 Level 3 (0.4 µg/mL). Using a 10 mL volumetric pipette, aliquot 10 mL of Level 6 into a 100 mL volumetric flask.
- 5.6.3.6 Level 2 (0.2 µg/mL). Using a 10 mL volumetric pipette, aliquot 10 mL of Level 5 into a 100 mL volumetric flask.
- 5.6.3.7 Level 1 (0.1 µg/mL). Using a 10 mL volumetric pipette, aliquot 10 mL of Level 4 into a 100 mL volumetric flask.
- 5.6.3.8 Make to volume with 80% Iso-octane / 20% Acetone, transfer to 40 mL VOA vials, and place a LIMS label on each vial.



5.6.3.9 Store refrigerated and assign an expiration date of 1 year from preparation date.

5.6.4 Technical Chlordane ICAL

NOTE: See Figures 7 and 8 for acceptable chromatograms.

5.6.4.1 Level 6 (1.0 µg/mL). Using a 100 µL syringe, aliquot 100 µL of 1000 µg/mL Technical Chlordane (See Section 5.4.4) and using a 250 µL syringe, aliquot 125 µL of 200 µg/mL surrogate TCMX/DCBP (See Section 5.7.4) into a 100 mL volumetric flask.

5.6.4.2 Level 5 (0.4 µg/mL). Using a 50 µL syringe, aliquot 40 µL of 1000 µg/mL Technical Chlordane (See Section 5.4.4) and using a 50 µL syringe aliquot 50 µL of 200 µg/mL surrogate TCMX/DCBP (See Section 5.7.4) into a 100 mL volumetric flask.

5.6.4.3 Level 4 (0.2 µg/mL). Repeat Section 5.6.4.2 above except into a 200 mL volumetric flask.

5.6.4.4 Level 3 (0.1 µg/mL). Using a 10 mL volumetric pipette, aliquot 10 mL of Level 6 above into a 100 mL volumetric flask.

5.6.4.5 Level 2 (0.04 µg/mL). Using a 10 mL volumetric pipette, aliquot 10 mL of Level 5 above into a 100 mL volumetric flask.

5.6.4.6 Level 1 (0.02 µg/mL). Using a 10 mL volumetric pipette, aliquot 10 mL of Level 4 above into a 100 mL volumetric flask.

5.6.4.7 Make to volume with 80% Iso-octane/20% Acetone, transfer to 40 mL VOA vials, and place LIMS label on each vial.

5.6.4.8 Store refrigerated and assign an expiration date of one year from preparation date.

5.6.5 PCBs: Refer to Method 8082 for calibration standards.

5.7 Surrogate Spike

5.7.1 Two surrogate stocks are prepared which will accommodate both laboratory situations.

5.7.2 Purchased mixes of TCMX and DCBP at 200 µg/mL each from either Absolute Standards or Restek.

5.7.3 Surrogate Spike Mix – DCBP and TCMX at 2.0 µg/mL

5.7.3.1 Using a 1 mL syringe, aliquot 1 mL of 200 µg/mL stock into a 100 mL volumetric flask. Make to volume with acetone, transfer to 40 mL VOA



vials, and store frozen when not in use. Assign a one year expiration date and affix a LIMS label.

- 5.7.3.2 A 100 μ L aliquot is used to spike samples and quality controls.
- 5.7.4 Surrogate Spike Mix – DCBP and TCMX at 6.0 μ g/mL
 - 5.7.4.1 Using a 1 mL syringe, aliquot 6 times 1 mL of the stock (See Section 5.7.2) into a 200 mL volumetric flask and make to volume with acetone.
 - 5.7.4.2 Transfer to 40 mL VOA vials, affix a LIMS label, store frozen, and assign a one year expiration date.
 - 5.7.4.3 The surrogate spike mix is used for spiking and field work. Spike with 100 μ L when final analysis volume is 10 mL.
- 5.7.5 Different concentrations and spiking volumes are acceptable depending on project specific goals and different final volumes.
- 5.8 LCS Spike Mix
 - 5.8.1 Spiking Solutions: LCS spiking standards are prepared by combining 1.0 mL of each of the stock mixes (Mix A and B) into 100 mL with acetone. A 200 μ L aliquot is usually added for LCSs or MS/MSDs. See Table 7 for concentrations. Transfer to 40 mL VOA vials, affix a LIMS label, store frozen, and assign an expiration date of one year from preparation date.

NOTE: The spiking solution contains surrogate. The concentration in the spiking solution needs to be added to the surrogate level in order to calculate percent recovery for the LCS and MS/MSD.
 - 5.8.2 Mix 2 Spiking Solution
 - 5.8.2.1 Use Stock Standard Mix 2 (See Section 5.4.2).
 - 5.8.2.2 Spiking is usually with 20 μ L of the stock.
 - 5.8.2.3 Alternative spiking Mix 2
 - 5.8.3 Toxaphene Spiking Solution, (100 μ g/mL): LCS/MS/MSD spiking standards are prepared by dilution of 1.0 mL of the purchased stock (See Section 5.4.3) into a 10 mL volumetric flask and made to volume with Acetone. A 0.08 mL aliquot is usually added for LCSs or MS/MSDs. Transfer to a 3 dram amber vial or other appropriate container, store frozen, and assign an expiration date of one year from preparation date.
 - 5.8.4 Technical chlordane (10.0 μ g/mL): LCS/MS/MSD spiking standards are prepared by dilution of 1.0 mL of the purchased stock (See Section 5.4.4) into a 100 mL



volumetric flask and made to volume with Acetone. A 100 μ L aliquot is usually added for LCSs or MS/MSDs. Transfer to 40 mL VOA vials, store frozen, and assign an expiration date of one year from preparation date.

5.8.5 PCBs: Refer to Method 8082 for spiking standards.

5.9 MS/MSD Spike Mix – Same solution as Section 5.8.1 through 5.8.5

5.10 Second Source: No second source standard is used due to this being a method considered as a low volume assay which we receive very few samples each year.

5.11 Internal Standard – Not applicable to this method

5.12 Breakdown Standards – 4, 4'-DDT and Endrin

NOTE: See Figures 9 through 12 for acceptable chromatograms.

5.12.1 Purchase stocks in 1 mL ampoules from Restek or Absolute Standards at 1000 μ g/mL.

5.12.2 Intermediate stock – 20 μ g/mL each DDT and Endrin into separate volumetric flasks.

5.12.2.1 Aliquot 1 mL of Endrin or DDT into separate 50 mL volumetric flasks and make to volume with 80% Iso-octane/20% Acetone. Transfer to 40 mL VOA vials, affix LIMS labels, store frozen, and assign a five year expiration date.

5.12.3 Breakdown injection standards – Endrin/DDT at 0.2 μ g/mL plus surrogate at 0.06 μ g/mL

5.12.3.1 Aliquot 2.0 mL of Endrin and DDT (20 μ g/mL) into separate 200 mL volumetric flasks.

5.12.3.2 Aliquot 2 mL of TCMX and DCBP (6.0 μ g/mL, See Section 5.7.4) into each volumetric flask.

5.12.3.3 Make each standard to volume with 80% Iso-octane/20% Acetone, transfer to 40 mL VOA vials, assign an expiration date of two years, affix LIMS labels, and store refrigerated. With the ICAL, an aliquot of each is transferred to injection vials and injected to check each GC system for degradation determinations.

6 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Water samples should be collected in 1 L amber glass bottles with Teflon® lined caps and stored on ice or refrigerated at 4 °C immediately after collection. In the laboratory, store samples at 4 °C and out of direct sunlight at all times. Water



samples must be extracted within 7 days of collection.

- 6.2 Soil samples should be collected in 4 ounce or larger amber glass jars with Teflon® lined caps. Samples are stored on ice or refrigerated at 4 °C immediately after collection. In the laboratory, store samples at 4 °C and out of direct sunlight at all times. Soil samples must be extracted within 14 days of collection.
- 6.3 Extracts are stored in the freezer and analyzed within 40 days of extraction.
- 6.4 There is no special preservation requirement for soil/water samples. Hold times may be increased for soils to 1 year as long as they are frozen within the 14 day hold time window.
- 6.5 Safety
 - 6.5.1 Recommend use of latex/rubber/nitrile gloves during collection of any and all samples.
 - 6.5.2 Recommend use of latex/rubber/nitrile gloves during weighing of soils and extraction of waters.
 - 6.5.3 If a bad (strong, unpleasant odor) sample is received, do not oven dry the sample for total solids in the open lab, but place the oven in the hood and/or do not dry the sample for total solids for the bad sample.
 - 6.5.4 Dichloromethane vapors are considered hazardous. Therefore, in order to minimize exposure, all separatory funnel extraction steps should be done in a hood.
 - 6.5.5 Mercury is considered a very hazardous material. Care must be exercised in its use and avoid spills at all costs. Cleanup of any spill is mandatory. ECCS maintains a mercury waste container for any spills and/or waste once used for sulfur treatments.

7 PROCEDURE

NOTE: If a sample requires 8141MS and 8081 assays, only one extract is needed for either soils or waters. Surrogates for both assays are added to unknown samples and method blanks, but a separate LCS and MS/MSD will be required for both the 8141MS and 8081 assays. Co-elution of target compounds from both assays occurs on the GC/ECD instrument. The addition of sodium chloride to aqueous samples prior to extraction is only done if the samples need to be analyzed for the 8141 compounds DEA or DIA. Please see the 8141MS method for specific details as samples are extracted according to the 8081 method.

7.1 Water Samples

- 7.1.1 Transfer the contents of the 1 L bottle as received (mark the water level on the



bottle prior to transferring) into a 2 L separatory funnel.

7.1.1.1 At a convenient time, refill the sample bottle to the mark with water, pour into a 1 L graduated cylinder and record the volume of sample extracted on the extraction log page. This step is done after the sample container has been rinsed in step 7.1.5.

7.1.1.2 For samples that have a high amount of particulate, the water may be decanted leaving the solid material in the sample bottle. Make sure to mark sample containers to obtain the volume extracted.

NOTE: Sample containers with a sediment layer are not rinsed with the extraction solvent.

7.1.2 Check the pH of the sample with wide-range pH paper and, if necessary, adjust the pH to approximately pH 7 using 12 N sulfuric acid or 10 N sodium hydroxide.

NOTE: Acceptable pH is 5 to 9.

7.1.3 Add 100 μL of the surrogate standard (TCMX and DCBP at 2.0 $\mu\text{g}/\text{mL}$, See Section 5.7.3) to all samples, blanks, and LCS/MS/MSDs.

7.1.4 For the sample in each analytical batch selected for MS, MSD, and LCS add 200 μL of the matrix spiking standard (See Section 5.8.1). See Table 7 for concentrations.

NOTE: The mixed standard is usually added to the MS, MSD, and LCS unless other analytes are required.

NOTE: Spiking LCS and MS/MSD may be done with one of the multi-component mixtures (i.e. toxaphene, technical chlordane, or PCBs; see Sections 5.8.2, 5.8.3, 5.8.4, or 5.8.5 respectively) instead of the individual component mix.

7.1.5 Rinse the bottle once with a 60 mL aliquot of dichloromethane and transfer the dichloromethane to the separatory funnel.

NOTE: Do not rinse the sample container if water was decanted and sediment remains in the sample container. If this occurs, add the 60 mL aliquot of dichloromethane directly to the sample in the separatory funnel and proceed to 7.1.6.

7.1.6 Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

NOTE: Dichloromethane creates excessive pressure very rapidly; therefore, initial venting should be done immediately after separatory funnel has been sealed and inverted. The separatory funnel should be vented into a hood to avoid



exposure to solvent vapors.

- 7.1.7 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. The extract is filtered through a funnel containing glass wool and 15 to 20 grams of sodium sulfate, into a 200 mL Zymark collection tube. Rinse the sodium sulfate with approximately 10 mL of dichloromethane after each of the 3 extracts is drained through the funnels.
- 7.1.7.1 If an emulsion forms in the separatory funnel, the following steps can be used to break it up.
- 7.1.7.2 If there is a very small emulsion present, this may be drained into the sodium sulfate. Mix the sodium sulfate with a disposable pipette to prevent the sodium sulfate from clumping and rinse as above.
- 7.1.7.3 If a small emulsion forms, many times these can be broken with swirling the separatory funnel or mixing the organic layer with a long glass rod or wood swizzle stick being careful not to damage the separatory funnel or the glass rod.
- 7.1.7.4 If there is an emulsion greater than 1/3 of the organic volume, drain the extract into a second dichloromethane rinsed 250 mL or larger separatory funnel. Stopper the second separatory funnel and shake vigorously. This will usually break the emulsion. If not, add more dichloromethane and shake vigorously. Keep adding and shaking until the emulsion breaks. Allow layers to settle and drain the dichloromethane layer through the filter funnel into the Zymark tube. Because some water still exists in the second separatory funnel, each subsequent 60 mL aliquot must be transferred to the second separatory funnel, and shaken before transferring into Zymark through the funnel.
- 7.1.8 Repeat the extraction two more times using a new 60 mL aliquot of dichloromethane (Sections 7.1.5 through 7.1.7). Combine the extracts in the Zymark collection tube.

NOTE: Rinse the funnel containing the sodium sulfate with dichloromethane after each extract has passed through into the Zymark tube.

- 7.1.9 Samples are now ready for concentration. Proceed to Step 7.3.

7.2 Soil/Solid Samples

- 7.2.1 Prepare a minimum of one blank and one LCS with each set of soil samples extracted. If more than 20 samples are to be extracted, prepare a new blank and LCS with each group of 20 or less samples. Use 5 grams of heated silica sand for the blank and LCS.
- 7.2.2 For soil samples, weigh 5 grams into a scintillation vial using a top loader and



select one of the samples to be the parent sample of the MS and MSD. In this case you will weigh the sample in triplicate, one for the sample, and one each for the MS and MSD.

NOTE: Sample should be mixed as thoroughly as possible prior to weighing. Eliminate stones and other debris from the sample jar so as to not include these types of material into the 5 gram sample weight.

- 7.2.3 Add 5 to 10 grams of sodium sulfate to each unknown or quality control sample and mix thoroughly with a spatula to dry the sample.

NOTE: This procedure may require a lot of effort if the samples are wet silts or clays. The sample must be totally dry prior to addition of extraction solvent (80% Iso-octane/20% Acetone). Failure to thoroughly dry the sample will negatively affect the recovery of target compounds.

NOTE: Addition of more sodium sulfate is acceptable and, if needed (due to sample being wet), the sample may be transferred to a 40 mL or 60 mL VOA vial. This may require addition of 20 mL of extraction solvent instead of 10 mL. Double the amount of surrogate added if increasing the extraction solvent volume to 20 mL.

NOTE: Sample must be a free flowing powder prior to proceeding with the extraction.

- 7.2.4 Add 100 μL of the surrogate standard solution (TCMX and DCBP at 2.0 $\mu\text{g}/\text{mL}$, (See Section 5.7.3) to each unknown or quality control sample.

NOTE: Amount of surrogate sample and concentration may be adjusted based on differing requirements.

- 7.2.5 For the MS/MSD sample in each analytical batch, prepare as above and add 200 μL of the Intermediate spiking standard to each MS/MSD sample (See Section 5.8.1).

- 7.2.6 For the LCS sample in each analytical batch, add 200 μL of the spiking standard solution (See Section 5.8.1).

NOTE: The mixed standard is usually added to the MS, MSD, and LCS unless other analytes are required.

NOTE: Spiking LCS and MS/MSD may be done with one of the multi-component mixtures (i.e. toxaphene) instead of the individual component mix.

- 7.2.7 Using a calibrated repipet, add 10 mL of 80% Iso-octane/20% Acetone to each vial and cap.



NOTE: Check pipette with 10 mL volumetric prior to sample addition. If not 10 mL, make adjustments and re-check being sure to use a dry volumetric for each check.

- 7.2.8 Shake sample vigorously for a minimum of 30 seconds and allow to settle for 5 minutes. Repeat shake for an additional 30 seconds and allow to settle for an additional 5 minutes.
- 7.2.9 Extract is stored in the 20 mL scintillation vial and stored in the extracted sample freezer by work order number.

NOTE: Sample extract may be transferred to a 3 dram vial at the discretion of the analyst.

- 7.2.10 Further cleanup may be considered following initial injection (See Section 7.4).
- 7.2.11 Samples are now ready for injection. Proceed to step 7.5.

7.3 Concentration and Transfer

- 7.3.1 To each sample prior to concentration, add 8 drops of tetradecane and approximately 8 mL of Iso-octane.
- 7.3.2 Set the Turbovap to 30 °C with nitrogen flowing at 10 to 15 PSI.
- 7.3.3 Evaporate the dichloromethane in the Turbovap until about 10 to 12 mL of solvent remains.
- 7.3.4 Add 30 mL of hexane to each tube and evaporate again to 5 to 7 mL of solvent. This is a solvent exchange to remove most of the dichloromethane.
- 7.3.5 Quantitatively transfer the extract to a 10 mL volumetric flask, rinsing the Zymark tube with several small portions of 80% Iso-octane/20% Acetone and make to volume with 80% Iso-octane/20% Acetone.
- 7.3.6 The sample is now ready to inject. Further cleanup will be considered following the initial injection (See Section 7.4). Proceed to step 7.5.

7.4 Cleanup Procedures (performed only if necessary)

- 7.4.1 Cleanup procedures are not necessary for relatively clean samples, 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. General guidance for sample extract cleanup is provided in this section and in EPA Method 3600.
- 7.4.2 If only PCBs and/or toxaphene are to be measured in the sample, concentrated



sulfuric acid cleanup (Method 3665) is recommended. Additional cleanup/fractionation by Alumina Cleanup (Method 3610), Silica-Gel Cleanup (Method 3630), or Florisil Cleanup (Method 3620), may be necessary.

NOTE: If acid cleanup is used for PCB or Toxaphene analysis the final result needs to be multiplied by a factor of 0.8. The final solvent is 80% Iso-octane/20% Acetone and once treated with sulfuric acid, only the Iso-octane remains, which concentrates the sample extract.

- 7.4.3 If both PCBs and pesticides are to be measured in the sample, isolation of the PCB fraction by Silica Cleanup (Method 3630) may be required.
- 7.4.4 If only pesticides are to be measured in the sample, isolation of the PCB fraction by Silica Cleanup (Method 3620 or 3630) is recommended.
- 7.4.5 Elemental sulfur, which may appear in certain sediments and industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur is removed from the extract with the use of liquid mercury.
 - 7.4.5.1 Sulfur is identified in the chromatogram by a large asymmetrical peak starting at about the elution time of Heptachlor epoxide and running out to the elution time of DDT on the RTX-35 column. It may be smaller and represent only a hump. On the RTX-1701 column, sulfur appears around the retention times of BHCs and heptachlor.

NOTE: It is possible that a cleanup step for sulfur may not be needed due to different retention times on the two analytical columns.

NOTE: Liquid mercury is toxic and should always be handled with great care. Use of a hood is recommended, and cleanup of any mercury spills is done immediately. Always place samples and mercury to be added to samples in a secondary container to contain any possible spills.

- 7.4.5.2 Transfer approximately 3 to 4 mL of sample extract to a 10 to 12 mL vial with a Teflon® lined cap.
- 7.4.5.3 Add a small amount of liquid mercury to each sample to be cleaned up. The amount of mercury is usually 4 to 10 drops using a disposable pipette.

NOTE: Mercury is considered a very hazardous chemical and therefore the handling should be done to minimize any and all spillage. Use of containment traps under the mercury container and doing all transfers within the containment tray is a must.

- 7.4.5.4 Cap vial and shake vigorously for 2 to 5 minutes. Repeat shaking as necessary. A black precipitate will begin to form if sulfur is present. Allow to settle and shake again for another 2 to 5 minutes. Allow to settle and



check for the presence of metallic liquid mercury. If present this step is done, if not, add more mercury and repeat above. There needs to be visible liquid mercury present to make sure all the sulfur has been removed.

- 7.4.5.5 Mercury treated sample extracts must be disposed of as follows. The extracts are poured into a container labeled mercury waste. This includes the black powder formed and the remaining liquid mercury in the sample. This is all done under a hood, and once the extracts are transferred, leave the cap off the waste container until all the solvent evaporates. Cap waste container and store in the hood. The mercury waste container can be properly disposed of at the City of Madison Clean Sweep Program for small businesses.

7.5 Instrument Conditions

7.5.1 Gas Chromatograph #1 RTX 35 Column (See Section 4.1.2.1)

Temperature Program

Initial Temp:	140 °C
Initial Hold:	1.0 min
Initial Rate:	8 °C/min
Final Temp (1):	340 °C
Hold Time (1):	2.0 min

Injector Temp:	250 °C
Detector Temp:	320 °C
Carrier Gas:	Helium
Head Pressure:	~7 PSI

Make up Gas:	Nitrogen
Flow Rate:	~30 mL/min

Split Vent:	~28 mL/min
Septum Purge:	< 1.0 mL/min
Equilibration Time:	1.50 min
Oven Max:	350 °C
Split-less Valve:	Split-less box not checked
On time:	0.5 min
Off time:	3.00 min

NOTE: All the above parameters are set to obtain separation of all the compounds in the current mixed standard (except α -chlordane and Endosulfan I) and to minimize breakdown of DDT and Endrin.



7.5.2 Gas Chromatograph #2 RTX-1701 Column (See Section 4.1.2.2)

Temperature Program

Initial Temp: 140 °C
Initial Hold: 1.0 min
Initial Rate: 5 °C/min
Final Temp: 280 °C
Hold Time: 2 min

Injector Temp: 250 °C
Detector Temp: 300 °C
Carrier Gas: Helium
Head Pressure: ~7 PSI

Make up Gas: Nitrogen
Flow Rate: ~30 mL/min

Split Vent: ~28 mL/min
Septum Purge: < 1.0 mL/min
Equilibration Time: 1.50 min
Oven Max: 285 °C
Split-less Valve: Split-less box not checked
On time: 0.5 min
Off time: 3.00 min

NOTE: Final temp for RTX-1701 columns needs to be strictly adhered as even programming above 280 °C causes rapid column degradation.

NOTE: All the above parameters are set to obtain separation of all the compounds in the current mixed standard and to minimize breakdown of DDT and Endrin.

NOTE: All the above chromatographic parameters may be modified to obtain acceptable chromatography and separations.

NOTE: The chromatograms in Figure 1 and Figure 2 portray the acceptable peak shape, peak resolution, compound response, and compound response ratios that are required on both chromatography columns for the analysis of standards and samples to proceed.

7.6 Preventive Maintenance

7.6.1 Routine evaluation of the DDT and Endrin breakdown is critical prior to starting any run. In all cases the system must have breakdowns of < 2% before starting a run.

7.6.2 Routine maintenance consists of clipping the column, replacing the Siltek® liner



and seal, cleaning the hat, and installing a new septum.

7.6.3 Changing only the septum is okay as long as the breakdowns are acceptable from Section 7.6.1.

7.6.4 Carryover on the GC is usually caused by a worn out injection syringe. When replacing the syringe, use fine sandpaper to file off the sharp point and/or burr. Rinse with solvent from the top to remove any grit left in the syringe tip.

7.7 Calibration

7.7.1 For each compound of interest, prepare a calibration curve with a minimum of 5 concentrations from the 8 available (See Table 6). One of the standards should be at a concentration near, but above, the method detection limit. The other standards should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

NOTE: The lowest standard used for soils and waters is L-1 for any of the standard curves in Section 5.6.

7.7.2 Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph. Plot the peak height or area responses against the concentration of the calibration standard using power regression. The coefficient of determination (R^2) for each compound should be > 0.995 or a correlation coefficient of > 0.9975 .

NOTE: Peak heights are normally used for all calculations.

$$\text{Regression: } Y = AX^B \text{ or } \ln Y = B \ln X + \ln A$$

Where: Y = Peak height
X = Concentration in $\mu\text{g/mL}$
A = Constant
B = Exponent

7.7.3 The working calibration curve is verified on each working day by the injection of one or more continuing calibration verification standards. If the response for any compound varies from theory by more than $\pm 20\%$, a new calibration curve must be injected, and/or data is qualified.

$$\text{Percent Difference} = \frac{R2 - R1}{R1} \times 100$$

Where: R1 = Theoretical concentration
R2 = Concentration from CCV



7.7.4 Method 8081 has several multi-component mixtures as target analytes. For this reason, the choice of target analytes for calibration should be limited to those specified in the project plan. Sites may require analysis for the organochlorine pesticides only or the PCBs only. Toxaphene and/or technical chlordane may also be specified at certain sites. In addition, where PCBs are specified in the project plan, injection of Aroclors 1016 and 1260 may suffice for the initial calibration of all Aroclors since they include most of the congeners present in the different regulated Aroclors.

Note: If target analytes include these multi-component analytes, inject a mid level standard with the ICAL. If not present, no further action needs to be taken. If present, either re-inject samples with a full calibration curve or report value as estimated based on a single point calibration.

NOTE: Exception to “E” qualifier in PCBs as single point ICALs is acceptable as long as one of the mixes has a full ICAL.

7.8 Retention Time Windows

7.8.1 Retention times obtained from the ICAL are put into the calibration file and used throughout the set.

7.8.2 The ICAL retention times once established, may be adjusted as processing the data occurs using CCVs, LCSs and MS/MSDs for reference retention times. Normally windows are established for the surrogate TCMX/DCBP at approximately 0.10 minutes. The surrogates are set as reference peaks and as such the rest of the peaks’ retention times will shift with the surrogate on an injection-by-injection basis.

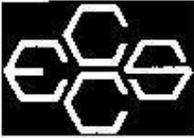
7.8.3 Retention windows for the other target analytes are usually set between 0.04 and 0.06 minutes.

7.9 Sample Analysis

7.9.1 Set up the GC system using the conditions described in Section 7.5.

7.9.2 Samples are analyzed in a set referred to as a GC run. The sequence usually begins with a standard curve (ICAL) followed by sample and quality control sample extracts interspersed with continuing calibration verification (CCV) standards. CCVs must be injected every 10 samples or less and at the end of the run. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded. Raw data is archived by run number (i.e. GC-43 is a run number). A run number is established from the master GC Run Logbook and is sequential.

NOTE: Run an initial calibration verification (ICV) immediately after the ICAL to verify the ICAL.



- 7.9.3 A portion of the standards, samples, and QC are all transferred using disposable pipets into properly labeled injection vials.

NOTE: Standard curves use 300 μ L inserts in the injection vials to preserve ICAL standard solutions.

- 7.9.4 Injection – Inject approximately 2 μ L of the sample. The data system will record the resulting peak size for each compound in peak height.
- 7.9.5 If any of the responses exceeds the theoretical value of the highest standard, dilute the extract and re-inject. Dilute the sample so that all peaks are on scale but in the upper half of the standard curve range, if possible. Overlapping peaks are not always evident when peaks are off scale. Peak height measurements are used for all quantitation.

NOTE: If during analysis a compound dilution is missed or has been diluted excessively, the result may be reported with an “E” qualifier for exceeding the upper calibration curve standard.

- 7.9.6 If peak detection is prevented by the presence of interferences, further cleanup may be required (e.g. mercury).
- 7.9.7 Tentative identification of a compound occurs when a peak from a sample extract falls within the retention time window. Tentative confirmation of that compound occurs when a peak from a sample extract falls within the retention time window for the second analytical column. Confirmation is absolute when the ~~40~~ concentrations of the extract on both analytical columns agree within ~~80~~ % of its highest value. If both columns show presence of a compound, always report the lower number if the difference is greater than ~~50~~%. If one column has a positive result and the other one doesn't, the result is not confirmed and the compound is determined to be not present.

090
DIFFERENCE
$$\frac{V_1 - V_2}{\frac{V_1 + V_2}{2}} \times 1000$$

change ok 29 APR 08

- 7.9.8 Multi-component mixtures (i.e. PCBs) are reviewed on a sample-by-sample basis, making sure to always be looking to identify them if present. If the analyte (multi-component mixture) is required, inject at a minimum a mid-level standard with the run for each multi-component mixture to be reported.

NOTE: Report with an “E” qualifier, for the single point ICAL if a multi-point ICAL is not run

- 7.9.9 Complete Data Package/Run

- 7.9.9.1 Copy of computer generated run sequence file
- 7.9.9.2 Copy of the acquisition method
- 7.9.9.3 Copy of the calculation method, if applicable



- 7.9.9.4 Copy of the calibration file including regression graphs, with dropped standards identified and reason for not using and initial and date. Initial and date on the front page plus acceptable standard curve noted as OK.
- 7.9.9.5 Filled out GC information sheet (See Figure 5)
- 7.9.9.6 Complete chromatographs for every injection.
 - 7.9.9.6.1 Initial and date every chromatograph injection.

NOTE: Any explanations needed to explain anomalies, data not used, or any explanations needed to clarify reported results must be on each affected chromatogram.
 - 7.9.9.6.2 Mark confirmed (c) or not confirmed (nc) for every compound with a positive detect on both quantitation and confirmation columns.
 - 7.9.9.6.3 Include any other comments used to explain each chromatogram (i.e. dropped standard, bad chromatograph, data not used, bad injection) and any other words needed to explain what was used.
- 7.9.9.7 All of the above is required for both the quantitation and confirmation columns.

7.10 Calculations

- 7.10.1 The concentration of each compound in the sample extract injected is determined by reverse extrapolation from the peak height response in the sample using power regression analysis to obtain concentration of each analyte in $\mu\text{g/mL}$. The concentration in $\mu\text{g/L}$ or $\mu\text{g/g}$ of each of the target compounds in the sample is then calculated as follows:

$$\text{Concentration } (\mu\text{g/L}) = \frac{A_x \times D \times V_e}{V_s}$$

Where: A_x = Concentration of compound in the extract in $\mu\text{g/mL}$
 D = Dilution factor, if applicable
 V_e = Volume of extract in mL
 V_s = Volume of sample extracted in Liters

$$\text{Concentration } (\mu\text{g/g}) = \frac{A_x \times D \times V_e}{W_s}$$

Where: A_x = Concentration of compound in the extract in $\mu\text{g/mL}$
 D = Dilution factor, if applicable
 V_e = Volume of extract in mL
 W_s = Weight of sample extracted in g



8 QUALITY CONTROL

- 8.1 Refer to Method 8000 in SW-846 for general GC quality control procedures.
- 8.2 Include a mid-level continuing calibration verification standard (CCV) after each group of 10 or less injections in the analysis sequence and at the end of the run. Control limits are $\pm 20\%$.
- 8.3 Method blanks consist of an aliquot of laboratory reagent water or sand prepared and processed through every step of the process. Method blanks must be free of target compounds and if present, $<50\%$ of the low standard. If contamination exists, the samples (including quality control samples) should be re-extracted once the problem is eliminated. Method blanks are analyzed on a 1 per 20 or less basis with a minimum of one per day.

NOTE: If re-analysis is not possible and the compound found in the blank is identified in a sample, the data must be qualified.

- 8.4 Laboratory control samples (LCS) consist of an aliquot of laboratory reagent water or sand spiked with the target compounds, prepared and processed through every step of the process. If the recovery of any of the target compounds is outside control limits of $\pm 40\%$ recovery from theory, the samples (including quality control samples) should be re-extracted.
- 8.5 Matrix spike/matrix spike duplicate (MS/MSD) samples consist of duplicate aliquots of sample spiked with the target compounds, prepared and processed through every step of the extraction process. MS/MSD control limits are 60 to 140% recovery, with a 20% RPD assuming the sample does not contain any of the target analytes.

$$RPD = \frac{R1 - R2}{\left(\frac{R1 + R2}{2}\right)} \times 100$$

Where: R1 = MS Result
R2 = MSD Result

- 8.6 DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner becomes contaminated with high boiling residue from sample injections or when the injector contains metal fittings or filings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and a standard containing only Endrin. Presence of 4,4'-DDE; 4,4'-DDD; Endrin ketone; or Endrin aldehyde indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration/analysis of the GC run.



8.6.1 Calculate percent breakdown as follows:

$$\% \text{ breakdown for 4,4' DDT} = \frac{\text{Total DDT degradation peak heights for (DDE + DDD)}}{\text{peak height (DDT + DDE + DDD)}} \times 100$$

$$\% \text{ breakdown for Endrin} = \frac{\text{Total Endrin degradation peak heights (aldehyde + ketone)}}{\text{peak height (Endrin + aldehyde + ketone)}} \times 100$$

8.6.2 The breakdown of DDT and Endrin should be measured before samples are analyzed and at the beginning of each 12 hours of the run and at the end of the analysis set. Injector maintenance and re-calibration should be completed if the breakdown is greater than 15% for either compound (See Section 8.6.1).

8.7 Calculate surrogate standard recovery on all samples, blanks, and spikes. The control limits for surrogates are 60 to 140%, with only 1 of the 2 surrogates required to be within this range. If both surrogates are out:

8.7.1 Confirm that there are no errors in calculations, surrogate solutions and check instrument performance.

8.7.2 Examine chromatograms for interfering peaks and for integrated peak heights.

8.7.3 Recalculate the data and/or re-analyze the extract if any of the above checks reveals a problem.

8.7.4 Re-extract and re-analyze the sample if none of the above are a problem or flag the data as estimated concentration with an "E" qualifier.

8.8 Whenever silica gel (Method 3630) or Florisil (Method 3620) cleanup is used, demonstrate that the fractionation scheme is reproducible. Batch to batch variation in the composition of the silica gel material or overloading the column may cause a change in the distribution patterns of the organochlorine pesticides and PCBs. When compounds are found in two fractions, add the concentrations in the fractions, and be sure to correct for any additional dilution.

9 METHOD PERFORMANCE

9.1 Estimated MDLs for eight replicates of silica sand and water are listed in Table 1.

9.2 Performance data for four replicates (IDCs) of silica sand and water spiked at an LCS concentration are given in Table 2.

10 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

10.1 Contingencies for out-of-control data should be evaluated on a case-by-case basis. A Corrective Action Form (CAF) must be completed for those times that acceptable QC results cannot be achieved. The CAF must be completed by the analyst and filed with the Quality Manager. Analytical results shall be qualified as necessary.



11 WASTE MANAGEMENT / POLLUTION PREVENTION

- 11.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.

12 REFERENCES

- 12.1 SW 846, Method 8081A, Update III Revision 0, December 1996.
- 12.2 SW 846, Method 3570, Micro-scale Extraction (MSE), Revision 0, November 2002.



TABLE 1
METHOD 8081 MDLS
ORGANOCHLORINE PESTICIDES

Compound	Water ($\mu\text{g/L}$)¹	Soil ($\mu\text{g/kg}$)²
α -BHC	0.00083	0.21
γ -BHC (Lindane)	0.00071	0.20
β -BHC	0.00068	0.31
δ -BHC	0.0010	0.23
Heptachlor	0.00068	0.18
Aldrin	0.00092	0.23
Heptachlor epoxide	0.00092	0.26
γ -Chlordane	0.00062	0.26
α -Chlordane	0.00094	0.25
4,4'-DDE	0.0018	0.48
Endosulfan I	0.00045	0.25
Dieldrin	0.0017	0.47
Endrin	0.0023	0.46
4,4'-DDD	0.0029	0.65
Endosulfan II	0.0019	0.58
4,4'-DDT	0.0015	0.47
Endrin aldehyde	0.0019	0.61
Methoxychlor	0.010	2.3
Endosulfan sulfate	0.0015	0.48
Endrin ketone	0.0019	0.50

NOTES: (1) MDL Data, GC-1297 03/07/06 RTX-1701 Column
(2) MDL Data, GC-1235 12/30/05 RTX-1701 Column



TABLE 1 (CONTINUED)
METHOD 8081 MDL'S
MULTI-COMPONENT COMPOUNDS

Compound	Column	Water Concentration (µg/L)	Soil Concentration (µg/kg)
Toxaphene	RTX-35	0.36 ¹	51 ¹
Toxaphene	RTX-1701	0.16 ¹	33 ¹
Technical Chlordane	RTX-200	NR	9.7 ²
Technical Chlordane	RTX-XLB	NR	12 ²
Aroclor 1254	RTX-35	0.183 ³	18.4 ³
Aroclor 1254	RTX-1701	0.256 ³	8.0 ³

NOTES: (1) GC-1297, 03/08/06
(2) GC-1071, 02/11/05
(3) GC-1307, 03/15/06

NR = Not run



TABLE 2

8081
TYPICAL IDCS NEED TO RUN



TABLE 3

TYPICAL RETENTION TIME (IN MINUTES) OF CHLORINATED PESTICIDES BY GC MIX 1

Compound	RTX-35	RTX-1701
TCMX (surrogate)	7.386	7.791
α -BHC	9.292	11.168
γ -BHC (Lindane)	10.365	12.561
β -BHC	10.596	15.538
Heptachlor	11.430	13.097
δ -BHC	11.558	16.051
Aldrin	12.314	13.888
Heptachlor epoxide	13.764	16.295
γ -Chlordane	14.267	17.404
α -Chlordane ¹	14.687	17.612
Endosulfan I ¹	14.687	17.051
4,4'-DDE	15.468	18.046
Dieldrin	16.331	18.834
Endrin	16.632	18.834
4,4'-DDD ¹	16.790	20.574
Endosulfan II ¹	16.790	20.478
4,4'-DDT	17.409	21.003
Endrin aldehyde	17.542	21.812
Endosulfan sulfate	17.944	22.825
Methoxychlor	19.357	23.080
Endrin ketone	19.681	23.837
DCBP (Surrogate)	22.687	26.528

NOTES: (1) Co-elute on RTX-35
(2) See Figures 1 and 2



TABLE 3 CONTINUED

TYPICAL RETENTION TIMES (IN MINUTES) OF CHLORONATED PESTICIDES BY GC
MIX 2

Compound	RTX-35	RTX-1701
Hexachlorobenzene	8.881	9.457
Pentachloronitrobenzene	10.077	11.594
2,4'-DDE	14.583	16.970
trans – Nonachlor	14.422	17.732
2,4'-DDD	15.824	19.059
2,4'-DDT	16.614	19.395
cis – Nonachlor	16.497	20.604
Mirex	19.863	21.880

NOTE: (1) See Figures 3 and 4



TABLE 4
8081
STOCK STANDARD CONCENTRATIONS

Mix A	µg/mL
Tetrachloro-m-xylene (TCMX)	80
α-BHC	80
γ-BHC (Lindane)	80
Heptachlor	80
Endosulfan I	80
Dieldrin	160
Endrin	160
4,4'-DDD	160
4,4'-DDT	160
Methoxychlor	800
Decachlorobiphenyl (DCBP)	160

Mix B	µg/mL
Tetrachloro-m-xylene (TCMX)	80
β-BHC	80
δ-BHC	80
Aldrin	80
Heptachlor epoxide	80
α-Chlordane	80
γ-Chlordane	80
4,4'-DDE	160
Endosulfan II	160
Endosulfan sulfate	160
Endrin aldehyde	160
Endrin ketone	160
Decachlorobiphenyl (DCBP)	160

Mix 2	µg/mL
Hexachlorobenzene	10
Pentachloronitrobenzene (PCNB)	10
cis-Nonachlor	10
trans-Nonachlor	10
Mirex	20
2,4'-DDD	20
2,4'-DDE	20
2,4'-DDT	20



TABLE 5

INTERMEDIATE ICAL SOLUTION

Compound	Concentration (µg/mL)
TCMX (surrogate)	2.0
α-BHC	1.0
γ-BHC (Lindane)	1.0
β-BHC	1.0
Heptachlor	1.0
δ-BHC	1.0
Aldrin	1.0
Heptachlor epoxide	1.0
γ-Chlordane	1.0
α-Chlordane	1.0
Endosulfan I	1.0
4,4'-DDE	2.0
Dieldrin	2.0
Endrin	2.0
4,4'-DDD	2.0
Endosulfan II	2.0
4,4'-DDT	2.0
Endrin aldehyde	2.0
Endosulfan sulfate	2.0
Methoxychlor	10.0
Endrin ketone	2.0
DCBP (Surrogate)	4.0

NOTES: (1) Aliquot 1.25 mL of individual Mix A and Individual Mix B (See Section 5.2.1) into a 100 mL volumetric flask.

(2) Solvent: 80% Iso-octane/20% Acetone



TABLE 6
 8081
 PREPARATION OF ORGANOCHLORINE PESTICIDE ICAL

Compound	CONCENTRATION ($\mu\text{g/mL}$)							
	L-1	L-2	L-3	L-4	L-5	L-6	L-7	L-8
TCMX (surrogate)	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
α -BHC	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
γ -BHC (Lindane)	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
β -BHC	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
δ -BHC	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
Heptachlor	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
Aldrin	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
Heptachlor epoxide	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
γ -Chlordane	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
α -Chlordane	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
4,4'-DDE	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
Endosulfan I	0.001	0.002	0.005	0.008	0.01	0.02	0.05	0.1
Dieldrin	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
Endrin	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
4,4'-DDD	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
Endosulfan II	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
4,4'-DDT	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
Endrin aldehyde	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
Methoxychlor	0.01	0.02	0.05	0.08	0.1	0.2	0.5	1.0
Endosulfan sulfate	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
Endrin ketone	0.002	0.004	0.01	0.016	0.02	0.04	0.1	0.2
DCBP (Surrogate)	0.004	0.008	0.02	0.032	0.05	0.08	0.2	0.4

	L-1	L-2	L-3	L-4	L-5	L-6	L-7	L-8
mL Stock ¹	10	10	10	8	2	2	5	10
Stock Concentration	L-5	L-6	L-7	L-8	Table 5	Table 5	Table 5	Table 5
Final Volume (mL)	100	100	100	100	200	100	100	100

NOTES: (1) Use volumetric pipettes
 (2) Solvent: 80% Iso-octane/20% Acetone



TABLE 6 (CONTINUED)

8081 MIX 2
 PREPARATION OF ORGANOCHLORINE PESTICIDE ICAL MIX 2

Compound	CONCENTRATION ($\mu\text{g}/\text{mL}$)						
	L-1	L-2	L-3	L-4	L-5	L-6	L-7
Hexachlorobenzene (HCB)	0.001	0.002	0.005	0.01	0.02	0.05	0.10
Pentachloronitrobenzene (PCNB)	0.001	0.002	0.005	0.01	0.02	0.05	0.10
cis – Nonachlor	0.001	0.002	0.005	0.01	0.02	0.05	0.10
trans – Nonachlor	0.001	0.002	0.005	0.01	0.02	0.05	0.10
MIREX	0.002	0.004	0.01	0.02	0.04	0.10	0.20
2,4' – DDE	0.002	0.004	0.01	0.02	0.04	0.10	0.20
2,4' – DDD	0.002	0.004	0.01	0.02	0.04	0.10	0.20
2,4' – DDT	0.002	0.004	0.01	0.02	0.04	0.10	0.20
TCMX	0.0012	0.003	0.006	0.012	0.03	0.06	0.12
DCBP	0.0012	0.003	0.006	0.012	0.03	0.06	0.12

	L-1	L-2	L-3	L-4	L-5	L-6	L-7
Aliquot (mL)	5	10	10	10	0.4	0.5	1.0
Stock ID	L-5	L-5	L-6	L-7	Step 5.5.1	Step 5.5.1	Step 5.5.1
Final Volume	100	100	100	100	200	100	100
Surrogate Aliquote (mL)					1.0	1.0	2.0
Surrogate stock ID					Step 5.7.4	Step 5.7.4	Step 5.7.4



TABLE 6 (CONTINUED)

TOXAPHENE PREPARATION OF ICAL

Compound	CONCENTRATION ($\mu\text{g}/\text{mL}$)						
	L-1	L-2	L-3	L-4	L-5	L-6	L-7
Volume stock (mL)	10	10	10	200	200	400	800
Stock Standard ID	L-4	L-5	L-6	Step 5.4.3	Step 5.4.3	Step 5.4.3	Step 5.4.3
Surrogate Stock ID	-	-	-	Step 5.7.4	Step 5.7.4	Step 5.7.4	Step 5.7.4
Volume Surrogate (μL)	-	-	-	1.0	1.0	2.0	4.0
Final Volume	100	100	100	200	100	100	100
Conc Toxaphene	0.10	0.20	0.40	1.0	2.0	4.0	8.0
Conc Surrogate	0.003	0.006	0.012	0.03	0.06	0.12	0.24

NOTE: (1) Final Solvent: 80% Iso-octane/20% Acetone

TABLE 6 (CONTINUED)

TECHNICAL CHORDANE PREPARATION OF ICAL

Compound	L-1	L-2	L-3	L-4	L-5	L-6
Volume stock (mL)	10	10	10	40	40	100
Stock Standard ID	L-4	L-5	L-6	Step 5.4.4	Step 5.4.4	Step 5.4.4
Surrogate Stock ID	-	-	-	Step 5.7.2	Step 5.7.2	Step 5.7.2
Volume Surrogate (μL)	-	-	-	50	50	0.125
Final Volume	100	100	100	200	100	100
Conc Technical Chlordane	0.02	0.04	0.1	0.2	0.4	1.0
Conc Surrogate	0.005	0.01	0.025	0.05	0.10	0.25

NOTE: (1) Final Solvent 80% Iso-octane/20% Acetone



TABLE 7
8081
CONCENTRATIONS OF LCS/MS/MSD SPIKE SOLUTION

Compound	Concentration ($\mu\text{g/mL}$)
TCMX (surrogate)	1.6
α -BHC	0.8
γ -BHC (Lindane)	0.8
β -BHC	0.8
δ -BHC	0.8
Heptachlor	0.8
Aldrin	0.8
Heptachlor epoxide	0.8
γ -Chlordane	0.8
α -Chlordane	0.8
4,4'-DDE	1.6
Endosulfan I	0.8
Dieldrin	1.6
Endrin	1.6
4,4'-DDD	1.6
Endosulfan II	1.6
4,4'-DDT	1.6
Endrin aldehyde	1.6
Methoxychlor	8.0
Endosulfan sulfate	1.6
Endrin ketone	1.6
DCBP (Surrogate)	3.2

Solvent: Acetone



FIGURE 1.1

CHROMATOGRAM OF MIX 1-5, RTX – 1701

Data File E:\HPCHEM\2\DATA\GC1885\010F1001.D Sample Name: L1-5 L143 P110
=====
Injection Date : 12/11/2007 3:41:11 PM Seq. Line : 10
Sample Name : L1-5 L143 P110 Location : Vial 10
Acq. Operator : FUZ Inj : 1
Inj Volume : Manually
Acq. Method : E:\HPCHEM\2\METHODS\GC1885.M
Last changed : 12/10/2007 2:51:55 PM by FUZ
Analysis Method : E:\HPCHEM\2\METHODS\GC1885A.M
Last changed : 1/28/2008 12:06:19 PM by jjk
(modified after loading)
Chlorinated pesticides and pcb's method (8081 & 8082). Column RTX-1701(S/N 835482) 30 M X 0.
53 mmID 0.25 micron film.
Splitless using siltex gooseneck liner, 2ul injection.
=====

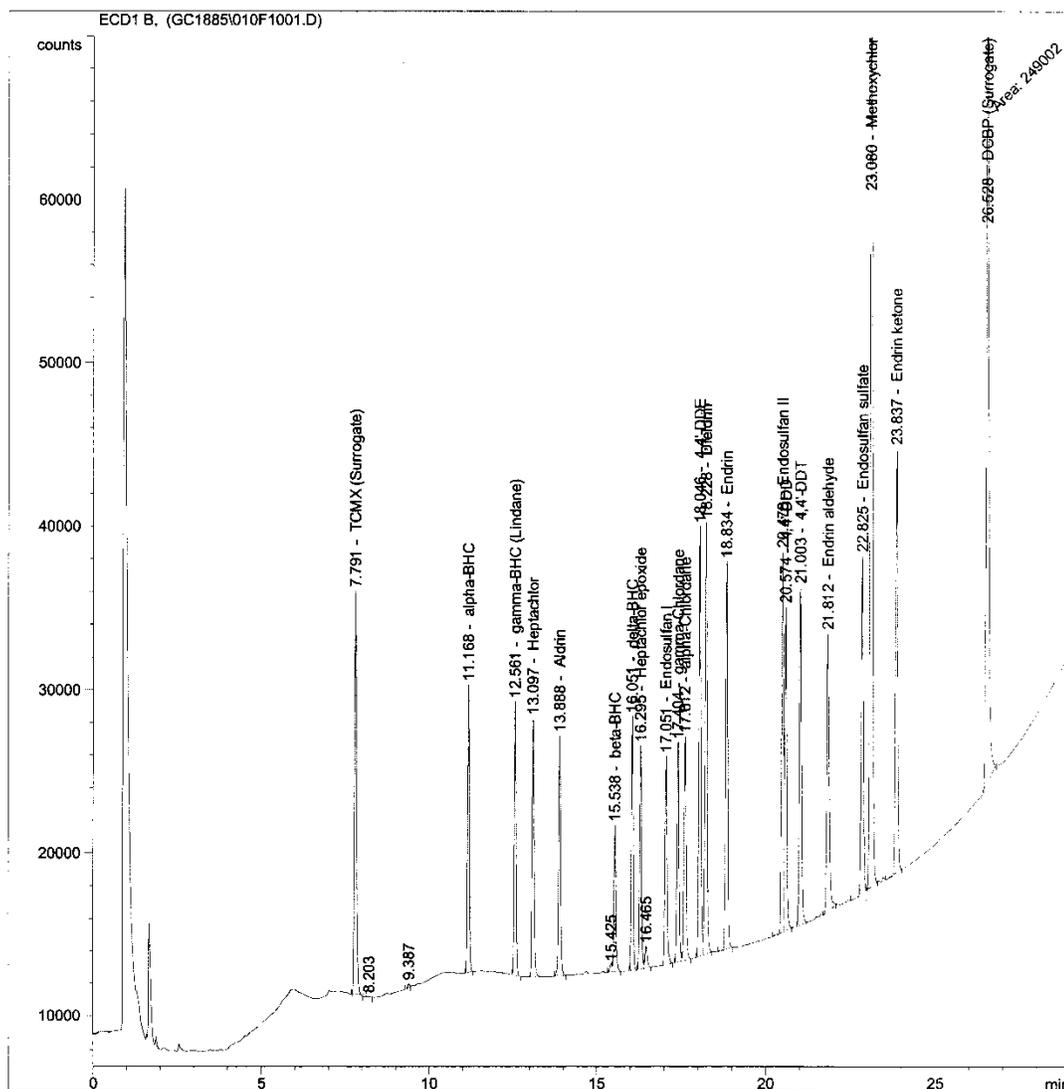




FIGURE 1.2

CHROMATOGRAM OF MIX 1-5, RTX – 1701

```

Data File E:\HPCHEM\2\DATA\GC1885\010F1001.D          Sample Name: L1-5 L143 P110
=====
Injection Date   : 12/11/2007 3:41:11 PM              Seq. Line   : 10
Sample Name     : L1-5 L143 P110                     Location    : Vial 10
Acq. Operator   : FUZ                                Inj         : 1
                                                    Inj Volume  : Manually

Acq. Method     : E:\HPCHEM\2\METHODS\GC1885.M
Last changed    : 12/10/2007 2:51:55 PM by FUZ
Analysis Method : E:\HPCHEM\2\METHODS\GC1885A.M
Last changed    : 1/28/2008 12:06:19 PM by jjk
                  (modified after loading)

Chlorinated pesticides and pcb's method (8081 & 8082). Column RTX-1701(S/N 835482) 30 M X 0.
53 mmID 0.25 micron film.
Splitless using siltex gooseneck liner, 2ul injection.
=====

External Standard Report
=====

Sorted By      :      Signal
Calib. Data Modified : 1/28/2008 11:56:00 AM
Multiplier     :      1.0000
Dilution       :      1.0000
Sample Amount  :      1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: ECD1 B,

RetTime Type Height Amt/Height Amount Grp Name
 [min] [counts] [UG/ML]
-----|-----|-----|-----|-----|-----
 7.791 BB + 2.48039e4 9.46545e-7 2.34780e-2 TCMX (Surrogate)
11.168 PB 1.76904e4 6.38145e-7 1.12891e-2 alpha-BHC
12.561 BB 1.68425e4 6.77013e-7 1.14026e-2 gamma-BHC (Lindane)
13.097 PB 1.59159e4 7.08778e-7 1.12809e-2 Heptachlor
13.688 BB 1.47812e4 7.43863e-7 1.09952e-2 Aldrin
15.538 VB 9019.18359 1.26454e-6 1.14051e-2 beta-BHC
16.051 PP 1.56310e4 7.32997e-7 1.14575e-2 delta-BHC
16.295 BV 1.37556e4 8.22894e-7 1.13194e-2 Heptachlor epoxide
17.051 PB 1.28280e4 8.85019e-7 1.13530e-2 Endosulfan I
17.404 FV 1.35143e4 8.44688e-7 1.14154e-2 gamma-Chlordane
17.612 VB 1.37501e4 8.32810e-7 1.14512e-2 alpha-Chlordane
18.046 PV 2.63673e4 8.90325e-7 2.34755e-2 4,4'-DDE
18.228 VB 2.64761e4 8.77017e-7 2.32200e-2 Dieldrin
18.834 PB 2.37807e4 9.91468e-7 2.35779e-2 Endrin
20.478 PV 2.32937e4 1.01401e-6 2.36200e-2 Endosulfan II
20.574 VB 1.98321e4 1.19022e-6 2.36045e-2 4,4'-DDD
21.003 PB 2.06947e4 1.15331e-6 2.38674e-2 4,4'-DDT
21.812 VB 1.70327e4 1.38124e-6 2.35262e-2 Endrin aldehyde
22.825 VP 2.06647e4 1.15370e-6 2.38408e-2 Endosulfan sulfate
23.080 VB 5.98940e4 2.06369e-6 1.23603e-1 Methoxychlor
23.837 VB 2.59896e4 9.34282e-7 2.42817e-2 Endrin ketone
26.528 MM + 4.97398e4 9.94996e-7 4.94909e-2 DCBP (Surrogate)

Totals :                               2217.88526

Results obtained with enhanced integrator!
=====
*** End of Report ***

```



FIGURE 2.1

CHROMATOGRAM OF MIX 1-5, RTX – 35

Data File E:\HPCHEM\1\DATA\GC1885C\010F1001.D Sample Name: L1-5 L143 P110
=====
Injection Date : 12/11/2007 2:44:54 PM Seq. Line : 10
Sample Name : L1-5 L143 P110 Location : Vial 10
Acq. Operator : FUZ Inj : 1
Inj Volume : Manually
Acq. Method : E:\HPCHEM\1\METHODS\GC1885C.M
Last changed : 12/10/2007 2:47:29 PM by FUZ
Analysis Method : E:\HPCHEM\1\METHODS\GC1885CA.M
Last changed : 1/28/2008 12:25:58 PM by jjk
(modified after loading)
Chlorinated pesticide and TOX method 8081/8082. RTX-35 (S/N 824251) 30M X 0.53mm ID 0.50
micron film. Split/splitless injection.
=====

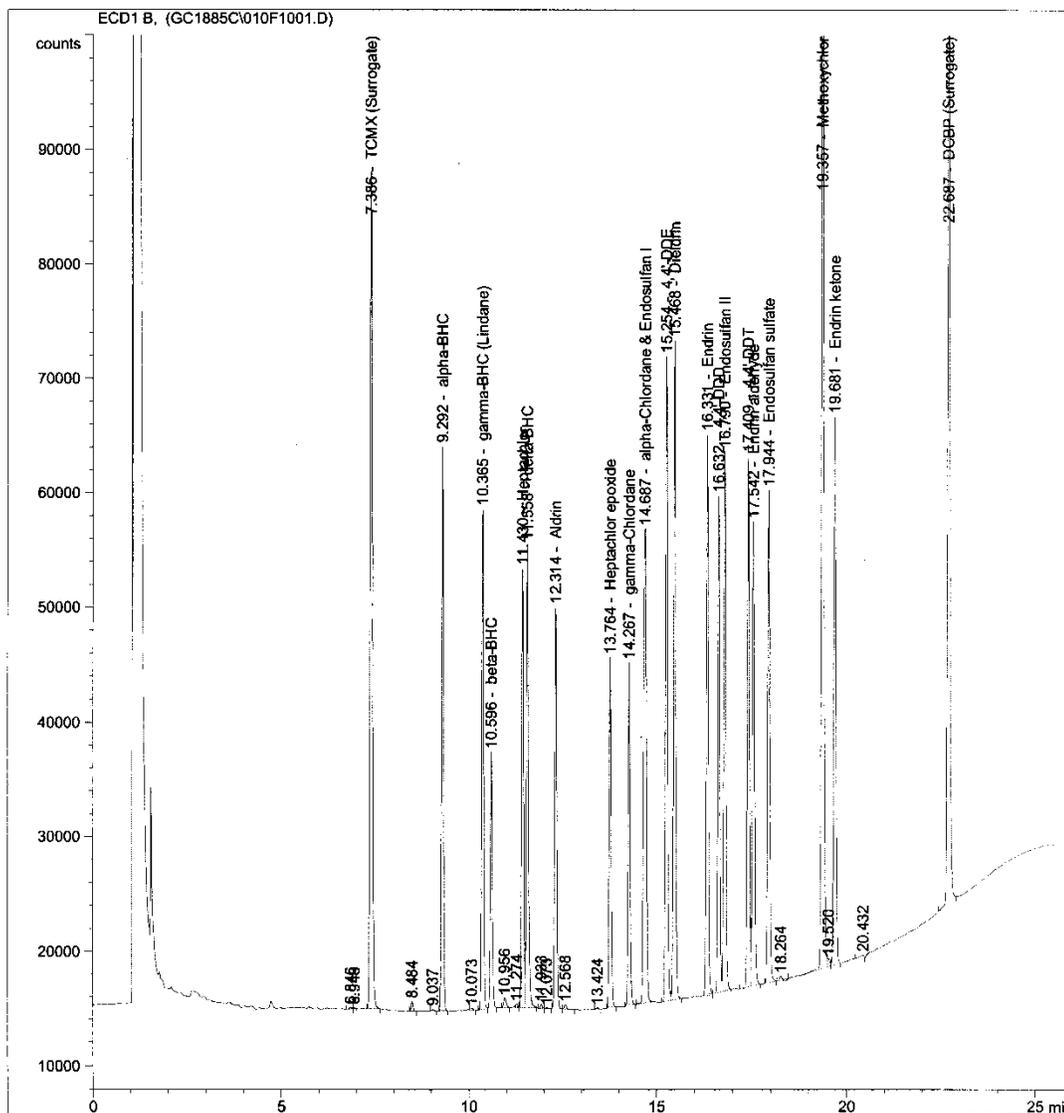




FIGURE 2.2

CHROMATOGRAM OF MIX 1-5, RTX – 35

```

Data File E:\HPCHEM\1\DATA\GC1885C\010F1001.D                               Sample Name: L1-5 L143 P110
=====
Injection Date : 12/11/2007 2:44:54 PM                               Seq. Line : 10
Sample Name    : L1-5 L143 P110                                       Location  : Vial 10
Acq. Operator  : FUZ                                                  Inj       : 1
                                                    Inj Volume: Manually

Acq. Method    : E:\HPCHEM\1\METHODS\GC1885C.M
Last changed   : 12/10/2007 2:47:29 PM by FUZ
Analysis Method: E:\HPCHEM\1\METHODS\GC1885CA.M
Last changed   : 1/28/2008 12:25:58 PM by jjk
                (modified after loading)
Chlorinated pesticide and TOX method 8081/8082. RTX-35 (S/N 824251) 30M X 0.53mm ID 0.50
micron film. Split/splitless injection.
=====
External Standard Report
=====
Sorted By      : Signal
Calib. Data Modified : 1/28/2008 12:26:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount   : 1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: ECD1 B,

RetTime  Type  Height  Amt/Height  Amount  Grp  Name
 [min]   [----] [counts] [-----] [-----] [---] [-----]
-----
 7.386 BB + 7.35041e4 2.54978e-7 1.87419e-2 TCMX (Surrogate)
 9.292 BP 4.93682e4 1.96071e-7 9.67970e-3 alpha-BHC
10.365 BB 4.38212e4 2.19050e-7 9.59905e-3 gamma-BHC (Lindane)
10.596 BE 2.25075e4 4.24538e-7 9.55528e-3 beta-BHC
11.430 VV 3.84820e4 2.45990e-7 9.46617e-3 Heptachlor
11.558 VE 4.06513e4 2.35645e-7 9.57927e-3 delta-BHC
12.314 BB 3.51872e4 2.68716e-7 9.45536e-3 Aldrin
13.764 PB 3.06899e4 3.08158e-7 9.45732e-3 Heptachlor epoxide
14.267 FP 3.00581e4 3.17920e-7 9.55608e-3 gamma-Chlordane
14.687 BE 4.14745e4 2.31549e-7 9.60337e-3 alpha-Chlordane & Endosulfan I
15.254 PV 5.63955e4 3.44300e-7 1.94170e-2 4,4'-DDE
15.468 VB 5.76730e4 3.33145e-7 1.92135e-2 Dieldrin
16.331 BP 4.90320e4 3.93856e-7 1.93116e-2 Endrin
16.632 VV 4.34419e4 4.44220e-7 1.92977e-2 4,4'-DDD
16.790 VP 4.71854e4 4.04488e-7 1.90859e-2 Endosulfan II
17.409 PV 4.65010e4 4.12948e-7 1.92025e-2 4,4'-DDT
17.542 VB 4.06174e4 4.66205e-7 1.89360e-2 Endrin aldehyde
17.944 PB 4.30726e4 4.42127e-7 1.90435e-2 Endosulfan sulfate
19.357 BV 1.07148e5 8.63280e-7 9.24985e-2 Methoxychlor
19.681 VE 4.79474e4 4.03138e-7 1.93294e-2 Endrin ketone
22.687 BBA + 8.09642e4 4.59857e-7 3.72320e-2 DCBP (Surrogate)

Totals :                               5165.73017

Results obtained with enhanced integrator!
=====
*** End of Report ***

```



FIGURE 3.1

CHROMATOGRAM OF MIX 2-5, RTX – 1701

Data File E:\HPCHEM\2\DATA\GC1885\015F1501.D
L173P64

Sample Name: CCV 8081 MIX 2-5

=====
Injection Date : 12/11/2007 6:40:39 PM Seq. Line : 15
Sample Name : CCV 8081 MIX 2-5 Location : Vial 15
Acq. Operator : FUZ Inj : 1
 Inj Volume : Manually

Acq. Method : E:\HPCHEM\2\METHODS\GC1885.M
Last changed : 12/10/2007 2:51:55 PM by FUZ
Analysis Method : E:\HPCHEM\2\METHODS\GC1885-2.M
Last changed : 1/28/2008 12:39:11 PM by jjk
Chlorinated pesticides and pcb's method (8081 & 8082). Column RTX-1701(S/N 661245) 30 M X 0.53 mmID 0.25 micron film.
Splitless using siltex gooseneck liner, 2ul injection.
=====

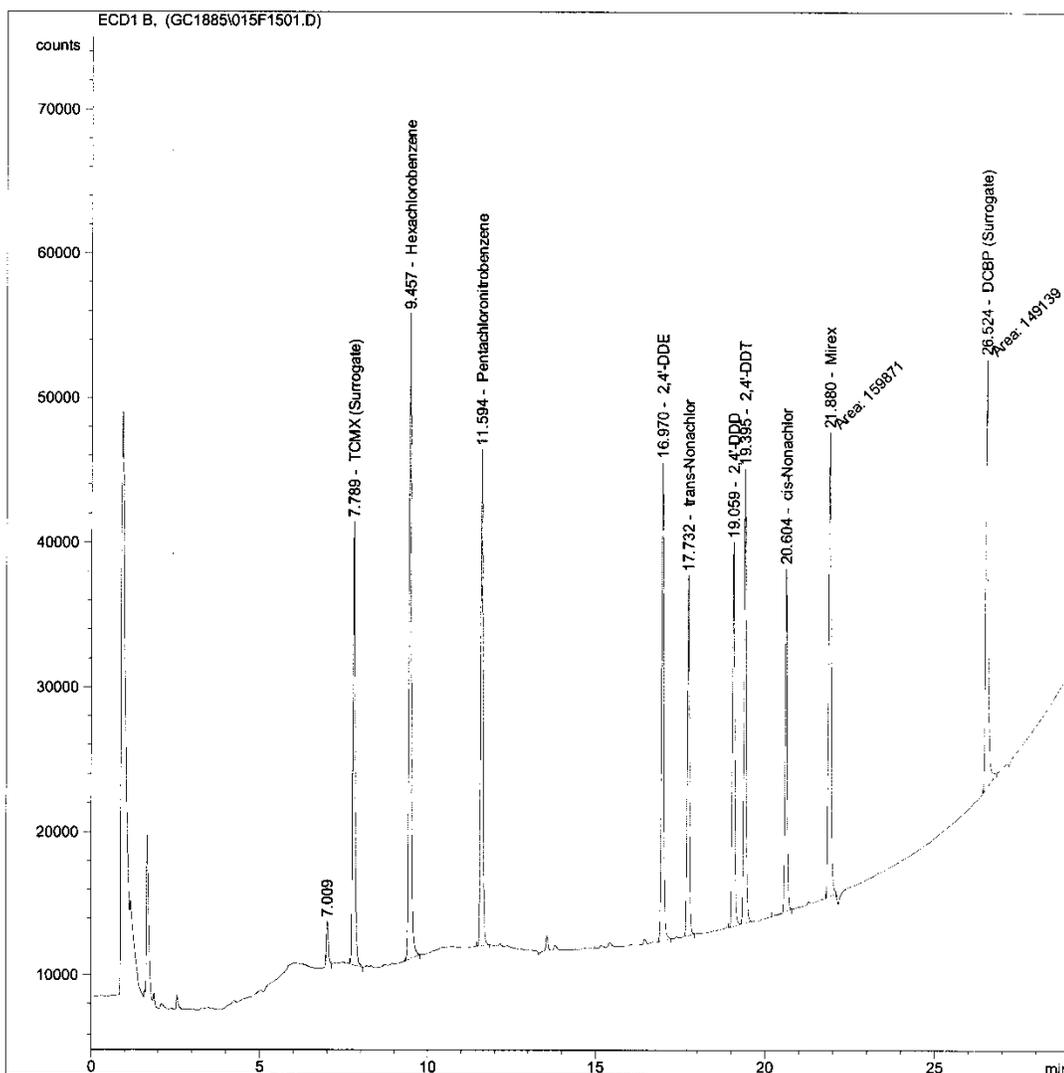




FIGURE 3.2
 CHROMATOGRAM OF MIX 2-5, RTX – 1701

Data File E:\HPCHEM\2\DATA\GC1885\015F1501.D Sample Name: CCV 8081 MIX 2-5
 L173P64

```

=====
Injection Date : 12/11/2007 6:40:39 PM      Seq. Line : 15
Sample Name   : CCV 8081 MIX 2-5          Location  : Vial 15
Acq. Operator : FUZ                       Inj      : 1
                                           Inj Volume: Manually

Acq. Method   : E:\HPCHEM\2\METHODS\GC1885.M
Last changed  : 12/10/2007 2:51:55 PM by FUZ
Analysis Method : E:\HPCHEM\2\METHODS\GC1885-2.M
Last changed  : 1/28/2008 12:39:11 PM by jjk
Chlorinated pesticides and pcb's method (8081 & 8082). Column RTX-1701(S/N 661248) 30 M X 0.
53 mmID 0.25 micron film.
Splitless using siltex gooseneck liner, 2ul injection.
=====
  
```

External Standard Report

```

=====
Sorted By      : Signal
Calib. Data Modified : 1/28/2008 12:38:44 PM
Multiplier    : 1.0000
Dilution      : 1.0000
Sample Amount  : 1.00000 [ug/ml] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: ECD1 B,

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [ug/ml]	Grp	Name
7.789	BP +	3.07873e4	1.94886e-6	6.00000e-2		TCMX (Surrogate)
9.457	PB	4.47608e4	4.46819e-7	2.00000e-2		Hexachlorobenzene
11.594	BB	3.44374e4	5.80765e-7	2.00000e-2		Pentachloronitrobenzene
16.970	BB	3.32204e4	1.20408e-6	4.00000e-2		2,4'-DDE
17.732	BB	2.50743e4	7.97628e-7	2.00000e-2		trans-Nonachlor
19.059	VB	2.66883e4	1.49878e-6	4.00000e-2		2,4'-DDD
19.395	BB	3.15090e4	1.26948e-6	4.00000e-2		2,4'-DDT
20.604	PB	2.38002e4	8.40329e-7	2.00000e-2		cis-Nonachlor
21.880	MM	3.21721e4	1.24086e-6	3.99211e-2		Mirex
26.524	MM +	2.97068e4	2.02257e-6	6.00840e-2		DCBP (Surrogate)

Totals : 3.60005e-1

Uncalibrated peaks RF : 1.00000

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [ug/ml]	Grp	Name
7.009	BP	3044.79517	1.00000	3044.79517	?	

Uncalib. totals : 3044.79517

Results obtained with enhanced integrator!
 1 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

*** End of Report ***



FIGURE 4.1

CHROMATOGRAM OF MIX 2-5, RTX – 35

Data File E:\HPCHEM\1\DATA\GC1885C\015F1501.D
L173P64

Sample Name: CCV 8081 MIX 2-5

=====
Injection Date : 12/11/2007 5:15:33 PM Seq. Line : 15
Sample Name : CCV 8081 MIX 2-5 Location : Vial 15
Acq. Operator : FUZ Inj : 1
 Inj Volume : Manually

Acq. Method : E:\HPCHEM\1\METHODS\GC1885C.M
Last changed : 12/10/2007 2:47:29 PM by FUZ
Analysis Method : E:\HPCHEM\1\METHODS\GC1885C2.M
Last changed : 1/28/2008 12:35:34 PM by jjk
 (modified after loading)

Chlorinated pesticide and TOX method 8081/8082. RTX-35 (S/N 720907) 30M X 0.53mm ID 0.50
micron film. Split/splitless injection.

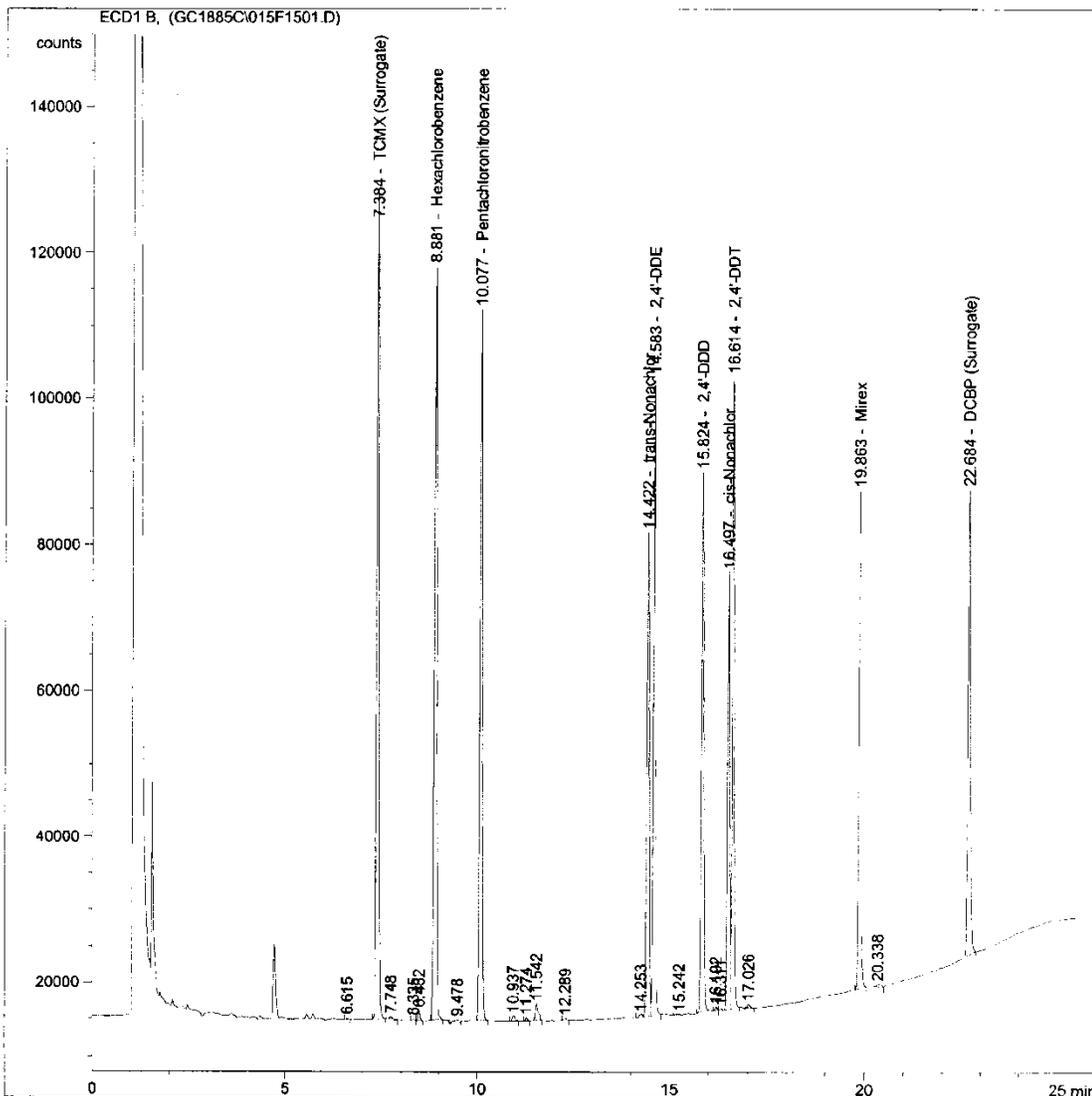




FIGURE 4.2

CHROMATOGRAM OF MIX 2-5, RTX – 35

Data File E:\HPCHEM\1\DATA\GC1885C\015F1501.D Sample Name: CCV 8081 MIX 2-5
 L173P64

```

=====
Injection Date   : 12/11/2007 5:15:33 PM      Seq. Line   : 15
Sample Name     : CCV 8081 MIX 2-5          Location    : Vial 15
Acq. Operator   : FUZ                      Inj         : 1
                                           Inj Volume  : Manually
Acq. Method     : E:\HPCHEM\1\METHODS\GC1885C.M
Last changed    : 12/10/2007 2:47:29 PM by FUZ
Analysis Method : E:\HPCHEM\1\METHODS\GC1885C2.M
Last changed    : 1/28/2008 12:35:34 PM by jjk
                  (modified after loading)
Chlorinated pesticide and TOX method 8081/8082. RTX-35 (S/N 720907) 30M X 0.53mm ID 0.50
micron film. Split/splitless injection.
=====
  
```

External Standard Report

```

=====
Sorted By       : Signal
Calib. Data Modified : 1/28/2008 12:35:33 PM
Multiplier      : 1.0000
Dilution        : 1.0000
Sample Amount   : 1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: ECD1 B,

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [UG/ML]	Grp	Name
7.384	BB +	1.14317e5	5.24856e-7	6.00000e-2		TCMX (Surrogate)
8.881	BE	1.03460e5	1.93312e-7	2.00000e-2		Hexachlorobenzene
10.077	BB	9.75669e4	2.04988e-7	2.00000e-2		Pentachloronitrobenzene
14.422	VV	6.65497e4	3.00527e-7	2.00000e-2		trans-Nonachlor
14.583	VB	8.76335e4	4.56446e-7	4.00000e-2		2,4'-DDE
15.824	PE	7.41088e4	5.39747e-7	4.00000e-2		2,4'-DDD
16.497	VV	6.01389e4	3.32563e-7	2.00000e-2		cis-Nonachlor
16.614	VB	8.66826e4	4.61453e-7	4.00000e-2		2,4'-DDT
19.863	PE	6.84233e4	5.84597e-7	4.00000e-2		Mirex
22.684	BB +	6.38261e4	9.40054e-7	6.00000e-2		DCBP (Surrogate)

Totals : 8670.34152

Results obtained with enhanced integrator!
 1 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

*** End of Report ***



FIGURE 5.1

CHROMATOGRAM OF TOXOPHENE 0.8 µg/mL, RTX – 1701

Data File E:\HPCHEM\2\DATA\GC1885\004F0401.D
L143P100

Sample Name: TOXAPHENE 0.8

=====
Injection Date : 12/11/2007 12:06:26 PM Seq. Line : 4
Sample Name : TOXAPHENE 0.8 Location : Vial 4
Acq. Operator : FUZ Inj : 1
 Inj Volume : Manually

Acq. Method : E:\HPCHEM\2\METHODS\GC1885.M
Last changed : 12/10/2007 2:51:55 PM by FUZ
Analysis Method : E:\HPCHEM\2\METHODS\GC1885A.M
Last changed : 1/28/2008 12:00:25 PM by jjk
 (modified after loading)

Chlorinated pesticides and pcb's method (8081 & 8082). Column RTX-1701(S/N 835482) 30 M X 0.53 mmID 0.25 micron film.
Splitless using siltex gooseneck liner, 2ul injection.

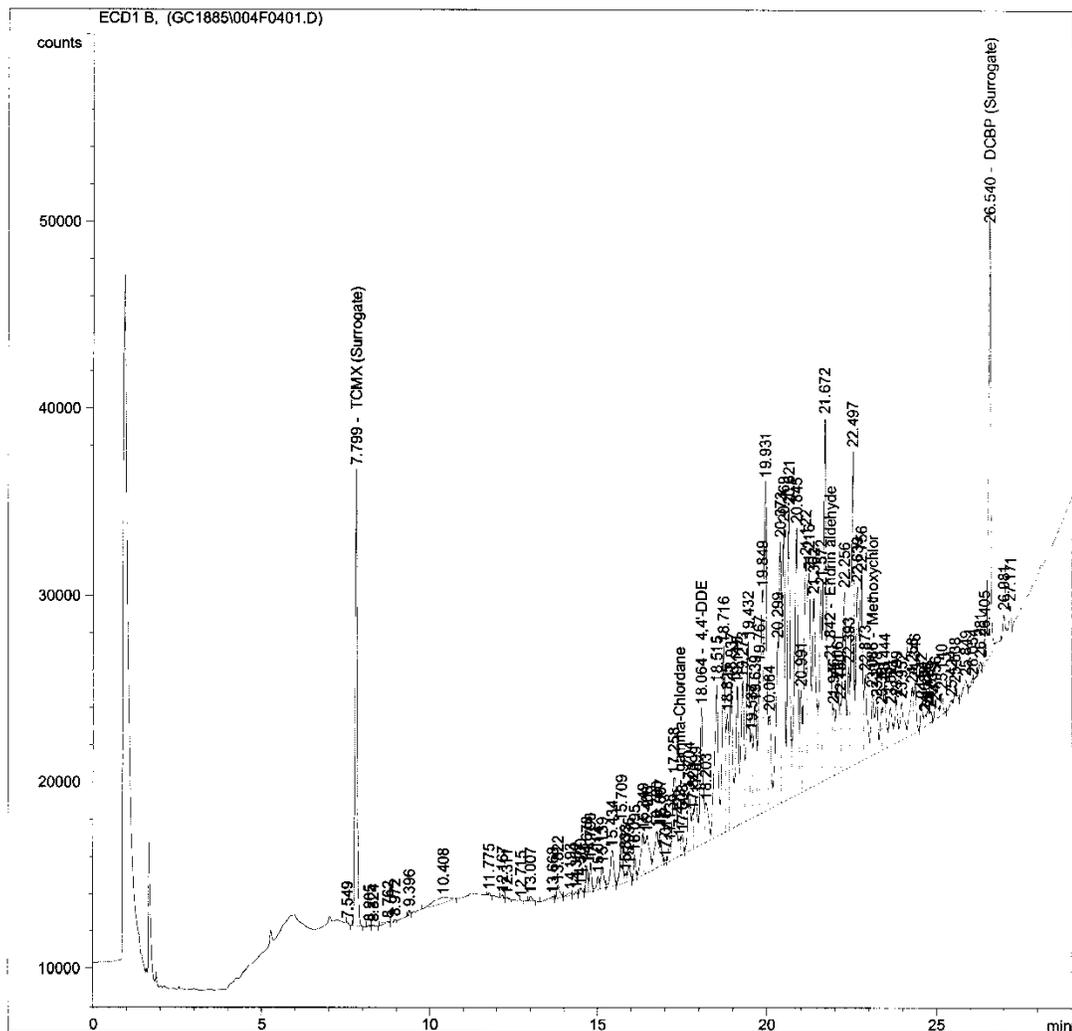




FIGURE 5.2

CHROMATOGRAM OF TOXOPHENE 0.8 µg/mL, RTX – 1701

Data File E:\HPCHEM\2\DATA\GC1885\004F0401.D
L143F100

Sample Name: TOXAPHENE 0.8

=====
Injection Date : 12/11/2007 12:06:26 PM Seq. Line : 4
Sample Name : TOXAPHENE 0.8 Location : Vial 4
Acq. Operator : FUZ Inj : 1
 Inj Volume : Manually

Acq. Method : E:\HPCHEM\2\METHODS\GC1885.M
Last changed : 12/10/2007 2:51:55 PM by FUZ
Analysis Method : E:\HPCHEM\2\METHODS\GC1885A.M
Last changed : 1/28/2008 12:00:25 PM by jjk
 (modified after loading)
Chlorinated pesticides and pcb's method (8081 & 8082). Column RTX-1701(S/N 835482) 30 M X 0.
53 mmID 0.25 micron film.
Splitless using siltex gooseneck liner, 2ul injection.
=====

=====
External Standard Report
=====

Sorted By : Signal
Calib. Data Modified : 1/28/2008 11:56:00 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: ECD1 B,

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [UG/ML]	Grp	Name
7.799	BB +	2.45179e4	9.46085e-7	2.31960e-2		TCMX (Surrogate)
11.178		-	-	-		alpha-BHC
12.571		-	-	-		gamma-BHC (Lindane)
13.108		-	-	-		Heptachlor
13.900		-	-	-		Aldrin
15.548		-	-	-		beta-BHC
16.062		-	-	-		delta-BHC
16.307		-	-	-		Heptachlor epoxide
17.063		-	-	-		Endosulfan I
17.422	VV	920.61322	8.86012e-7	8.15674e-4		gamma-Chlordane
17.623		-	-	-		alpha-Chlordane
18.064	VV	7354.71289	9.27909e-7	6.82451e-3		4,4'-DDE
18.240		-	-	-		Dieldrin
18.845		-	-	-		Endrin
20.490		-	-	-		Endosulfan II
20.584		-	-	-		4,4'-DDD
21.014		-	-	-		4,4'-DDT
21.842	VV	6182.20508	1.37519e-6	8.50168e-3		Endrin aldehyde
22.837		-	-	-		Endosulfan sulfate
23.086	VV	3438.51611	1.90872e-6	6.56318e-3		Methoxychlor
23.848		-	-	-		Endrin ketone
26.540	VV +	2.37907e4	9.60261e-7	2.28453e-2		DCBP (Surrogate)

Totals : 3.89777e5

Results obtained with enhanced integrator!

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

=====
*** End of Report ***



FIGURE 6.2

CHROMATOGRAM OF TOXOPHENE 0.8 µG/ML, RTX – 35

Data File E:\HPCHEM\1\DATA\GC1885C\004F0401.D
 L143P100

Sample Name: TOXAPHENE 0.8

```

=====
Injection Date : 12/11/2007 11:45:20 AM      Seq. Line :    4
Sample Name    : TOXAPHENE 0.8              Location  : Vial 4
Acq. Operator  : FUZ                        Inj       :    1
                                           Inj Volume: Manually

Acq. Method    : E:\HPCHEM\1\METHODS\GC1885C.M
Last changed   : 12/10/2007 2:47:29 PM by FUZ
Analysis Method : E:\HPCHEM\1\METHODS\GC1885CA.M
Last changed   : 1/28/2008 12:23:16 PM by jjk
                (modified after loading)

Chlorinated pesticide and TOX method 8081/8082. RTX-35 (S/N 824251) 30M X 0.53mm ID 0.50
micron film. Split/splitless injection.
=====
  
```

External Standard Report

```

=====
Sorted By      :      Signal
Calib. Data Modified : 1/28/2008 12:18:24 PM
Multiplier     :      1.0000
Dilution       :      1.0000
Sample Amount   :      1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: ECD1 B.

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [UG/ML]	Grp	Name
7.385	VB +	7.95970e4	2.56348e-7	2.04045e-2		TCMX (Surrogate)
9.297	VP	120.40765	2.21006e-7	2.66108e-5		alpha-BHC
10.365		-	-	-		gamma-BHC (Lindane)
10.596		-	-	-		beta-BHC
11.431		-	-	-		Heptachlor
11.558		-	-	-		delta-BHC
12.315		-	-	-		Aldrin
13.766	VV	4795.10205	3.09519e-7	1.48418e-3		Heptachlor epoxide
14.267		-	-	-		gamma-Chlordane
14.690		-	-	-		alpha-Chlordane & Endosulfan I
15.268	VV	1.78591e4	3.55771e-7	6.35374e-3		4,4'-DDE
15.469		-	-	-		Dieldrin
16.331		-	-	-		Endrin
16.632		-	-	-		4,4'-DDD
16.791		-	-	-		Endosulfan II
17.409		-	-	-		4,4'-DDT
17.561	VV	4.35805e4	4.66829e-7	2.03446e-2		Endrin aldehyde
17.928	VV	2.23656e4	4.43279e-7	9.91419e-3		Endosulfan sulfate
19.357		-	-	-		Methoxychlor
19.681		-	-	-		Endrin ketone
22.686	PV +	4.50185e4	4.39754e-7	1.97971e-2		DCBP (Surrogate)

Totals : 8.84839e5

Results obtained with enhanced integrator!
 1 Warnings or Errors :

Warning : Calibrated compound(s) not found

*** End of Report ***



FIGURE 7.2

CHROMATOGRAM OF TECHNICAL CHLORDANE 0.8 µG/ML, RTX – 1701

Data File E:\HPCHEM\2\DATA\GC1885\005F0501.D
 L143P99

Sample Name: TECH CL

```

=====
Injection Date : 12/11/2007 12:42:23 PM      Seq. Line : 5
Sample Name    : TECH CL                    Location  : Vial 5
Acq. Operator  : FUZ                       Inj       : 1
                                           Inj Volume : Manually

Acq. Method    : E:\HPCHEM\2\METHODS\GC1885.M
Last changed   : 12/10/2007 2:51:55 PM by FUZ
Analysis Method : E:\HPCHEM\2\METHODS\GC1885A.M
Last changed   : 1/28/2008 12:00:25 PM by jjk
                (modified after loading)

Chlorinated pesticides and pcb's method (8081 & 8082). Column RTX-1701(S/N B35482) 30 M X 0.
53 mmID 0.25 micron film.
Splitless using siltex gooseneck liner, 2ul injection.
=====
  
```

External Standard Report

```

Sorted By      : Signal
Calib. Data Modified : 1/28/2008 11:56:00 AM
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount   : 1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: ECD1 B,

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [UG/ML]	Grp	Name
7.800	VB +	5.14617e4	9.75985e-7	5.02258e-2		TCMX (Surrogate)
11.191	BB	66.76406	9.17355e-7	6.12463e-5		alpha-BHC
12.589	PV	4606.88574	7.23620e-7	3.33363e-3		gamma-BHC (Lindane)
13.106	VB	9094.20410	7.14863e-7	6.50111e-3		Heptachlor
13.900	-	-	-	-		Aldrin
15.547	-	-	-	-		beta-BHC
16.061	-	-	-	-		delta-BHC
16.287	VV	1531.95508	8.70940e-7	1.33424e-3		Heptachlor epoxide
17.062	-	-	-	-		Endosulfan I
17.414	VV	1.61419e4	8.42024e-7	1.35919e-2		gamma-Chlordane
17.621	VV	1.29906e4	8.33501e-7	1.08277e-2		alpha-Chlordane
18.056	-	-	-	-		4,4'-DDE
18.257	VV	1160.85205	1.03633e-6	1.20302e-3		Dieldrin
18.844	-	-	-	-		Endrin
20.489	-	-	-	-		Endosulfan II
20.582	-	-	-	-		4,4'-DDD
21.012	-	-	-	-		4,4'-DDT
21.822	-	-	-	-		Endrin aldehyde
22.835	-	-	-	-		Endosulfan sulfate
23.088	-	-	-	-		Methoxychlor
23.846	-	-	-	-		Endrin ketone
26.538	MM +	4.76691e4	9.92960e-7	4.73335e-2		DCBP (Surrogate)

Totals : 6.30059e4

Results obtained with enhanced integrator!
 1 Warnings or Errors :

Warning : Calibrated compound(s) not found

*** End of Report ***



FIGURE 8.2

CHROMATOGRAM OF TECHNICAL CHLORDANE 0.8 µG/ML, RTX – 35

Data File E:\HPCHEM\1\DATA\GC1885C\005F0501.D
 L143P99

Sample Name: TECH CL 0.08

```

=====
Injection Date : 12/11/2007 12:15:20 PM      Seq. Line : 5
Sample Name    : TECH CL 0.08                Location  : Vial 5
Acq. Operator  : FUZ                          Inj       : 1
                                           Inj Volume : Manually

Acq. Method    : E:\HPCHEM\1\METHODS\GC1885C.M
Last changed   : 12/10/2007 2:47:29 PM by FUZ
Analysis Method : E:\HPCHEM\1\METHODS\GC1885CA.M
Last changed   : 1/28/2008 12:25:58 PM by jjk
                 (modified after loading)

Chlorinated pesticide and TOX method 8081/8082, RTX-35 (S/N 824251) 30M X 0.53mm ID 0.50
micron film. Split/splitless injection.
=====
  
```

External Standard Report

```

=====
Sorted By      : Signal
Calib. Data Modified : 1/28/2008 12:26:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount   : 1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: ECD1 B,

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [UG/ML]	Grp	Name
7.386	BV +	1.86840e5	2.71499e-7	5.07269e-2		TCMX (Surrogate)
9.294		-	-	-		alpha-BHC
10.366		-	-	-		gamma-BHC (Lindane)
10.597		-	-	-		beta-BHC
11.430	VV	2.68222e4	2.45047e-7	6.57269e-3		Heptachlor
11.559		-	-	-		delta-BHC
12.316		-	-	-		Aldrin
13.766		-	-	-		Heptachlor epoxide
14.267	VV	4.36375e4	3.18323e-7	1.38908e-2		gamma-Chlordane
14.665	VV	3.36925e4	2.32082e-7	7.81942e-3		alpha-Chlordane & Endosulfan I
15.255		-	-	-		4,4'-DDE
15.469		-	-	-		Dieldrin
16.331		-	-	-		Endrin
16.633		-	-	-		4,4'-DDD
16.791		-	-	-		Endosulfan II
17.410		-	-	-		4,4'-DDT
17.542		-	-	-		Endrin aldehyde
17.946		-	-	-		Endosulfan sulfate
19.357		-	-	-		Methoxychlor
19.681		-	-	-		Endrin ketone
22.686	BBA +	1.07634e5	4.69938e-7	5.05814e-2		DCBP (Surrogate)

Totals : 1.95175e5

Results obtained with enhanced integrator!
 1 Warnings or Errors :

Warning : Calibrated compound(s) not found

*** End of Report ***



FIGURE 9.1

CHROMATOGRAM ENDRIN BREAKDOWN, RTX – 1701

Data File E:\HPCHEM\2\DATA\GC1885\003F0301.D
L143P94

Sample Name: ENDRIN BREAK

=====
Injection Date : 12/11/2007 11:30:26 AM Seq. Line : 3
Sample Name : ENDRIN BREAK Location : Vial 3
Acq. Operator : FUZ Inj : 1
 Inj Volume : Manually

Acq. Method : E:\HPCHEM\2\METHODS\GC1885.M
Last changed : 12/10/2007 2:51:55 PM by FUZ
Analysis Method : E:\HPCHEM\2\METHODS\GC1885A.M
Last changed : 1/28/2008 11:56:55 AM by jjk
(modified after loading)

Chlorinated pesticides and pcb's method (8081 & 8082). Column RTX-1701(S/N 835482) 30 M X 0.53 mmID 0.25 micron film.
Splitless using siltex gooseneck liner, 2ul injection.

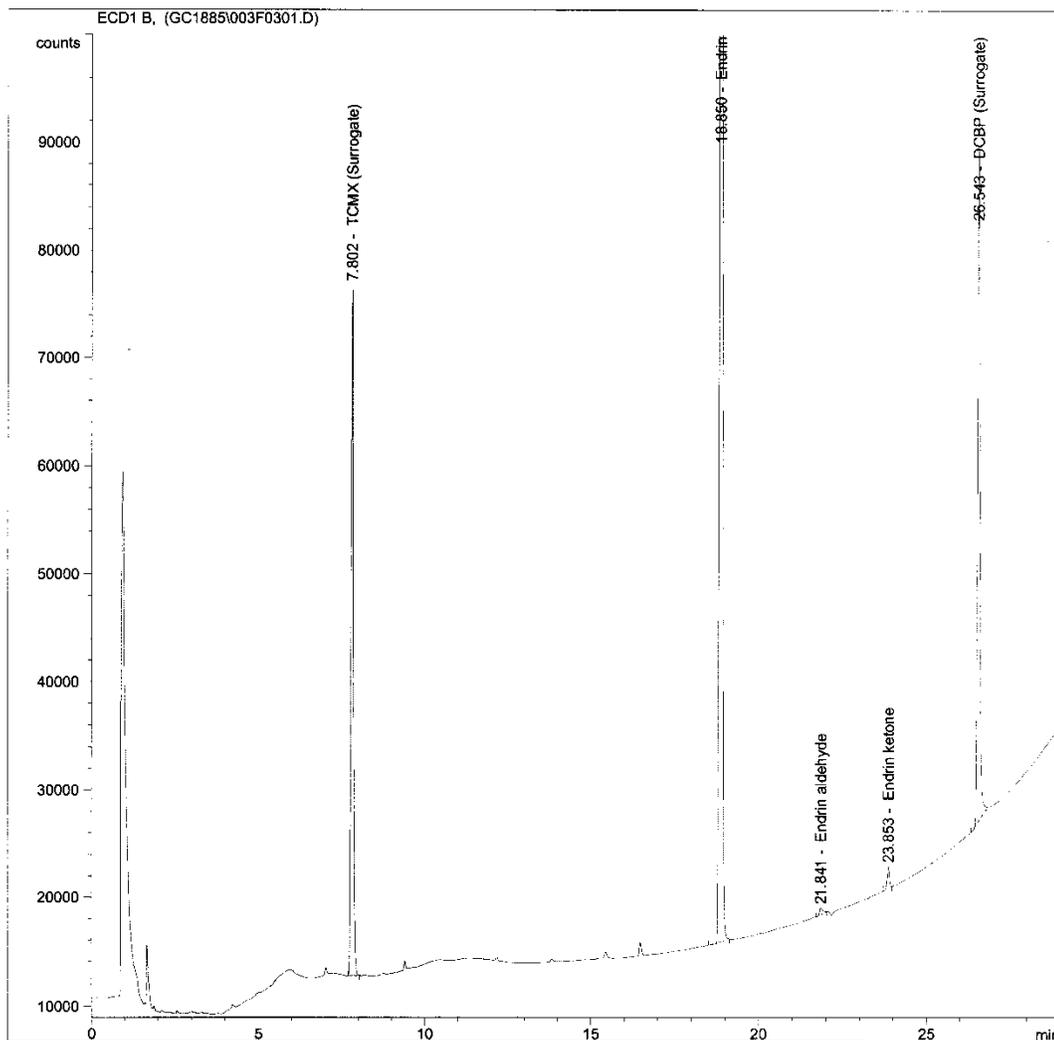




FIGURE 9.2

CHROMATOGRAM ENDRIN BREAKDOWN, RTX – 1701

Data File E:\HPCHEM\2\DATA\GC1885\003F0301.D Sample Name: ENDRIN BREAK
 L143P94

=====
 Injection Date : 12/11/2007 11:30:26 AM Seq. Line : 3
 Sample Name : ENDRIN BREAK Location : Vial 3
 Acq. Operator : FUZ Inj : 1
 Inj Volume : Manually

Acq. Method : E:\HPCHEM\2\METHODS\GC1885.M
 Last changed : 12/10/2007 2:51:55 PM by FUZ
 Analysis Method : E:\HPCHEM\2\METHODS\GC1885A.M
 Last changed : 1/28/2008 11:56:55 AM by jjk
 (modified after loading)

Chlorinated pesticides and pcb's method (8081 & 8082). Column RTX-1701(S/N 835482) 30 M X 0.53 mmID 0.25 micron film.
 Splitless using siltex gooseneck liner, 2ul injection.

=====
 External Standard Report
 =====

Sorted By : Signal
 Calib. Data Modified : 1/28/2008 11:56:00 AM
 Multiplier : 1.0000
 Dilution : 1.0000
 Sample Amount : 1.00000 [UG/ML] (not used in calc.)
 Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: ECD1 B,

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [UG/ML]	Grp	Name
7.802	PB +	6.41792e4	9.85072e-7	6.32212e-2		TCMX (Surrogate)
11.181		-	-	-		alpha-BHC
12.574		-	-	-		gamma-BHC (Lindane)
13.111		-	-	-		Heptachlor
13.903		-	-	-		Aldrin
15.551		-	-	-		beta-BHC
16.065		-	-	-		delta-BHC
16.310		-	-	-		Heptachlor epoxide
17.066		-	-	-		Endosulfan I
17.418		-	-	-		gamma-Chlordane
17.626		-	-	-		alpha-Chlordane
18.060		-	-	-		4,4'-DDE
18.243		-	-	-		Dieldrin
18.850	PB	2.17266e5	8.95663e-7	1.94597e-1		Endrin
20.493		-	-	-		Endosulfan II
20.587		-	-	-		4,4'-DDD
21.017		-	-	-		4,4'-DDT
21.841	PB	665.27057	1.36197e-6	9.06076e-4		Endrin aldehyde
22.840		-	-	-		Endosulfan sulfate
23.093		-	-	-		Methoxychlor
23.853	BP	2060.77490	9.89822e-7	2.03980e-3		Endrin ketone
26.543	BB +	6.23105e4	1.00586e-6	6.26754e-2		DCBP (Surrogate)

Totals : 3.23440e-1

Results obtained with enhanced integrator!
 1 Warnings or Errors :

Warning : Calibrated compound(s) not found

=====
 *** End of Report ***



FIGURE 10.1

CHROMATOGRAM ENDRIN BREAKDOWN, RTX - 35

Data File E:\HPCHEM\1\DATA\GC1885C\003F0301.D
L143P94

Sample Name: ENDRIN BREAK

=====
Injection Date : 12/11/2007 11:15:22 AM Seq. Line : 3
Sample Name : ENDRIN BREAK Location : Vial 3
Acq. Operator : FUZ Inj : 1
 Inj Volume : Manually

Acq. Method : E:\HPCHEM\1\METHODS\GC1885C.M
Last changed : 12/10/2007 2:47:29 PM by FUZ
Analysis Method: E:\HPCHEM\1\METHODS\GC1885CA.M
Last changed : 1/28/2008 12:19:57 PM by jjk
 (modified after loading)
Chlorinated pesticide and TOX method 8081/8082. RTX-35 (S/N 824251) 30M X 0.53mm ID 0.50
micron film. Split/splitless injection.
=====

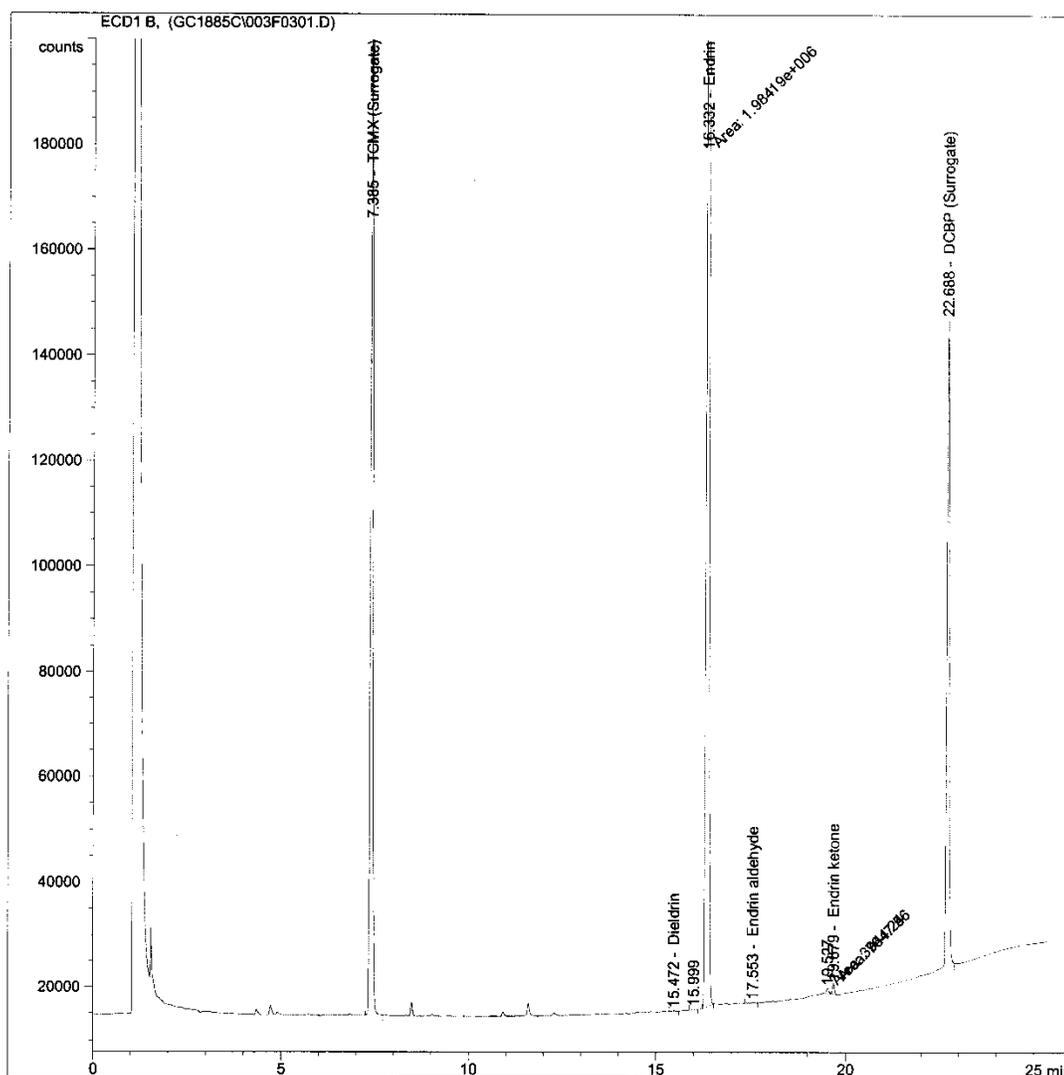




FIGURE 10.2

CHROMATOGRAM ENDRIN BREAKDOWN, RTX – 35

Data File E:\HPCHEM\1\DATA\GC1885C\003F0301.D Sample Name: ENDRIN BREAK
 L143P94

```

=====
Injection Date   : 12/11/2007 11:15:22 AM      Seq. Line   :    3
Sample Name     : ENDRIN BREAK                Location    : Vial 3
Acq. Operator   : FUZ                        Inj         :    1
                                           Inj Volume  : Manually

Acq. Method     : E:\HPCHEM\1\METHODS\GC1885C.M
Last changed    : 12/10/2007 2:47:29 PM by FUZ
Analysis Method : E:\HPCHEM\1\METHODS\GC1885CA.M
Last changed    : 1/28/2008 12:19:57 PM by jjk
                  (modified after loading)

Chlorinated pesticide and TOX method 8081/8082. RTX-35 (S/N 824251) 30M X 0.53mm ID 0.50
micron film. Split/splitless injection.
=====
  
```

External Standard Report

```

=====
Sorted By       :      Signal
Calib. Data Modified : 1/28/2008 12:18:24 PM
Multiplier      :      1.0000
Dilution        :      1.0000
Sample Amount   :      1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: ECD1 B,

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [UG/ML]	Grp	Name
7.385	BB +	2.10785e5	2.73711e-7	5.76942e-2		TCMX (Surrogate)
9.293		-	-	-		alpha-BHC
10.366		-	-	-		gamma-BHC (Lindane)
10.596		-	-	-		beta-BHC
11.432		-	-	-		Heptachlor
11.559		-	-	-		delta-BHC
12.316		-	-	-		Aldrin
13.766		-	-	-		Heptachlor epoxide
14.268		-	-	-		gamma-Chlordane
14.691		-	-	-		alpha-Chlordane & Endosulfan I
15.255		-	-	-		4,4'-DDE
15.472	PP	116.89239	3.97669e-7	4.64845e-5		Dieldrin
16.332	MM	4.87429e5	3.68166e-7	1.79455e-1		Endrin
16.633		-	-	-		4,4'-DDD
16.792		-	-	-		Endosulfan II
17.410		-	-	-		4,4'-DDT
17.553	PP	162.34389	4.19725e-7	6.81398e-5		Endrin aldehyde
17.947		-	-	-		Endosulfan sulfate
19.358		-	-	-		Methoxychlor
19.679	FM	2111.14648	4.14191e-7	8.74418e-4		Endrin ketone
22.688	BEA +	1.22685e5	4.74645e-7	5.82317e-2		DCBP (Surrogate)

Totals : 1210.76520

Results obtained with enhanced integrator!
 1 Warnings or Errors :

Warning : Calibrated compound(s) not found

*** End of Report ***



FIGURE 11.2

CHROMATOGRAM OF DDT BREAKDOWN, RTX – 1701

Data File E:\HPCHEM\2\DATA\GC1885\002F0201.D
 L143P96

Sample Name: DDT BREAK

```

=====
Injection Date : 12/11/2007 10:54:28 AM      Seq. Line : 2
Sample Name    : DDT BREAK                    Location  : Vial 2
Acq. Operator  : FUZ                          Inj       : 1
                                           Inj Volume: Manually

Acq. Method    : E:\HPCHEM\2\METHODS\GC1885.M
Last changed   : 12/10/2007 2:51:55 PM by FUZ
Analysis Method : E:\HPCHEM\2\METHODS\GC1885A.M
Last changed   : 1/28/2008 11:56:55 AM by jjk
                (modified after loading)

Chlorinated pecticides and pcb's method (8081 & 8082). Column RTX-1701(S/N 835482) 30 M X 0.
53 mmID 0.25 micron film.
Splitless using siltex gooseneck liner, 2ul injection.
=====
  
```

External Standard Report

```

=====
Sorted By      :      Signal
Calib. Data Modified : 1/28/2008 11:56:00 AM
Multiplier     :      1.0000
Dilution       :      1.0000
Sample Amount  :      1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: ECD1 B,

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [UG/ML]	Grp	Name
7.906	PB +	5.92567e4	9.81779e-7	5.81770e-2		TCMX (Surrogate)
11.186		-	-	-		alpha-BHC
12.579		-	-	-		gamma-BHC (Lindane)
13.115		-	-	-		Heptachlor
13.908		-	-	-		Aldrin
15.555		-	-	-		beta-BHC
16.069		-	-	-		delta-BHC
16.315		-	-	-		Heptachlor epoxide
17.070		-	-	-		Endosulfan I
17.422		-	-	-		gamma-Chlordane
17.631		-	-	-		alpha-Chlordane
18.064	PB	830.06830	9.95835e-7	8.26611e-4		4,4'-DDE
18.248		-	-	-		Dieldrin
18.853		-	-	-		Endrin
20.483	MF	281.12198	1.13036e-6	3.17768e-4		Endosulfan II
20.597	FM	220.21907	1.59671e-6	3.51626e-4		4,4'-DDD
21.025	MM	1.53559e5	1.07051e-6	1.64387e-1		4,4'-DDT
21.831		-	-	-		Endrin aldehyde
22.844		-	-	-		Endosulfan sulfate
23.098		-	-	-		Methoxychlor
23.856		-	-	-		Endrin ketone
26.548	PB +	5.52554e4	1.00005e-6	5.52582e-2		DCBP (Surrogate)

Totals : 2121.53713

Results obtained with enhanced integrator!
 1 Warnings or Errors :

Warning : Calibrated compound(s) not found

*** End of Report ***



FIGURE 12.1
CHROMATOGRAM OF DDT BREAKDOWN, RTX – 35

Data File E:\HPCHEM\1\DATA\GC1885C\002F0201.D
L143P96

Sample Name: DDT BREAK

```
=====  
Injection Date : 12/11/2007 10:45:21 AM      Seq. Line : 2  
Sample Name    : DDT BREAK                  Location  : Vial 2  
Acq. Operator  : FUZ                       Inj       : 1  
                                           Inj Volume: Manually  
  
Acq. Method    : E:\HPCHEM\1\METHODS\GC1885C.M  
Last changed   : 12/10/2007 2:47:29 PM by FUZ  
Analysis Method : E:\HPCHEM\1\METHODS\GC1885CA.M  
Last changed   : 1/28/2008 12:19:57 PM by jjk  
                (modified after loading)  
Chlorinated pesticide and TOX method 8081/8082. RTX-35 (S/N 824251) 30M X 0.53mm ID 0.50  
micron film. Split/splitless injection.  
=====
```

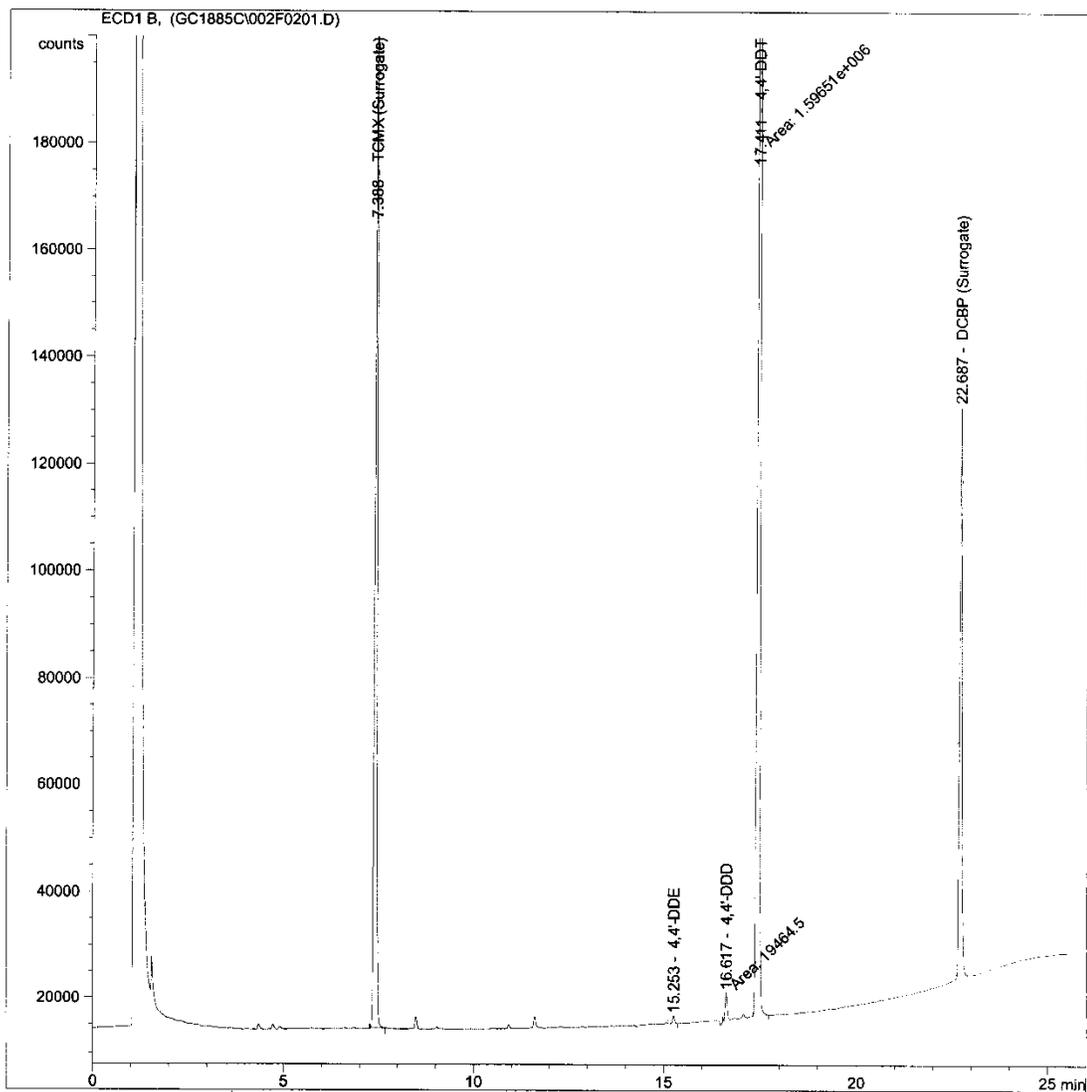




FIGURE 12.2
 CHROMATOGRAM OF DDT BREAKDOWN, RTX – 35

Data File E:\HPCHEM\1\DATA\GC1885C\002F0201.D Sample Name: DDT BREAK
 L143P96

```

=====
Injection Date : 12/11/2007 10:45:21 AM      Seq. Line : 2
Sample Name    : DDT BREAK                    Location  : Vial 2
Acq. Operator  : FUZ                          Inj       : 1
                                           Inj Volume: Manually

Acq. Method    : E:\HPCHEM\1\METHODS\GC1885C.M
Last changed   : 12/10/2007 2:47:29 PM by FUZ
Analysis Method : E:\HPCHEM\1\METHODS\GC1885CA.M
Last changed   : 1/28/2008 12:19:57 PM by jjk
                (modified after loading)
Chlorinated pesticide and TOX method 8081/8082. RTX-35 (S/N 824251) 30M X 0.53mm ID 0.50
micron film. Split/splitless injection.
=====
  
```

External Standard Report

```

=====
Sorted By      : Signal
Calib. Data Modified : 1/28/2008 12:18:24 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount   : 1.00000 [UG/ML] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: ECD1 B,

RetTime [min]	Type	Height [counts]	Amt/Height	Amount [UG/ML]	Grp	Name
7.388	BB +	1.87957e5	2.71608e-7	5.10505e-2		TCMX (Surrogate)
9.295		-	-	-		alpha-BHC
10.368		-	-	-		gamma-BHC (Lindane)
10.598		-	-	-		beta-BHC
11.433		-	-	-		Heptachlor
11.560		-	-	-		delta-BHC
12.318		-	-	-		Aldrin
13.767		-	-	-		Heptachlor epoxide
14.269		-	-	-		gamma-Chlordane
14.692		-	-	-		alpha-Chlordane & Endosulfan I
15.253	VB	1436.34607	3.82271e-7	5.49073e-4		4,4'-DDE
15.471		-	-	-		Dieldrin
16.333		-	-	-		Endrin
16.617	MM	5302.30273	4.71822e-7	2.50174e-3		4,4'-DDD
16.792		-	-	-		Endosulfan II
17.411	MM	4.06680e5	3.89361e-7	1.58345e-1		4,4'-DDT
17.544		-	-	-		Endrin aldehyde
17.947		-	-	-		Endosulfan sulfate
19.358		-	-	-		Methoxychlor
19.683		-	-	-		Endrin ketone
22.687	PBA +	1.06938e5	4.69706e-7	5.02294e-2		DCBP (Surrogate)

Totals : 2.62676e-1

Results obtained with enhanced integrator!
 1 Warnings or Errors :

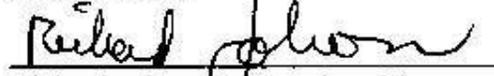
Warning : Calibrated compound(s) not found

*** End of Report ***



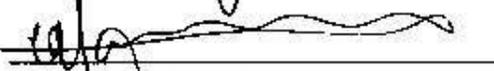
The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

Reviewed By:


Richard Johnson, Operations Manager

22 FEB 08

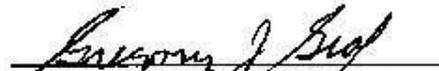
Date


Nick Nigro, President

2/21/08

Date

Approved By:


Gregory V. Graf, Quality Manager

2/21/08

Date



ALKYLATED PAHS

POLYNUCLEAR AROMATIC HYDROCARBONS AND THEIR ALKYLATED HOMOLOGUES BY SELECTIVE ION MONITORING (SIM) GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1 SCOPE AND APPLICATION

This is a capillary GC/MS-SIM method used to determine the concentration of polynuclear aromatic hydrocarbons (PAH) and their alkylated homologues in a variety of matrices. The following compounds may be determined by this method. The alkylated homologues are represented by a group of isomers and there are not CAS registry numbers available.

<u>Compound</u>	<u>CAS. No.</u>
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo(a)anthracene	56-55-3
C1-Benzo(a)anthracene/chrysene	
C2-Benzo(a)anthracene/chrysene	
C3-Benzo(a)anthracene/chrysene	
C4-Benzo(a)anthracene/chrysene	
Benzo(a)pyrene	50-32-8
Benzo(e)pyrene	192-97-2
Benzo(b)fluoranthene	205-99-2
Benzo(ghi)perylene	191-24-2
Benzo(k)fluoranthene	207-08-9
Chrysene	218-01-9
Dibenzo(ah)anthracene	53-70-3
Fluoranthene	206-44-0
C1-Fluoranthene/pyrene	
C2-Fluoranthene/pyrene	
C3-Fluoranthene/pyrene	
Fluorene	86-73-7
C1-Fluorene	
C2-Fluorene	
C3-Fluorene	
Indeno(1,2,3-cd)pyrene	193-39-5
Naphthalene	91-20-3
C1-Naphthalene	
C2-Naphthalene	
C3-Naphthalene	
C4-Naphthalenes	
Perylene	198-55-0
Phenanthrene	85-01-8
C1-Phenanthrene/anthracene	
C2-Phenanthrene/anthracene	
C3-Phenanthrene/anthracene	
C4-Phenanthrene/anthracene	
Pyrene	129-00-0

1.1 This method is based on Method 8270B, which is used to determine semi-volatile



organic compounds in a variety of matrices. This method is applicable to water, soil, and waste samples. Part per billion (ppb) concentrations of the PAHs are determined in soils. Part per trillion (ppt) concentrations can be determined in waters. Concentrations determined in wastes vary depending upon matrix affects and the levels of interferences encountered.

- 1.2 Tables 13 and 14 provide report limits and statistically determined method detection limits (MDLs) for the target compounds in reagent water and silica sand, respectively. Limits of detection and reporting limits are based on the concentration of the low standard using an MDL study to support accuracy and precision of low concentrations.
- 1.3 Typical Initial Demonstration of Capability (IDC) data for reagent water and silica sand are reported in Tables 15 and 16.
- 1.4 The Lead Chemist should be a chromatography expert capable of operating and maintaining the GC/MS system and experienced in the analysis of PAHs.
- 1.5 Quantitative standard solutions are not available for all the homologue configurations possible in a group of alkylated PAHs. However, single component solutions of specific alkylated PAHs are available. This method utilizes a representative single alkylated PAH to represent the entire group of possible compounds in an alkylated homologue group. Similarly, the response factor determined from an initial calibration for this representative PAH is used to quantitate the entire group of homologues in that alkylated group. This approach provides better accuracy than utilizing the response factor of the parent PAH to quantitate the entire groups of homologues. Table 8 provides a list of the representative single alkylated PAH and corresponding homologue group.
- 1.6 Alkylated PAH homologue groups are defined as PAH compounds with alkyl functional groups that differ in the number and length of their sidechain(s). An example is the C4-naphthalene homologue group, which includes any tetra-methyl naphthalene, di-methyl ethyl naphthalene, di-ethyl naphthalene, or butyl naphthalene compounds.

2 SUMMARY OF METHOD

- 2.1 The soils and solid wastes are dried with anhydrous sodium sulfate, and then extracted with a 90% Dichloromethane/10% Acetone mixture using a self-contained mini-extraction. Waters and liquid wastes are extracted by either of two methods. Water method A is self contained and uses hexane as the extraction solvent with no concentration. Water method B extracts water with dichloromethane in a separatory funnel and concentrated with a TurboVap[®] concentrator. An aliquot of the soil, water, or waste extract is transferred to an injection vial and 2 μ L of the extract is injected into the GC/MS system. A narrow-bore capillary column in the gas chromatograph (GC) is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS). The MS is operated in the SIM mode



and only the response of specific ions for the PAHs is measured. A single select alkylated homologue (when available) is used to calculate an average response factor or standard curve which is then applied to the entire homologue group for sample quantitation.

- 2.2 Identification of target analytes is accomplished by comparing the relative response of specific target and qualifier ions of authentic standards or a standard reference material containing the alkylated homologues. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using average response factor or standard curve calculation methodology.
- 2.3 The method includes specific calibration and quality control steps.

3 INTERFERENCES

- 3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Poor quality matches of qualifier ion ratios relative to the target ion used for quantitation may indicate the presence of interferences in the sample.

Analysis of method and reagent blanks provides information about the presence of contaminants in the extraction procedure. When potential interfering peaks are noted in method blanks, the analyst should analyze additional blanks to identify the source of contamination and eliminate the problem.

- 3.2 Contamination may occur when a sample containing low concentrations of PAHs is analyzed immediately after a sample containing high concentrations of PAHs. After analysis of a sample containing high concentrations of PAHs, an instrument blank should be analyzed to check for cross contamination. Cross-contamination is minimized in the extraction procedure for soils with single use glassware.
- 3.3 Visual screening of the sample extracts prior to GC/MS analysis is advantageous for identifying samples containing high concentrations of PAHs and then calculating necessary sample dilutions. Soil sample extracts that are dark and discolored usually indicate high levels of PAHs and an initial dilution should be performed on the extract. Water extracts using Method B that are dark and discolored may be only concentrated to a final volume of 10 mL rather than the normal 2 mL.
- 3.4 This method integrates the response of selective ion chromatograms over a wide time range representative for the elution of a diverse group of isomers present in a particular homologue group. Other compounds may elute within this window and result in false positive results, biased high results or poor qualifier ion matches. Interpretation of the selective ion chromatograms for a homologue group weighs heavily upon the experience of the analyst.

Results for specific analytes within a sample that have atypical ion ratios will be qualified with an "N1" as tentative identifications.



4 APPARATUS AND MATERIALS

- 4.1 Gas chromatograph: HP 5890A equipped with a split/splitless-injector
 - 4.1.1 Carrier Gas: Helium
 - 4.1.2 LEAP Autosampler A200SE
- 4.2 Capillary Column: 30 m x 0.32 mm I.D., 0.25 μ film, Rtx-5ms capillary column, Restek Part #
- 4.3 Mass Spectrometer: HP5972 mass spectrometer capable of scanning from 35 to 500 amu every 1 sec. or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is tuned using the maximum sensitivity auto tune program.
- 4.4 Data system: Agilent MS ChemStation utilizing EnviroQuant data analysis software that allows the continuous acquisition and storage of mass spectra obtained throughout the duration of the gas chromatographic program. Reference mass spectra of all analytes are generated from the analysis of calibration standards.
- 4.5 Dispenser: EMD Optifix[®] or equivalent, capable of accurately delivering 10.0 mL of solvent
- 4.6 Balances:
 - 4.6.1 Top Loader capable of weighing to 0.01 gram
 - 4.6.2 Analytical capable of weighing to 0.0001 gram
- 4.7 Micro-syringes: 10, 25, 100, 250, 500, and 1,000 μ L, usually Gas-Tight[®]
- 4.8 Glass scintillation vials: 20 mL, with polypropylene screw-caps for solvent extraction of soils
- 4.9 Spatula: Stainless steel
- 4.10 Eppendorf Air displacement pipetter with 1 ml disposable tips
- 4.11 Eppendorf 10-250 μ L repeater with various size disposable Combitips[®]
- 4.12 1.8 ml amber GC injection vials with Teflon[®]/silicon lined caps
- 4.13 Separatory Funnels with Teflon[®] Stopcock: Glass 1L and 250 mL capacity
- 4.14 TurboVap[®] Concentrator with connection to a dewar of liquid nitrogen for supply of dry down gas
- 4.15 TurboVap[®] Concentrator Tubes: glass 200 mL capacity for concentration of water



extracts

- 4.16 Volumetric Glassware: various sizes, (Class A)
- 4.17 2 mL volumetric flasks for volumetric determination of water extracts
- 4.18 Glass disposable Pasteur pipettes: 5 ¾” and 9” lengths
- 4.19 Volumetric pipettes: various sizes, 1 to 50 mL capacity (Class A)

5 REAGENTS

5.1 Solvents

- 5.1.1 Iso-octane - pesticide quality or equivalent
- 5.1.2 Acetone - pesticide quality or equivalent
- 5.1.3 Carbon Disulfide - pesticide quality or equivalent
- 5.1.4 Dichloromethane - pesticide quality or equivalent
- 5.1.5 Hexane - pesticide quality or equivalent
- 5.1.6 90% Dichloromethane/10% Acetone: With a 2 L graduated cylinder measure 1800 mL of dichloromethane and 200 mL of acetone. Mix the two solvents in an empty 4 L dichloromethane solvent bottle. Record the preparation in LIMS under Laboratory>Standards.
- 5.1.7 Keeper Solvent – 80% Iso-octane/20% Acetone: Combine 3200 mL of Iso-octane with 800 mL of Acetone in a 4 L solvent bottle. Label appropriately and assign a 6 month expiration date. Record the preparation in LIMS under Laboratory>Standards.

5.1.8 Acids and Bases

- 5.1.8.1 10 N Sodium Hydroxide, NaOH – purchased from VWR
- 5.1.8.2 Concentrated sulfuric acid (H₂SO₄), ACS reagent grade or better
- 5.1.8.3 12 N Sulfuric Acid: Carefully and slowly add 333 mL of concentrated sulfuric acid to approximately 500 mL of DI water in a 1 L volumetric flask. Use of an ice bath is encouraged to help dissipate the heat generated from this dilution. Allow to cool and make to approximately 1 L with DI water, allowing the solution to cool again. Final room temperature volume is 1 L. Record the preparation in LIMS under Laboratory>Standards. Transfer to a labeled 1 L container and store in the refrigerator.

5.2 Sodium Sulfate-reagent grade



- 5.2.1 Sodium sulfate (Na_2SO_4) is baked in a muffle furnace at 400 °C overnight prior to use. Store in sealed bottles away from moisture prior to use.
- 5.3 Silica Sand
- 5.3.1 Silica sand is baked in a muffle furnace at 400 °C overnight prior to use. Store in sealed bottles away from moisture
- 5.4 Sodium Chloride-reagent grade
- 5.4.1 Sodium Chloride is baked in a muffle furnace at 400 °C overnight prior to use. Store in sealed bottles away from moisture.
- 5.5 Stock PAH Standard Solutions - Stock solutions of PAHs are purchased from Absolute Standards, AccuStandard, or ChemService at certified concentrations. Sixteen of the parent PAHs are purchased as a mix, each at a concentration of 2,000 $\mu\text{g}/\text{mL}$. Three of the PAHs, benzo(e)pyrene, 1-methyl naphthalene, and 2-methyl naphthalene, are purchased as individual solutions at a concentration of 1,000 $\mu\text{g}/\text{mL}$. Many of the alkylated PAHs are purchased from AccuStandard at a concentration of 50 $\mu\text{g}/\text{mL}$, except 2,3,5-Trimethyl naphthalene which is purchased from ChemService at a concentration of 100 $\mu\text{g}/\text{mL}$. Table 3 details the Absolute, AccuStandard or ChemService part numbers and lists the PAHs included in these stock standards. These standards may be used up to the expiration date provided by the vendor.
- 5.5.1 Neat PAH Standards – five of the alkylated PAHs are purchased as neat standards. These PAHs include 1,8-Dimethyl fluorene, 9-Ethyl-10-Methyl phenanthrene, 1-Methyl fluorene, 2-(t-Butyl) Anthracene, and 2,6-Diethyl naphthalene. The neat materials are purchased from Aldrich Chemical. Weigh 0.0500 g of each compound into a glass scintillation vial using an analytical balance. Add 20 mL of dichloromethane to each vial. Cap and sonicate each mixture until the compounds are completely dissolved. Quantitatively transfer the solutions to a 100 mL volumetric flask containing 20 mL Carbon Disulfide with a glass Pasteur pipette, rinsing the vials with three 2 mL portions of dichloromethane, and dilute to volume with dichloromethane. The resulting stock solution will contain each alkylated PAH at a concentration of 500 $\mu\text{g}/\text{mL}$. These stock standards may be used for one year and is stored in a refrigerator. Table 3 provides details for the neat standards.
- 5.5.2 Purity of Stock Standards – the stock solutions were analyzed by GC/MS operated in the scan mode to access purity of the standards. Peak areas from the total ion chromatograms were measured from the stock standard analysis. Two of the stock solutions were found to be less than 95% pure. These stock solutions included 1,8-Dimethyl fluorene at 86.6% and 9,10-Dimethyl anthracene at 24.1%. Adjustment to the volumes used in preparing the intermediate calibration standard (Section 5.9) and matrix spike solution is necessary based upon this purity.
- 5.6 Stock Surrogate Standard Solution (p-Terphenyl- d_{14} , 2000 $\mu\text{g}/\text{mL}$): The surrogate



standard solution is prepared from neat materials. The surrogate standard is p-Terphenyl-d₁₄. The neat material is purchased from CDN Isotopes as part # D-87. Weigh 0.2000 g of the neat material into a glass scintillation vial using an analytical balance. Add 8 mL of carbon disulfide and 8 mL of dichloromethane to the vial. Cap and sonicate the mixture until the p-Terphenyl is completely dissolved. Quantitatively transfer the solution from the vial to a 100 mL volumetric using a glass Pasteur pipette. Rinse the vial with three 2 mL portions of dichloromethane and add these to the flask. Add an additional 12 mL of carbon disulfide to the flask and bring to volume with dichloromethane. The resulting solution should be 20% carbon disulfide in dichloromethane. The resulting concentration of the stock surrogate standard is 2,000 µg/mL (See Table 4). The standard may be used for one year and it is stored in a refrigerator.

5.7 Stock Internal Standard Solution (750 µg/mL): The stock internal standard solution is prepared from neat materials. The internal standards are acenaphthene-d₁₀, chrysene-d₁₂, and perylene-d₁₂. The neat materials are also purchased from CDN Isotopes. Weigh 0.0750 g of each compound into separate glass scintillation vials using an analytical balance. Add 5 mL of carbon disulfide and 5 mL of dichloromethane to each vial. Cap and sonicate each mixture until the compounds are completely dissolved. Quantitatively transfer the solutions to a 100 mL volumetric flask with a glass Pasteur pipette, rinsing the vials with three 2 mL portions of dichloromethane. Add an additional 15 mL of carbon disulfide to the volumetric flask and dilute to volume with dichloromethane. The final solvent should be 20% carbon disulfide in dichloromethane. The resulting stock solution will contain each internal standard at a concentration of 750 µg/mL (See Table 5). This stock standard may be used for one year and is stored in a refrigerator.

5.8 Working Surrogate Solutions

5.8.1 Soils (50 µg/mL): Using a 2 mL volumetric pipette and a 500 µL syringe, accurately transfer 2.5 mL of the stock surrogate standard solution (Section 5.6) to a 100 mL volumetric flask and dilute to volume with acetone. The resulting concentration of the working surrogate standard is 50 µg/mL. This standard may be used for one year and is stored in a refrigerator (See Table 4).

5.8.2 Waters (5.0 µg/mL): Using a 10 mL volumetric pipette, accurately transfer 10 mL of the working surrogate standard solution for soils (Section 5.8.1) to a 100 mL volumetric flask and dilute to volume with acetone. The resulting concentration of the working surrogate standard is 5.0 µg/mL. This standard may be used for one year and is stored in a refrigerator (See Table 4).

5.9 Intermediate Calibration Standard: Using a 2 mL volumetric pipette add 2 mL of the working surrogate solution (Section 5.8.1) to a 50 mL volumetric flask. Using a 100 µL syringe, accurately add 50 µL of the stock PAH mix (Section 5.5) at 2,000 µg/mL. Using a 100 µL syringe, accurately add 100 µL of each of the three individual PAH stock standards (Section 5.5) at 1,000 µg/mL. Using a 1 mL syringe, accurately add 2 mL of each of the alkylated PAH stock solutions at 50 µg/mL (Section 5.5), except



- 9,10-Dimethyl anthracene. Add 8.30 mL of 9,10-Dimethyl anthracene due to its low purity at 24.1% (Section 5.5.2). Add 1 mL of the alkylated PAH stock solution at 100 $\mu\text{g/mL}$ (Section 5.5). Using a 250 μL syringe, add 200 μL of each of the alkylated PAH standards at 500 $\mu\text{g/mL}$ prepared from neat materials (Section 5.5.1), except 1,8-Dimethyl fluorene. Add 231 μL of 1,8-Dimethyl fluorene due to its low purity at 86.6 % (Section 5.5.2). Bring the 50 mL volumetric flask to volume with 90% Dichloromethane/10% Acetone. The final concentration of the PAHs and surrogate standard is 2.0 $\mu\text{g/mL}$ (see Table 6). This standard may be used for one year and is stored in a refrigerator.
- 5.9.1 Working Calibration Standards: The working calibration standards are prepared at seven levels by diluting the intermediate calibration standard (Section 5.9). Using a 100 μL syringe, accurately add 250 μL of the intermediate calibration standard (Section 5.9) to a 25 mL volumetric flask and bring to volume with 90% Dichloromethane/10% Acetone. This completes preparation of the Level 3 working calibration standard at 20 ng/mL. The remaining levels include 5, 50, 100, 200, 500 and 1,000 ng/mL. Table 7 describes preparation of the seven levels of working calibration standard.
- 5.10 Working Internal Standard Solution: The working internal standard solution is prepared by diluting the stock internal standard (Section 5.7) in a 50 mL volumetric flask. Using a 5 mL volumetric pipette add 5 mL of the stock internal standard solution (Section 5.7) to a 50 mL volumetric flask. Bring to volume with acetone. The resulting concentration of the working internal standard solution is 75 $\mu\text{g/mL}$ (See Table 5).
- 5.11 Matrix Spike/LCS Solution: The matrix spike solution is prepared at 10 $\mu\text{g/mL}$ by dilution of stock solutions of PAHs and select representative alkylated PAHs. Not all of the representative alkylated PAHs are included in the matrix spike solution. Using a 250 μL syringe, accurately measure 250 μL of the stock PAH stock standard (16 PAHs) at 2,000 $\mu\text{g/mL}$ (Section 5.5) into a 50 mL volumetric flask. Using a 500 μL syringe, accurately add 500 μL of each of the three PAHs (benzo(e)pyrene, 1-methyl naphthalene, 2-methyl naphthalene) at 1,000 $\mu\text{g/mL}$ (Section 5.5) to the flask. Using a 1 mL syringe, add 1.0 mL of each of the alkylated PAH standards at 500 $\mu\text{g/mL}$ prepared from neat materials (Section 5.5.1) and add 1.15 mL of 1,8-Dimethyl fluorene at 433 $\mu\text{g/mL}$. Table 10 describes preparation of the matrix spike solution.
- 5.12 Alkylated PAH Homologue Reference Material: The reference standard is prepared from contaminated soil collected at a coal burning energy facility. Weigh 30 grams of contaminated soil in a 250 mL beaker and mix with 50 grams of anhydrous sodium sulfate until dry. Add 100 mL of 90% Dichloromethane/10% Acetone, mix and sonicate the soil for 15 minutes. Filter the extract into a TurboVap tube through a qualitative filter paper with a layer of anhydrous sodium sulfate seated on a glass funnel. Concentrate the extract to ~ 15 mL at 30 °C and 10 psi. Dilute the extract to a final volume of 25 mL.
- 5.13 Second Source (200 ng/mL): A second source standard is prepared from stock



standards from a different manufacturer and/or a second independent weighing of neat materials by a second analyst.

NOTE: Some of the alkylated PAH standards may not be available from different sources and this is acceptable.

6 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 6.1 Samples are commonly collected by the field consultant. Every effort is made by the laboratory to supply the consultant with the appropriate containers and coolers in planning the field sampling event. A Chain of Custody accompanies the bottle order to be filled out by the consultant.
- 6.2 Soil samples are typically collected in 4 oz wide mouth amber jars with Teflon[®]-lined caps. The samples are stored in a cooler on ice during shipment. If ice remains in the cooler upon receipt at the laboratory, the sample custodian specifies “received on ice” on the chain of custody. Samples that are delivered to the fixed base lab on the same day as they are collected are likely not to have reached a fully chilled temperature. This is acceptable if there is evidence that chilling has begun. Samples that are received on site by a field laboratory within 15 minutes of collection do not require chilling. The laboratory will place the samples into the refrigerator upon receipt. Once received by the laboratory, the samples are stored in a refrigerator at 4 °C.
- 6.3 Water samples are collected in 1 L amber wide mouth bottles with Teflon[®]-lined caps. The consultant is advised to collect one sample in triplicate for analysis of matrix spike samples. Water samples are typically shipped on ice in a cooler. Once received by the laboratory, the waters are stored in a refrigerator at 4 °C.
- 6.4 Soil samples must be extracted within 14 days after collection. Soil extracts must be analyzed within 7 days after extraction. Water samples are unpreserved and must be extracted within 7 days after collection. Water extracts must be analyzed within 7 days after extraction.

7 PROCEDURE

- 7.1 Self Contained Extraction for Soils:
 - 7.1.1 Accuracy of the Top Loading Balance: The top loading balance should be capable of measuring weights to the hundredths (0.00) of a gram. Verify the accuracy of the top loading balance with an appropriate size weight.
 - 7.1.2 Weigh 5.0 g of the soil sample into a tared 20 mL scintillation extraction vial. For every twenty samples extracted, weigh one sample in triplicate for use as a matrix spike/matrix spike duplicate (MS/MSD) sample. Also for every twenty samples extracted, prepare one laboratory control sample and soil blank consisting of 5 grams of silica sand. Add 5 or more grams of anhydrous sodium sulfate to dry the soil samples and mix well. Allow approximately 20 minutes or longer for the soil to dry. The sample should pour like salt when dry. Using a repeater and a 2.5 mL



tip, add 50 μL of the working surrogate standard solution (Section 5.8) to all soil, matrix spike, laboratory control, and blank samples. For matrix spike, matrix spike duplicate, and laboratory control samples also add 200 μL of the matrix spike standard (Section 5.11). Add 10.0 mL of the 90% Dichloromethane/10% Acetone extraction solvent, and shake for 30 seconds, allow to settle for ten minutes, shake again for 15 seconds, and allow settling for two minutes.

- 7.1.3 Soil extracts may be left in the 20 mL scintillation vial and/or using a disposable Pasteur pipette transfer the soil extract into a 12 mL amber dram vial, being careful not to include any of the settled soil. Store the extracts in a freezer until proceeding to the GC/MS analysis.

7.2 Extraction of Water Samples

7.2.1 Method A – Self Contained Bottle Extraction

- 7.2.1.1 Prepare a minimum of one blank, one LCS, and a MS/MSD with each set of water samples extracted. If more than 20 samples are to be extracted, prepare a new blank and LCS with each group of 20 or less samples. Use deionized water for the blank and LCS.
- 7.2.1.2 Transfer 800 grams or mL of water to a 1 L narrow mouth bottle containing 280 grams of purified NaCl.
- NOTE: Check the pH of the sample with wide-range pH paper and, if necessary, adjust the pH to 6-8 using 12 N sulfuric acid or 10 N sodium hydroxide prior to the addition of hexane.
- 7.2.1.3 Cap and shake the container to dissolve most of the NaCl. Not all of the NaCl will dissolve.
- 7.2.1.4 To all samples and QC samples add 250 μL of Surrogate Spike 5.0 $\mu\text{g}/\text{mL}$ (Section 5.8.2).
- 7.2.1.5 Spike the MS/MSD/LCS samples with 100 μL of matrix spiking standard at 10 $\mu\text{g}/\text{mL}$ (Section 5.11).
- 7.2.1.6 Add 5 mL of hexane to every sample and shake vigorously for a minimum of 45 seconds.
- 7.2.1.7 Allow sample to stand for a minimum of 10 minutes and repeat the shaking again for a minimum of 45 seconds.
- 7.2.1.8 Allow sample to stand for a minimum of 10 minutes and add adequate DI water to bring the solvent up into the neck of the extraction vessel.
- 7.2.1.9 Transfer most of the hexane extract to a 20 mL scintillation vial containing a small amount of Na_2SO_4 . The solvent is not concentrated prior to sample



analysis. Leave the extract in the scintillation vial for storage.

NOTE: Do not transfer any of the sample water to the scintillation vial.

NOTE: If an emulsion occurs, pull the emulsion up into a disposable pipette and squirt back into the bottle. Doing this action several times will usually break up the emulsion adequately to obtain solvent to inject. This process may have to be repeated several times and by adding additional water to the bottle. Please exercise patience and maintain attention to detail.

7.2.1.10 The sample is now ready for Sample Analysis (Section 7.6).

7.2.2 Method B – Separatory Funnel Extraction

NOTE: Method B should be used for all fixed-base extractions, where appropriate glassware and concentrators are available.

7.2.2.1 Prepare a minimum of one blank, MS/MSD and LCS with each set of water samples extracted. If more than 20 samples are to be extracted, prepare a new blank, MS/MSD and LCS with each group of 20 samples. Use 1000 mL of reagent grade water for the blank and LCS.

7.2.2.2 Using a 1 L graduated cylinder, measure 1 L (nominal) of sample and transfer it quantitatively to the 2 L separatory funnel. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 L.

Or

Transfer the contents of the 1 L bottle as received (mark the meniscus of the water on the bottle with black marker prior to transferring) into a 2 L separatory funnel. Fill the container with tap water to the black mark after completing Step 7.2.8 and pour into a 1 L graduated cylinder. Record the volume of water in the LIMS bench sheet.

7.2.2.3 Check the pH of the sample with wide-range pH paper and, if necessary, adjust the pH to 6-8 using 12 N sulfuric acid or 10 N sodium hydroxide prior to the addition of dichloromethane.

7.2.2.4 Add 100 g of sodium chloride to the separatory funnel. Dissolve the sodium chloride.

7.2.2.5 With a repeater, add 100 μ L of the surrogate standard solution for waters (See Section 5.8.2) to all unknowns and quality control samples.

7.2.2.6 For the LCS sample, add 40 μ L of the LCS spiking standard solution (See Section 5.11).



7.2.2.7 For the MS/MSD samples in each analytical batch, add 40 μL of the matrix spiking standard to each sample (See Section 5.11).

7.2.2.8 Add 60 mL of dichloromethane to the sample container (to rinse the container) and transfer to the separatory funnel.

NOTE: Sample container is only rinsed with the first aliquot of dichloromethane.

7.2.2.9 Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

NOTE: Dichloromethane creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted once. The separatory funnel should be vented into a hood to avoid exposure of the analyst to solvent vapors.

7.2.2.10 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. The dichloromethane extract is dried by passing it through a funnel containing glass wool and ~ 15 to 20 grams of sodium sulfate. The solvent extract is collected in a 200 mL Zymark evaporation tube.

NOTE: If an emulsion interface occurs between the layers, the analyst must employ physical techniques to complete the phase separation. The optimum technique depends upon the sample and usually uses one of two techniques. If the emulsion is small, you may drain it through sodium sulfate. Mix the sodium sulfate to ensure that it doesn't form a large clump. If the emulsion is half of the dichloromethane layer or larger, you may drain dichloromethane and the emulsion into a second, smaller (usually 250 mL) separatory funnel. Shake vigorously. If the emulsion breaks, allow to settle and drain through the sodium sulfate into the labeled Zymark. If the emulsion doesn't break, add more dichloromethane to the second separatory funnel. Shake vigorously. Repeat addition of dichloromethane, shaking until emulsion breaks. Once the second separatory funnel technique has been chosen, both of the next extracts (Sections 7.2.2.11 and 7.2.2.12) must go through the second separatory funnel.

7.2.2.11 Repeat the extraction using a second 60 mL aliquot of dichloromethane. Follow Sections 7.2.2.9 and 7.2.2.10.

7.2.2.12 Repeat the extraction using a third 60 mL aliquot of dichloromethane. Follow Sections 7.2.2.9 and 7.2.2.10. Rinse the separatory funnel with 20 mL of dichloromethane and dry as in Section 7.2.2.10.

7.3 Zymark TurboVap[®] Concentration for Water Extracts (Only applies to Method B Water Extraction)

7.3.1 Set the TurboVap[®] to 30 °C with nitrogen flowing at 10 to 12 psi.



7.3.2 Evaporate the solvent until ~ 8 mL of solvent remains. If the extract appears clear at this point proceed with the evaporation and skip to Section 7.3.3. If the extract is colored continue to Section 7.3.2.1.

7.3.2.1 Transfer the extract to a 10 mL volumetric flask, rinsing the Zymark tube with several small portions of 90% Dichloromethane/10% Acetone. Invert to mix 3 times. Transfer the extract to an amber storage vial with Teflon[®]-lined cap and store in freezer.

7.3.3 Add 1.5 mL of 80% Iso-octane/20% Acetone and continue the evaporation. When the remaining solvent only occupies the bottom nipple of the TurboVap[®] tube remove the tube from the TurboVap[®].

NOTE: Care must be taken during all evaporation steps to ensure the minimum volumes specified are not exceeded. If the volume goes below the minimum, naphthalene recoveries will be < 60% and many other compound recoveries will also be reduced.

7.3.4 Draw up the remaining sample extract in the TurboVap[®] tube with a 9 inch Pasteur pipette and transfer to a 2 mL volumetric flask. Rinse only the bottom 10 mL portion of the wall of the tube with 300 μ L of 90% Dichloromethane/10% Acetone solvent and transfer this rinse to the 2 mL vial. Add the 90%/10% solvent to the volumetric flask to a final volume of 2 mL. Cap and invert the vial 3 times to mix. Transfer the extract to an amber storage vial with Teflon[®]-lined cap and store in freezer. Store the extract in a freezer until proceeding to the GC/MS analysis.

7.4 GC/MS Conditions

7.4.1 GC/MS Pump Down: Allow the GC/MS system to stabilize under high vacuum when calibrating from a vented state. Before initial calibration, allow at least 4 hours for the MS to stabilize after pump-down.

7.4.2 GC Conditions:

Column:	RTX-5ms, 30 m x 0.32 mm, 0.25 μ film
Injector Temp:	300 °C
Transfer Line Temp:	320 °C
Column Head Pressure:	5 psi
Column Flow:	2 mL/min
Split Flow:	35 mL/min
Septum Purge:	1.5 mL/min
Splitless Injection:	2 μ L volume
Split Vent:	Initial State: Off Final State: On after 1.5 minutes
Oven Temperature Program	
Initial Temperature:	80 °C
Initial Hold:	1.5 minutes



First Temperature Ramp: 22 °C/ minute
First Final Temperature: 150 °C
Second Temperature Ramp: 8 °C/minute
Second Final Temperature: 300 °C
Third Temperature Ramp: 22 °C/ minute
Third Final Temperature: 340 °C
Final Hold Time: 2.0 minutes

7.4.3 MS Conditions

Tune: Maximum Sensitivity Autotune
EM Voltage: 2200 (200 above Autotune)
Scan Mode: Selective Ion Monitoring
Resolution: Low
Solvent Delay: 3.5 minutes

SIM Group: 1
Start Time: 3.5 min
Mass/Dwell Time(ms): 164/20 162/20 128/20
129/20 142/20 141/20
156/20 170/20 155/20
184/20 169/20 152/20
151/20 154/20 153/20
166/20 165/20 180/20
194/20 179/20 208/20
193/20 178/20 192/20
191/20

SIM Group: 2
Start Time: 11.30 min
Mass/Dwell Time(ms): 208/20 193/20 240/20
236/20 192/20 191/20
206/20 220/20 205/20
234/20 219/20 202/20
200/20 216/20 215/20
230/20 244/20 229/20
122/20 228/20 229/20
242/20 241/20 256/20
270/20 255/20 284/20
269/20 194/20 179/20

SIM Group: 3
Start Time: 19.50 min
Mass/Dwell Time(ms): 256/35 241/35 270/35
255/35 284/35 269/35
264/35 260/35 252/35
253/35 125/35 278/35



139/35	279/35	276/35
138/35	277/35	138/35
242/35		

7.5 GC/MS Calibration

7.5.1 Maximum Sensitivity Autotune. The GC/MS system is software-tuned by successfully completing a maximum sensitivity autotune. Perfluorotributylamine (PFTBA) is used as the tuning compound. A hardcopy report is generated from the tune (See Figure 1). This tune should be performed prior to each initial calibration. The tune report serves as a useful diagnostic tool.

7.5.1.1 Tune calibration check decafluorotriphenylphosphine (DFTPP) is required to be injected once every 12 hours and must pass acceptance criteria.

7.5.1.2 Detection of Leaks in the GC/MS System: The presence of a base peak at m/z 28 (N_2) that is higher than m/z 69 (base peak of PFTBA) in the tune report indicates the presence of a gross leak in the GC/MS vacuum system. A common source of the leak is a loose transfer line nut sealing the GC capillary column to the MS transfer line. Tightening this nut often eliminates a leak.

7.5.1.3 Water in the GC/MS System: The presence of a base peak at m/z 18 that is higher than m/z 69 in the tune report indicates excessive water remains in the MS manifold. The MS vacuum system does not efficiently remove water and this condition indicates that a longer equilibration time is needed prior to initial calibration.

7.5.1.4 Electron Multiplier Voltage: An electron multiplier voltage higher than 2700 in the tune report indicates that the multiplier needs replacement and/or the MS source needs cleaning.

7.5.1.5 Peak Shape/Resolution of PFTBA Calibration Peaks: The appearance of the PFTBA peaks (m/z 69, 219, 502) used to calibrate the MS should be symmetric without any shoulders. Isotope masses of these same peaks (m/z 70, 220, 503) should be present and indicated in the autotune report. Non-symmetric peak shape or the non-detection of isotope masses usually indicates that the MS source needs to be cleaned.

7.5.2 Calibration is performed using the internal standard technique. Typically seven different levels of calibration standards are used to calculate an average response factor.

Add 20 μ L of the working internal standard solution (See Section 5.10) to a 2 mL injection vial. Transfer 0.80 mL of each calibration standard to the vial. Prepare a row of five vials at a time. Make sure all vials to be analyzed are prepared the same.



7.5.3 Calibration: Inject each calibration standard, collect the data and tabulate the area response of the characteristic ions against the concentration for each target compound and each internal standard. Characteristic ions are listed in Table 1. The internal standard selected for the calculation of these ratios should be the internal standard that has a retention time closest to the compound being measured. The internal standard used for each target compound is provided in Table 11. Calculate amount ratios and response ratios for each target compound and at each calibration level relative to the appropriate internal standard. The amount ratio and response ratios are calculated as follows:

$$\text{Amount Ratio} = \frac{C(s)}{C(is)} \quad \text{Response Ratio} = \frac{A(s)}{A(is)}$$

Where:

- A(s) = Peak area of the target compound or surrogate.
- A(is) = Peak area of the internal standard.
- C(s) = Concentration of the target compound or surrogate in ng/mL.
- C(is) = Concentration of the internal standard in ng/mL

7.5.3.1 Calculation of Response Factors (RF) and Relative Standard Deviation (% RSD): Calculation of response factors are used to evaluate the initial calibration against specific criteria and are also used to quantitate sample concentrations. Individual response factors are calculated by the following formula for each calibration level:

$$\text{Response Factor (RF)} = \frac{\text{Response Ratio}}{\text{Amount Ratio}}$$

The % RSD is calculated by the following formula:

$$\% \text{ RSD} = (RF_{STDDEV} / RF_{AVE}) \times 100$$

Where:

- RF_{STDDEV} = Standard Deviation of Individual Response Factors
- RF_{AVE} = Average of Individual Response Factors

Typical response factors calculated for target compounds are listed in Table 12. Response factors used for the alkylated homologue groups are those determined from their representative alkylated PAHs (Table 8), except as described below in Section 7.5.3.2.

NOTE: ChemStation prints a report for Response Factors and % RSD. The report only lists 6 standards but all are used to calculate the mean and % RSD. The response factor report must be in all raw data packets.

7.5.3.2 Fixed Response Factors: When a representative PAH is not available for a



homologue group, the group's response factor has been permanently fixed based upon the best available data and is listed in Table 12. Those alkylated homologue groups without representative alkylated PAHs include C-3 Fluorene, C2-Fluoranthene/Pyrene, C3-Fluoranthene/Pyrene, and C4-Benzo(a)anthracene/Chrysene. Only Level 1 in the calibration table is used for these homologue groups. All other areas automatically updated in the other levels must be manually deleted with each initial calibration. The peak area response for Level 1 must be manually updated with each initial calibration depending upon the new peak area response of the appropriate internal standard by the following equation:

$$A_{(HG)} = A_{(IS)} \times RF_{(HG)}$$

Where:

- $A_{(HG)}$ = Peak Area Response for the Alkylated Homologue Group Manually Updated in Calibration Table, Level 1
- $A_{(IS)}$ = Peak Area Response of the Appropriate Internal Standard in Level 1 from the Initial Calibration
- $RF_{(HG)}$ = Fixed Response Factor for the Alkylated Homologue Group as listed in Table 12

7.5.3.3 Fixed Relative Abundance of Qualifier Ion Ratios: Ion ratios are updated with each initial calibration using the CCV level at 200 ng/mL. Typical relative abundances of qualifier ions are provided in Table 1. Ion ratios from the representative alkylated PAH (Table 8) are used to update alkylated PAH homologue groups, except for those groups that do not have a representative alkylated PAH. Those alkylated homologue groups without representative alkylated PAHs include C-3 Fluorene, C2-Fluoranthene/Pyrene, C3-Fluoranthene/Pyrene, and C4-Benzo(a)anthracene/Chrysene. Ion ratios for these groups must be updated manually with the fixed ion ratios provided in Table 1.

7.5.3.4 Acceptability of Initial Calibration: The % RSD of the response factors must be $\leq 20\%$ for individual PAHs and representative alkylated PAHs.

If an analyte has a % RSD $> 20\%$, the analyst may drop one of the standards to obtain an acceptable % RSD. The analyst must document why they chose to drop that particular standard (i.e. highest value, lowest value, some percentage higher or lower than the other eight values).

The analyst has other choices to obtain an acceptable calibration for a particular analyte up to and including use of regression analysis (i.e. linear and/or quadratic with no weighting factor or a $1/x$ weighting factor). To repeat dropping of a standard requires documentation.



If the analyst must drop more than one standard, evaluate the GC/MS for possible maintenance issues.

If the standard to be dropped is the upper point of the curve, then the highest calculated analyte in an unknown becomes the highest acceptable standard in that analyte's remaining standard curve. Due to the number of standards in each standard curve dropping one midpoint standard is acceptable assuming a valid reason is given. If the lowest standard is dropped, the analyst has the following options, 1) raise the reporting limit up to the level of the next lowest acceptable standard, 2) Reinject the lowest standard later in the run and include the reinjection in the standard curve (Note this reinjection is only allowed once), 3) As a last possibility use the lower reporting limit but qualify the data between the lowest acceptable standard and the reporting limit.

- 7.5.4 Continuing Calibration Verification: The working calibration curve must be verified after every ten sample injections and on each working day by the injection of continuing calibration verification (CCV) standards. The concentration of the CCVs are 50 and/or 200 ng/mL. The Percent Difference between the concentration reported for the PAHs in the CCV standard and the theoretical concentration of 50 and/or 200 ng/mL must not vary by more than 20% in order for the calibration to remain valid. If the Percent Difference of any compound varies by more than 20%, then a new calibration curve must be prepared and affected samples reanalyzed. If reanalysis is not an option, the analytical results shall be qualified as estimated.

$$\text{Percent Difference} = \left(\frac{|Conc_{Exp} - Conc_{Act}|}{Conc_{Exp}} \right) \times 100$$

Where:

Conc_{Exp} = Expected concentration of target compound from CCV
Conc_{Act} = Actual calculated concentration of target compound from CCV

7.6 Sample Analysis

- 7.6.1 Samples are analyzed in a group referred to as an analytical run. The sequence begins with instrument calibration followed by sample extracts interspersed with CCV standards. The sequence ends when the entire sequence has been injected or when qualitative and/or quantitative QC criteria are exceeded. All sequences must end with a CCV as the last injection.
- 7.6.2 Add 20 µL of the working internal standard solution (See Section 5.10) to a 2 mL injection vial. Transfer exactly 0.80 mL of each sample to the vial. Prepare vials in sets of five at a time. Prepare each vial (standard, sample or QC sample) the same. Inject the samples and collect and process the data using a chromatography workstation.



- 7.6.3 High levels of PAHs in samples must be diluted to not exceed the upper limit of the calibration curve. Concentrations of parent PAHs in a sample analysis must not exceed 2,000 ng/mL or the highest level used for calibration. Concentrations of alkylated PAH homologue groups must not exceed the levels listed in the table below:

Degree of Alkylation	Maximum Sample Concentration (ng/mL)
C1	4,000
C2	8,000
C3	10,000
C4	15,000

7.7 Qualitative Analysis

- 7.7.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the extracted ion chromatograms of the sample with the characteristic extracted ion chromatograms from a reference standard (See Section 5.12). The reference standard must be generated using the conditions of the method. The characteristic ions for this method are provided in Table 2. Compounds are identified as present when the following criteria are met.
- 7.7.1.1 The characteristic masses of each analyte of interest must be in the same scan or co-maximize within one scan of each other. The retention time must fall within ± 0.06 minutes of the retention time of the authentic compound or alkyl homologue grouping determined by the analysis of the daily calibration check or reference material, respectively (see Table 9).
- 7.7.1.2 The alkylated PAH homologue groupings (e.g. C4-naphthalene) appear as a group of structural isomers. The pattern of each group and the retention time window for the group is established by the analysis of the alkylated PAH reference material (See Section 5.12 and Table 9). Each group of alkylated homologues is integrated in its entirety and the total response is used to determine the concentration of the entire group.
- 7.7.1.3 The relative response of the secondary confirmation ions, compared to the primary quantitation ion, must fall within $\pm 30\%$ of the relative intensities of these masses in a reference mass spectrum (See Table 1). The reference mass spectrum is obtained from the continuing calibration solution or the reference material for the parent compounds or the alkylated homologues, respectively. In some instances, a compound that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by a qualified mass spectrometrists.

7.8 Quantitative Analysis



- 7.8.1 Once a compound has been identified, the quantitation of that compound is based on the integrated abundance from the extracted ion current chromatogram of the primary characteristic ion using the internal standard technique. A listing of the internal standards and their respective target compounds is provided in Table 11. The concentration of any analyte is determined using the average response factor/regression analysis as described in Section 7.5.3.1 by reverse extrapolation.

7.9 Sample Calculations

- 7.9.1 The concentration of the sample in $\mu\text{g/L}$, or $\mu\text{g/kg}$, is then calculated as follows:

$$\text{Aqueous Samples } \left(\frac{\mu\text{g}}{\text{L}}\right) = \frac{(A_x \times D \times V_e)}{V_s}$$

$$\text{Soil Samples } \left(\frac{\mu\text{g}}{\text{kg}}\right) = \frac{(A_x \times D \times V_e)}{V_s}$$

Where:

A_x	=	Concentration of the analyte in the extract in ng/mL.
D	=	Dilution factor, if applicable.
V_e	=	Volume of extract in mL.
V_s	=	Amount of sample extracted in mL or g.

8 QUALITY CONTROL

- 8.1 The MS must successfully complete a maximum sensitivity autotune (Section 7.4.1) (Figure 1) and DFTPP Tune Check.
- 8.2 There must be an initial seven point calibration of the GC/MS system. The % RSD of response factors must be less than 20% for the target compounds or R value ≥ 0.9950 for regression analysis.
- 8.3 Continuing calibration verification (CCV) is performed at the beginning of an analysis sequence, after every ten or fewer injections and at the end of the run. Standards of 50 and/or 200 ng/mL are used for continuing calibration verification (CCV). If the calculated amount of the CCV standard differs by more than $\pm 20\%$, the instrument should be recalibrated. If any target compound in a CCV fails, then corrective action must be initiated. Corrective action may include, but not be limited to, preparing a new calibration curve, reanalyzing the affected samples, or GC/MS maintenance. Qualifying data is an option but other options should be explored first.
- 8.4 The response of the internal standards must not vary by $< 50\%$ or $> 200\%$ from their response of the mid-point standard (200 ng/mL) from the initial calibration.
- 8.5 An analytical set consists of 20 or fewer samples. Quality control samples should be analyzed with each set with the following frequency:



- Blanks - One per 20 or fewer samples, minimum one per day
- LCSs - One per 20 or fewer samples, minimum one per day
- MS/MSDs - One MS/MSD per 20 or fewer samples, minimum one set per day

- 8.6 A method blank is prepared and analyzed on each analysis day and after every twenty samples. For soils, the method blank includes silica sand and is carried through all the steps of sample preparation and analysis. In the event that PAHs are measured in the blank, the results cannot be subtracted from sample results. The sample data must be qualified when target compounds are measured in the blank and/or samples reextracted.
- 8.7 Matrix spike and matrix spike duplicate (MS/MSD) samples are analyzed on each analysis day and after every twenty samples. Acceptable recoveries are 70-130%. The RPD between MS/MSD duplicate analyses must be $\leq 20\%$. If the spike level is less than the concentration of the analyte in the parent sample, the MS/MSD is not evaluated and qualification is not necessary. Any indication of a potential matrix effect should be discussed in the sample narrative and the sample data is appropriately qualified.
- 8.8 Surrogate standard is added to all samples as a system monitoring compound for each analysis. An acceptable recovery for the surrogate is 70-130%. Samples that have surrogate recoveries outside of control limits are reanalyzed or the sample data is qualified if reanalysis is not possible.

9 PERFORMANCE DATA

- 9.1 The method detection limit (MDL) for PAHs and Alkylated PAH homologues has been determined for waters and soils from the analysis of eight replicates fortified at 0.02 $\mu\text{g/L}$ and 20 $\mu\text{g/kg}$, respectively. The MDLs and report limits are presented in Table 13 and 14 for waters and soils, respectively.
- 9.2 An initial demonstration of capability (IDC) was performed for waters and soils by analyzing 4 replicate samples. The summary of IDCs is listed in Table 5.

10 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

- 10.1 Contingencies for out-of-control data should be evaluated on a case-by-case basis. A Corrective Action Form (CAF) must be completed for those times that acceptable QC results cannot be achieved. The CAF must be completed by the analyst and filed with the Quality Manager. Analytical results shall be qualified as necessary.

11 WASTE MANAGEMENT / POLLUTION PREVENTION

- 11.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.



12 REFERENCES

- 12.1 Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8270b, SW-846, Test Methods for Evaluating Solid Wastes, Update III Revision 2, September 1994.
- 12.2 Micro-scale Solvent Extraction (MSE), Method 3570, SW-846, Test Methods for Evaluating Solid Wastes, Revision), November 2002.
- 12.3 Separatory Funnel Extraction, Method 3510, SW-846, Test Methods for Evaluating Solid Wastes, Update III Revision 2, September 1994.



TABLE 1

CHARACTERISTIC MASSES (M/Z) AND RELATIVE ABUNDANCES FOR
 POLYNUCLEAR AROMATIC HYDROCARBONS AND ALKYLATED HOMOLOGUE
 GROUPS

Compound	Primary Quantitation Ion	Secondary Qualifier Ion(s)	Typical % Relative Abundance of Qualifier Ion(s)
Acenaphthene	154	153, 152	108, 52
Acenaphthylene	152	151, 153	19, 19
Anthracene	178	179	15
Benzo(a)anthracene	228	229	74
C1-Benz(a)anthracene/chrysene	242	241	40
C2-Benz(a)anthracene/chrysene	256	241	7.9
C3-Benz(a)anthracene/chrysene	270	255	35
C4-Benz(a)anthracene/chrysene	284	269	35*
Benzo(a)pyrene	252	253, 125	29,23
Benzo(b)fluoranthene	252	253, 125	22, 18
Benzo(e)pyrene	252	253, 125	24, 28
Benzo(ghi)perylene	276	138, 277	168, 22
Benzo(k)fluoranthene	252	253, 125	21,21
Chrysene	228	229	74
Dibenzo(ah)anthracene	278	139, 279	32,26
Fluoranthene	202	200	19
C1-Fluoranthene/pyrene	216	215	70
C2-Fluoranthene/pyrene	230	215	50*
C3-Fluoranthene/pyrene	244	229	50*
Fluorene	166	165	92
C1-Fluorene	180	165	137
C2-Fluorene	194	179	138
C3-Fluorene	208	193	125*
Indeno(1,2,3-cd)pyrene	276	138, 227	161, 23
1-Methyl Naphthalene	142	141	86
2-Methyl Naphthalene	142	141	86
Naphthalene	128	129	11
C1-Naphthalene	142	141	86
C2-Naphthalene	156	141	135
C3-Naphthalene	170	155	95
C4-Naphthalene	184	169	196
Phenanthrene	178	179	15
C1-Phenanthrene/anthracene	192	191	47
C2-Phenanthrene/anthracene	206	191	60
C3-Phenanthrene/anthracene	220	205	156
C4-Phenanthrene/anthracene	234	219	165
Pyrene	202	200	19

* % Relative Abundance Permanently Fixed based upon best Available Data



TABLE 1 (CONTINUED)

CHARACTERISTIC MASSES (M/Z) AND RELATIVE ABUNDANCES FOR
POLYNUCLEAR AROMATIC HYDROCARBONS AND ALKYLATED HOMOLOGUE
GROUPS

Compound	Primary Quantitation Ion	Secondary Qualifier Ion(s)	Typical % Relative Abundance of Qualifier Ion(s)
<u>Internal Standards</u>			
Acenaphthene-d10	164	162	93
Chrysene-d12	240	236	25
Perylene-d12	264	260	22
<u>Surrogate Standard</u>			
p-Terphenyl-d14	244	122	22

* % Relative Abundance Permanently Fixed based upon best Available Data



TABLE 2

TYPICAL RETENTION TIME ORDER

Compound	Absolute RT
Naphthalene	4.45
2-Methyl Naphthalene	5.34
1-Methyl Naphthalene	5.49
Acenaphthylene	6.74
1,2-Dimethyl Naphthalene	6.76
Acenaphthene-d10 (IS)	7.01
Acenaphthene	7.07
2,3,5-Trimethyl Naphthalene	7.82
Fluorene	8.05
2,6-Diethyl Naphthalene	8.22
1-Methyl Fluorene	9.38
Phenanthrene	10.26
Anthracene	10.37
1,8-Dimethyl Fluorene	10.78
1-Methyl Anthracene	11.88
Fluoranthene	13.50
Pyrene	14.12
9,10-Dimethyl Anthracene	14.33
2-(t-Butyl) Anthracene	12.53
9-Ethyl-10-Methyl Phenanthrene	14.59
p-Terphenyl-d14 (SS)	14.83
1-Methyl Pyrene	15.82
Benzo(a)anthracene	17.75
Chrysene-d12 (IS)	17.79
Chrysene	17.87
1-Methyl Benz(a)anthracene	18.94
3,9-Dimethyl Benz(a)anthracene	20.26
Benzo(b)fluoranthene	20.84
Benzo(k)fluoranthene	20.91
8,9,11-Trimethyl Benz(a)anthracene	21.73
Benzo(e)pyrene	21.56
Benzo(a)pyrene	21.69
Perylene-d12 (IS)	21.84
Indeno(1,2,3-cd)pyrene	24.32
Dibenzo(ah)anthracene	24.39
Benzo(ghi)perylene	24.76

Column: RTX-5ms, 30 m x 0.32 mm I.D., 0.25 μ film



TABLE 3

LIST OF PAHS IN STOCK STANDARD SOLUTIONS AND NEAT MATERIALS

STOCK STANDARD SOLUTION MIXTURE:

**Absolute, CLP Semi-Volatiles PAH Standard, Catalog #10017,
2,000 µg/mL each component**

Acenaphthene	Benzo(ghi)perylene	Indeno(1,2,3-cd)pyrene
Acenaphthylene	Benzo(k)fluoranthene	Naphthalene
Anthracene	Chrysene	Phenanthrene
Benzo(a)anthracene	Dibenz(ah)anthracene	Pyrene
Benzo(a)pyrene	Fluoranthene	
Benzo(b)fluoranthene	Fluorene	

INDIVIDUAL STOCK SOLUTIONS:

Compound	Vendor	Part #	Conc. (µg/mL)
Benzo(e)pyrene,	Absolute	71016	1000
1-Methyl Naphthalene	Absolute	70313	1000
2-Methyl Naphthalene	Absolute	70214	1000
1,2-Dimethyl Naphthalene	AccuStandard	H-197S	50
2,3,5-Trimethyl Naphthalene	ChemService	F1057JS	100
1-Methyl Pyrene	AccuStandard	H-233S	50
1-Methyl Anthracene	AccuStandard	H-222S	50
9,10-Dimethyl Anthracene	AccuStandard	H-190S	50
1-Methyl Benz(a)anthracene	AccuStandard	H-213S	50
3,9-Dimethyl Benz(a)anthracene	AccuStandard	H-191S	50
8,9,11-Trimethyl Benz(a)anthracene	AccuStandard	H-227S	50

NEAT STANDARDS:

Compound	Vendor	Part #	Purity	CAS RN
2,6-Diethyl Naphthalene	Aldrich	525456-50G	97%	59919-41-4
1-Methyl Fluorene	Aldrich	M46594-250 mg	98%	1730-37-6
1,8-Dimethyl Fluorene	Aldrich	S244678	NA	NA
9-Ethyl-10-Methyl Phenanthrene	Aldrich	S116637	NA	NA
2-(t-Butyl) Anthracene	Aldrich	222267-1G	98%	18801-00-8

SECOND SOURCE:

RESTEK, 8310 PAH Mixture, CAT#31841, 500 µg/mL each

Acenaphthene	Benzo(ghi)perylene	Indeno(1,2,3-cd)pyrene
Acenaphthylene	Benzo(k)fluoranthene	1-methylnaphthalene
Anthracene	Chrysene	2-methylnaphthalene
Benzo(a)anthracene	Dibenzo(a,h)anthracene	Naphthalene
Benzo(a)pyrene	Fluoranthene	Phenanthrene
Benzo(b)fluoranthene	Fluorene	Pyrene

NOTE: Second source preparation will be detailed later.



TABLE 4

PREPARATION OF STOCK SURROGATE AND WORKING SURROGATE SOLUTIONS

Stock Surrogate Solution

Neat Material: p-Terphenyl-d₁₄, CDN Isotopes Part D-87, 98.8 atom %D

Compound	Weight (grams)	Final Volume (mL)	Final Concentration (µg/mL)	Final Solvent
p-Terphenyl-d ₁₄	0.2000	100	2,000	80% Dichloromethane/ 20% Carbon Disulfide

Working Surrogate Solution -Soils

Compound	2000 µg/mL Stock Used (mL)	Final Volume (mL)	Final Concentration (µg/mL)	Final Solvent
p-Terphenyl-d ₁₄	2.5	100	50	Acetone

Working Surrogate Solution -Waters

Compound	50 µg/mL Soils Surrogate	Final Volume (mL)	Final Concentration (µg/mL)	Final Solvent
p-Terphenyl-d ₁₄	10	100	5.0	Acetone



TABLE 5

PREPARATION OF STOCK INTERNAL STANDARD AND WORKING INTERNAL STANDARD SOLUTIONS

Stock Internal Standard Solution

NEAT STANDARDS:

Compound	Vendor	Part #	Isotopic Enrichment
Acenaphthene-d ₁₀	CDN Isotopes	D-42	98.8 atom %D
Chrysene-d ₁₂	CDN Isotopes	D-402	98.8 atom %D
Perylene-d ₁₂	CDN Isotopes	D-670	98.8 atom %D

Compound	Weight (grams)	Final Volume (mL)	Final Concentration (µg/mL)	Final Solvent
Acenaphthene-d ₁₀	0.0750	100	750	80% Dichloromethane/ 20% Carbon Disulfide
Chrysene-d ₁₂	0.0750	100	750	80% Dichloromethane/ 20% Carbon Disulfide
Perylene-d ₁₂	0.0750	100	750	80% Dichloromethane/ 20% Carbon Disulfide

Working Internal Standard Solution

Compound	750 µg/mL Stock Standard Used (mL)	Final Volume (mL)	Final Concentration (µg/mL)	Final Solvent
Acenaphthene-d ₁₀	5.0	50	75	Acetone
Chrysene-d ₁₂	5.0	50	75	Acetone
Perylene-d ₁₂	5.0	50	75	Acetone



TABLE 6

PREPARATION OF INTERMEDIATE CALIBRATION STANDARD
IN 90% DICHLOROMETHANE/10% ACETONE

Compound	Part#	Initial Conc. ($\mu\text{g/mL}$)	Volume Used (mL)	Final Volume (mL)	Final Conc. ($\mu\text{g/mL}$)
p-Terphenyl -d ₁₄		50	2.00	50	2.0
16 PAHs	Absolute 10017	2,000	0.050	50	2.0
Benzo(e)pyrene	Absolute 71016	1,000	0.100	50	2.0
1-Methyl Naphthalene	Absolute 70313	1,000	0.100	50	2.0
2-Methyl Naphthalene	Absolute 70214	1,000	0.100	50	2.0
1,2-Dimethyl Naphthalene	AccuStandard H-197S	50	2.00	50	2.0
2,3,5-Trimethyl Naphthalene	ChemService F1057JS	100	1.00	50	2.0
2,6-Diethyl Naphthalene	From Neat (5.5.1)	500	0.200	50	2.0
1-Methyl Fluorene	From Neat (5.5.1)	500	0.200	50	2.0
1,8-Dimethyl Fluorene	From Neat (5.5.1)	433	0.231	50	2.0
1-Methyl Anthracene	AccuStandard H-222S	50	2.00	50	2.0
9,10-Dimethyl Anthracene	AccuStandard H-190S	12.05	8.30	50	2.0
9-Ethyl-10-Methyl Phenanthrene	From Neat (5.5.1)	500	0.200	50	2.0
2-(t-Butyl) Anthracene	From Neat (5.5.1)	500	0.200	50	2.0
1-Methyl Pyrene	AccuStandard H-233S	50	2.00	50	2.0
1-Methyl Benz(a)anthracene	AccuStandard H-213S	50	2.00	50	2.0
3,9-Dimethyl Benz(a)anthracene	AccuStandard H-191S	50	2.00	50	2.0
8,9,11-Trimethyl Benz(a)anthracene	AccuStandard H-227S	50	2.00	50	2.0



TABLE 7

PREPARATION OF WORKING CALIBRATION AND REPORT LIMIT STANDARD SOLUTIONS IN 90% DICHLOROMETHANE/10% ACETONE

ICAL Level	7	6	5	4	3	2	1
Spike Solution ID	INT	INT	INT	INT	INT	INT	INT
Volume Added (mL)	12.5	6.25	5.0	1.25	1.25	0.250	0.0625
Final Volume (mL)	25	25	50	25	50	25	25
Final Concentration (ng/mL)	1,000	500	200	100	50	20	5

INT = Intermediate Standard Solution, see Tables 4 and 6

All solutions brought to volume with 90% Dichloromethane/10% Acetone.



TABLE 8

REPRESENTATIVE PAHS FOR ALKYLATED HOMOLOGUE GROUPS

Alkylated PAH Homologue Group	Representative Alkylated PAH
C1-Naphthalene	1-Methyl Naphthalene
C2-Naphthalene	1,2-Dimethyl Naphthalene
C3-Naphthalene	2,3,5-Trimethyl Naphthalene
C4-Naphthalene	2,6-Diethyl Naphthalene
C1-Fluorene	1-Methyl Fluorene
C2-Fluorene	1,8-Dimethyl Fluorene
C3-Fluorene	(Standard not available)
C1-Phenanthrene/anthracene	1-Methyl Anthracene
C2-Phenanthrene/anthracene	9,10-Dimethyl Anthracene
C3-Phenanthrene/anthracene	9-Ethyl-10-Methyl Phenanthrene
C4-Phenanthrene/anthracene	2-(t-Butyl) Anthracene
C1-Fluoranthene/pyrene	1-Methyl pyrene
C2-Fluoranthene/pyrene	(Standard not available)
C3-Fluoranthene/pyrene	(Standard not available)
C1-Benz(a)anthracene/chrysene	1-Methyl Benz(a)anthracene
C2-Benz(a)anthracene/chrysene	3,9-Dimethyl Benz(a)anthracene
C3-Benz(a)anthracene/chrysene	8,9,11-Trimethyl Benz(a)anthracene
C4-Benz(a)anthracene/chrysene	(Standard not available)



TABLE 9
RETENTION TIME WINDOWS FOR PAHS AND ALKYLATED PAH HOMOLOGUE
GROUPS

Homologue Group/PAH	Absolute Retention Times	Relative Retention Time
Acenaphthene-d ₁₀ (IS)	7.01	1.000
Naphthalene	4.45	0.635
C1-Naphthalene	5.29 – 5.68	0.755 – 0.810
C2-Naphthalene	6.15 – 6.84	0.877 – 0.976
C3-Naphthalene	7.11 – 8.16	1.014 – 1.164
C4-Naphthalene	7.95 – 9.62	1.134 – 1.372
Acenaphthylene	6.73	0.960
Acenaphthene	7.06	1.007
Fluorene	8.06	1.150
C1-Fluorene	9.22 – 9.63	1.315 – 1.374
C2-Fluorene	10.48 – 11.56	1.495 – 1.649
C3-Fluorene	11.70 – 13.03	1.669 – 1.859
Phenanthrene	10.25	1.462
Anthracene	10.36	1.478
Chrysene-d ₁₂ (IS)	17.79	1.000
C1-Phenanthrene/Anthracene	11.54 – 11.99	0.649 – 0.674
C2-Phenanthrene/Anthracene	12.60 – 14.62	0.708 – 0.822
C3-Phenanthrene/Anthracene	13.90 – 15.64	0.781 – 0.879
C4-Phenanthrene/Anthracene	14.42 – 16.66	0.811 – 0.936
Fluoranthene	13.50	0.759
Pyrene	14.12	0.794
C1-Fluoranthene/Pyrene	14.79 – 15.89	0.831 – 0.893
C2-Fluoranthene/Pyrene	16.31 – 17.43	0.917 – 0.980
C3-Fluoranthene/Pyrene	17.63 – 18.92	0.991 – 1.064
p-Terphenyl	14.83	0.834
Benzo(a)anthracene	17.75	0.998
C1-Benzo(a)anthracene/Chrysene	18.80 – 19.59	1.057 – 1.101
C2-Benzo(a)anthracene/Chrysene	20.08 – 21.19	1.129 – 1.191
C3-Benzo(a)anthracene/Chrysene	21.08 – 22.07	1.185 – 1.241
C4-Benzo(a)anthracene/Chrysene	22.11 – 23.48	1.243 – 1.320
Chrysene	17.87	1.004
Perylene-d ₁₂ (IS)	21.84	1.000
Benzo(b)fluoranthene	20.85	0.955
Benzo(k)fluoranthene	20.91	0.957
Benzo(e)pyrene	21.56	0.987
Benzo(a)pyrene	21.69	0.993
Indeno(1,2,3-cd)pyrene	24.32	1.114
Dibenz(ah)anthracene	24.39	1.117
Benzo(ghi)perylene	24.76	1.134



TABLE 10

PREPARATION OF MATRIX SPIKE SOLUTION IN ACETONE

Compound	Part#	Initial Conc. ($\mu\text{g/mL}$)	Volume Used (mL)	Final Volume (mL)	Final Conc. ($\mu\text{g/mL}$)
16 PAHs	Absolute 10017	2000	0.250	50	10.0
Benzo(e)pyrene	Absolute 71016	1000	0.500	50	10.0
1-Methyl Naphthalene	Absolute 70313	1000	0.500	50	10.0
2-Methyl Naphthalene	Absolute 70214	1000	0.500	50	10.0
2,6-Diethyl Naphthalene	From Neat (5.5.1)	500	1.00	50	10.0
1-Methyl Fluorene	From Neat (5.5.1)	500	1.00	50	10.0
1,8-Dimethyl Fluorene	From Neat (5.5.1)	433	1.16	50	10.0
9-Ethyl-10-Methyl Phenanthrene	From Neat (5.5.1)	500	1.00	50	10.0
2-(t-Butyl) Anthracene	From Neat (5.5.1)	500	1.00	50	10.0



TABLE 11

INTERNAL STANDARDS AND CORRESPONDING TARGET COMPOUNDS

Acenaphthene-d₁₀		
Naphthalene	Acenaphthylene	C3-Fluorene
C1-Naphthalene	Acenaphthene	Phenanthrene
C2-Naphthalene	Fluorene	Anthracene
C3-Naphthalene	C1-Fluorene	
C4-Naphthalene	C2-Fluorene	

Chrysene-d₁₂		
C1-Phenanthrene/Anthracene	C3-Fluoranthene/Pyrene	
C2-Phenanthrene/Anthracene	p-Terphenyl (surrogate)	
C3-Phenanthrene/Anthracene	Benzo(a)anthracene	
C4-Phenanthrene/Anthracene	C-1 Benzo(a)anthracene/Chrysene	
Fluoranthene	C-2 Benzo(a)anthracene/Chrysene	
Pyrene	C-3 Benzo(a)anthracene/Chrysene	
C1-Fluoranthene/Pyrene	C-4 Benzo(a)anthracene/Chrysene	
C2-Fluoranthene/Pyrene	Chrysene	

Perylene-d₁₂		
Benzo(b)fluoranthene	Dibenz(ah)anthracene	
Benzo(k)fluoranthene	Indeno(1,2,3-cd)pyrene	
Benzo(e)pyrene	Benzo(ghi)perylene	
Benzo(a)pyrene		



TABLE 12
TYPICAL RELATIVE RESPONSE FACTORS DETERMINED FOR TARGET
COMPOUNDS

Compound	Response Factor
<u>Acenaphthene-d₁₀ (I.S)</u>	1.000
Naphthalene	2.62
C1-Naphthalene	1.56
C2-Naphthalene	1.13
C3-Naphthalene	0.904
C4-Naphthalene	0.560
Acenaphthylene	2.22
Acenaphthene	1.24
Fluorene	1.36
C1-Fluorene	0.771
C2-Fluorene	0.391
C3-Fluorene	0.300*
Phenanthrene	1.74
Anthracene	1.58
<u>Chrysene-d₁₂ (I.S)</u>	1.000
C1-Phenanthrene/Anthracene	1.36
C2-Phenanthrene/Anthracene	0.680
C3-Phenanthrene/Anthracene	0.511
C4-Phenanthrene/Anthracene	0.322
Fluoranthene	1.69
Pyrene	1.75
C1-Fluorathene/Pyrene	0.984
C2-Fluorathene/Pyrene	0.650*
C3-Fluorathene/Pyrene	0.350*
p-Terphenyl (surrogate)	0.837
Benzo(a)anthracene	1.29
C1-Benzo(a)anthracene/Chrysene	0.912
C2-Benzo(a)anthracene/Chrysene	1.06
C3-Benzo(a)anthracene/Chrysene	0.541
C4-Benzo(a)anthracene/Chrysene	0.350*
Chrysene	1.25
<u>Perylene-d₁₂ (I.S.)</u>	1.00
Benzo(b)fluoranthene	1.40
Benzo(k)fluoranthene	1.13
Benzo(e)pyrene	1.25
Benzo(a)pyrene	1.15
Dibenz(ah)anthracene	0.818
Indeno(1,2,3-cd)pyrene	0.896
Benzo(ghi)perylene	1.04

NOTE: Response Factors for Alkylated Homologue Groups Acquired from their Representative Alkylated PAH (Table 8)

* Response Factors Permanently Fixed Based Upon Best Available Data – Representative Alkylated PAH not available



TABLE 13

METHOD DETECTION LIMITS (MDLS) AND REPORT LIMITS FOR WATER SAMPLES
PAHS AND ALKYLATED HOMOLOGUE GROUPS

Compound	MDL (µg/L)	Report Limit (µg/L)
Naphthalene	0.018	0.050
C1-Naphthalene	0.021	0.050
C2-Naphthalene	0.0047	0.040
C3-Naphthalene	0.0054	0.050
C4-Naphthalene	0.0031	0.075
Acenaphthylene	0.0019	0.010
Acenaphthene	0.0024	0.010
Fluorene	0.0022	0.010
C1-Fluorene	0.0045	0.020
C2-Fluorene	0.0048	0.040
C3-Fluorene	N.D.	0.050
Phenanthrene	0.0046	0.010
Anthracene	0.0028	0.010
C1-Phenanthrene/Anthracene	0.0037	0.020
C2-Phenanthrene/Anthracene	0.0017	0.040
C3-Phenanthrene/Anthracene	0.0046	0.050
C4-Phenanthrene/Anthracene	0.0022	0.075
Fluoranthene	0.0030	0.010
Pyrene	0.0021	0.010
C1-Fluoranthene/Pyrene	0.0017	0.020
C2-Fluoranthene/Pyrene	N.D.	0.040
C3-Fluoranthene/Pyrene	N.D.	0.050
Benzo(a)anthracene	0.0042	0.010
C1-Benzo(a)anthracene/Chrysene	0.0031	0.020
C2-Benzo(a)anthracene/Chrysene	0.0019	0.040
C3-Benzo(a)anthracene/Chrysene	0.0027	0.050
C4-Benzo(a)anthracene/Chrysene	N.D.	0.075
Chrysene	0.0038	0.010
Benzo(b)fluoranthene	0.0052	0.010
Benzo(k)fluoranthene	0.0039	0.010
Benzo(e)pyrene	0.0020	0.010
Benzo(a)pyrene	0.0016	0.010
Indeno(1,2,3-cd)pyrene	0.0022	0.010
Dibenz(ah)anthracene	0.0091	0.010
Benzo(ghi)perylene	0.0045	0.010

Note 1: MDLs for Alkylated Homologue Groups determined from their Representative Alkylated PAH (Table 8)

Note 2: Report Limits for Alkylated Homologue Groups adjusted based upon nominal widths of their Retention Time Windows (Table 9) and Relative Response Factors (Table 12)

Data: GC Run 1740 Analyzed on 07/19/07



TABLE 14

METHOD DETECTION LIMITS (MDLS) AND REPORT LIMITS FOR SOIL SAMPLES
 PAHS AND ALKYLATED HOMOLOGUE GROUPS

Compound	Spike Level (µg/kg)	Mean Recovery	Percent Recovery	Percent RSD	MDL (µg/kg)	Report Limit (µg/kg)
Naphthalene	20	20.16	101	2.7	1.6	10
C1-Naphthalene	20	19.85	99.3	3.4	2.0	20
C2-Naphthalene	20	19.95	99.8	6.0	3.6	40
C3-Naphthalene	20	19.51	97.6	3.5	2.0	50
C4-Naphthalene	20	18.01	90.0	4.3	2.3	75
Acenaphthylene	20	18.97	94.9	3.6	2.0	10
Acenaphthene	20	19.57	97.8	5.0	2.9	10
Fluorene	20	18.96	94.8	3.4	1.9	10
C1-Fluorene	20	17.79	89.0	2.4	1.3	20
C2-Fluorene	17.32	15.26	88.1	6.4	2.9	40
C3-Fluorene	ND	ND	ND	ND	ND	50
Phenanthrene	20	18.97	94.9	2.5	1.4	10
Anthracene	20	17.25	86.3	5.1	2.6	10
C1-Phenanthrene/Anthracene	20	16.89	84.5	5.7	2.9	20
C2-Phenanthrene/Anthracene	4.82	3.75	77.7	13.4	1.5	40
C3-Phenanthrene/Anthracene	20	17.79	88.9	5.2	2.8	50
C4-Phenanthrene/Anthracene	20	14.52	72.6	8.6	3.7	75
Fluoranthene	20	17.71	88.6	4.7	2.5	10
Pyrene	20	18.16	90.8	3.7	2.0	10
C1-Fluoranthene/Pyrene	20	16.08	80.4	5.3	2.5	20
C2-Fluoranthene/Pyrene	ND	ND	ND	ND	ND	40
C3-Fluoranthene/Pyrene	ND	ND	ND	ND	ND	50
Benzo(a)anthracene	20	22.20	111	4.5	3.0	10
C1-Benzo(a)anthracene/Chrysene	20	15.02	75.1	4.5	2.0	20
C2-Benzo(a)anthracene/Chrysene	20	14.02	70.1	4.8	2.0	40
C3-Benzo(a)anthracene/Chrysene	20	14.43	72.2	10.9	4.7	50
C4-Benzo(a)anthracene/Chrysene	ND	ND	ND	ND	ND	75
Chrysene	20	18.22	91.1	4.4	2.4	10
Benzo(b)fluoranthene	20	17.27	86.4	6.7	2.4	10
Benzo(k)fluoranthene	20	16.70	83.5	5.8	3.5	10
Benzo(e)pyrene	20	17.79	89.0	5.5	2.9	10
Benzo(a)pyrene	20	15.41	77.0	6.5	2.9	10
Indeno(1,2,3-cd)pyrene	20	15.09	75.4	5.1	3.2	10
Dibenz(ah)anthracene	20	15.15	75.7	7.0	2.3	10
Benzo(ghi)perylene	20	16.93	84.6	5.3	2.7	10

Data: GC Run 1736 Analyzed on 07/17/07

N.D.=Not Determined

Note: MDLs for Alkylated Homologue Groups determined from their Representative Alkylated PAH (Table 8)

Note: Report Limits for Alkylated Homologue Groups adjusted based upon nominal widths of their Retention Time Windows (Table 9) and Relative Response Factors (Table 12)



TABLE 15

INITIAL DEMONSTRATION OF CAPABILITY (IDCS) FOR SOIL SAMPLES PAHS AND ALKYLATED PAH HOMOLOGUE GROUPS

Compound	Spike Level (µg/kg)	Average Recovery (µg/kg)	Average % Recovery	Percent RSD
Naphthalene	200	201	100	0.93
C1-Naphthalene	200	193	96.3	1.2
C2-Naphthalene	200	194	96.9	2.1
C3-Naphthalene	200	188	94.2	2.6
C4-Naphthalene	200	182	90.9	0.94
Acenaphthylene	200	185	92.3	1.0
Acenaphthene	200	192	95.8	0.63
Fluorene	200	186	93.2	0.98
C1-Fluorene	200	180	89.9	1.4
C2-Fluorene	173	155	89.4	0.88
C3-Fluorene	ND	ND	ND	ND
Phenanthrene	200	187	93.4	1.6
Anthracene	200	175	87.7	2.6
C1-Phenanthrene/Anthracene	200	170	84.9	4.2
C2-Phenanthrene/Anthracene	48	35.7	74.1	2.1
C3-Phenanthrene/Anthracene	200	182	90.8	1.2
C4-Phenanthrene/Anthracene	200	157	78.3	3.6
Fluoranthene	200	179	89.4	1.3
Pyrene	200	179	89.7	1.3
C1-Fluoranthene/Pyrene	200	175	87.6	1.0
C2-Fluoranthene/Pyrene	ND	ND	ND	ND
C3-Fluoranthene/Pyrene	ND	ND	ND	ND
Benzo(a)anthracene	200	175	87.4	1.3
C1-Benzo(a)anthracene/Chrysene	200	172	85.9	2.7
C2-Benzo(a)anthracene/Chrysene	200	164	82.0	3.1
C3-Benzo(a)anthracene/Chrysene	200	163	81.7	5.9
C4-Benzo(a)anthracene/Chrysene	ND	ND	ND	ND
Chrysene	200	187	93.4	2.1
Benzo(b)fluoranthene	200	185	92.7	2.7
Benzo(k)fluoranthene	200	180	90.0	3.5
Benzo(e)pyrene	200	185	92.5	1.8
Benzo(a)pyrene	200	167	83.4	2.6
Indeno(1,2,3-cd)pyrene	200	165	82.6	2.3
Dibenz(ah)anthracene	200	173	86.5	2.5
Benzo(ghi)perylene	200	179	89.6	1.8

ND=Not Determined

RSD=Relative Standard Deviation

Note: IDCs for Alkylated Homologue Groups determined from their Representative Alkylated PAH (Table 8)



TABLE 16

INITIAL DEMONSTRATION OF CAPABILITY (IDCS) FOR WATER SAMPLES PAHS
AND ALKYLATED PAH HOMOLOGUE GROUPS

Compound	Spike Level ($\mu\text{g/L}$)	Average % Recovery	Percent RSD
Naphthalene	0.20	98	6.9
C1-Naphthalene	0.20	93	4.6
C2-Naphthalene	0.20	85	6.2
C3-Naphthalene	0.20	89	5.9
C4-Naphthalene	0.20	87	7.6
Acenaphthylene	0.20	93	6.7
Acenaphthene	0.20	89	6.6
Fluorene	0.20	97	5.7
C1-Fluorene	0.20	97	4.2
C2-Fluorene	0.17	100	4.7
C3-Fluorene	0	ND	ND
Phenanthrene	0.20	104	4.0
Anthracene	0.20	98	4.3
C1-Phenanthrene/Anthracene	0.20	110	5.7
C2-Phenanthrene/Anthracene	0.048	77	8.7
C3-Phenanthrene/Anthracene	0.20	108	2.3
C4-Phenanthrene/Anthracene	0.20	110	2.3
Fluoranthene	0.20	111	4.7
Pyrene	0.20	111	2.5
C1-Fluoranthene/Pyrene	0.20	110	3.5
C2-Fluoranthene/Pyrene	0	ND	ND
C3-Fluoranthene/Pyrene	0	ND	ND
Benzo(a)anthracene	0.20	107	4.5
C1-Benzo(a)anthracene/Chrysene	0.20	107	3.5
C2-Benzo(a)anthracene/Chrysene	0.20	108	3.9
C3-Benzo(a)anthracene/Chrysene	0.20	107	5.8
C4-Benzo(a)anthracene/Chrysene	0	ND	ND
Chrysene	0.20	102	4.4
Benzo(b)fluoranthene	0.20	108	2.3
Benzo(k)fluoranthene	0.20	102	4.8
Benzo(e)pyrene	0.20	105	3.8
Benzo(a)pyrene	0.20	103	5.5
Indeno(1,2,3-cd)pyrene	0.20	110	4.3
Dibenz(ah)anthracene	0.20	104	4.9
Benzo(ghi)perylene	0.20	102	4.1

N.D.=Not Determined

RSD=Relative Standard Deviation

Note: IDCs for Alkylated Homologue Groups determined from their Representative Alkylated PAH (Table 8)

Data: GC Run 1740 Analyzed on 07/19/07



FIGURE 1

MAXIMUM SENSITIVITY AUTOTUNE REPORT

HP5972 Maximum Sensitivity Autotune
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Fri Jul 13 13:37:44 2007
C:\HPCHEM\1\5972\ATUNE.U

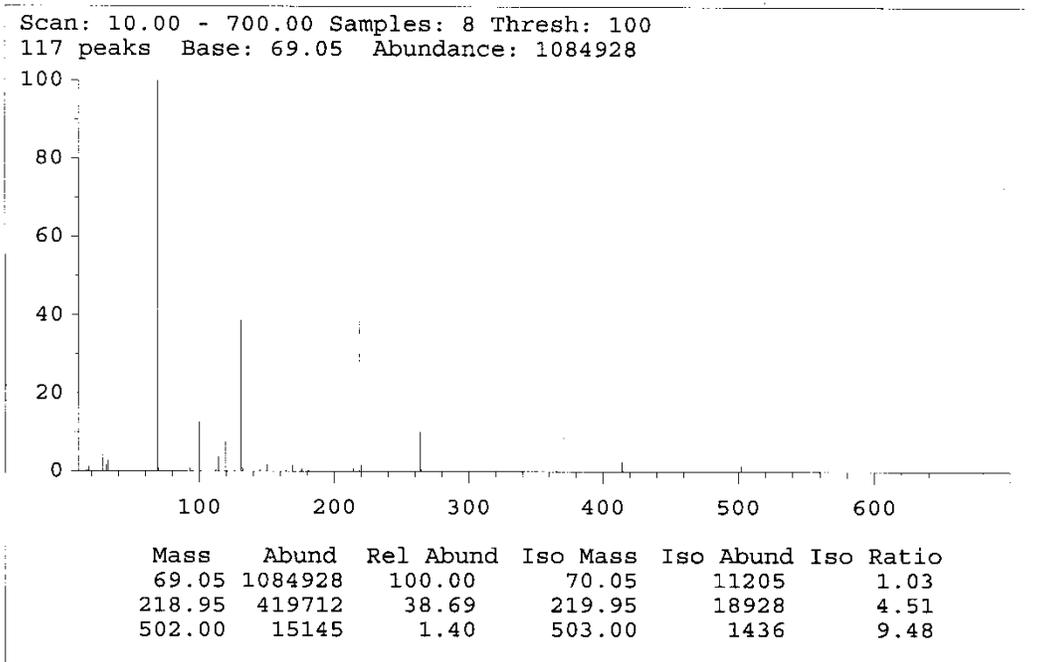
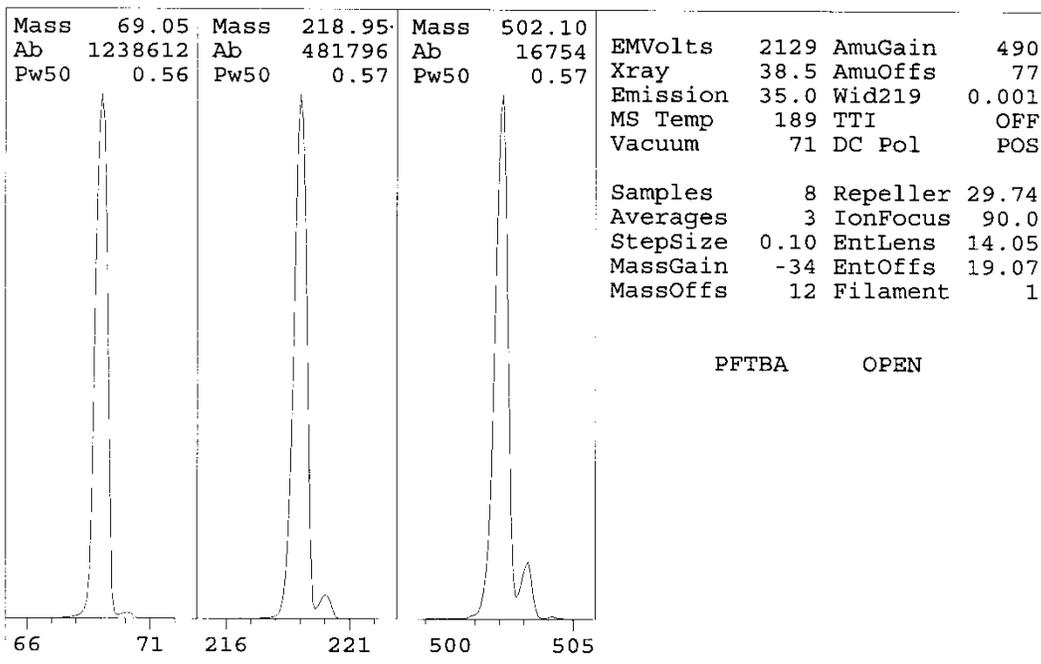




FIGURE 2

TOTAL ION CHROMATOGRAM OF A CCV

Quantitation Report (Not Reviewed)

Data File : K:\1\DATA\GC-1725\006.D Vial: 6
Acq On : 3 Jul 2007 3:03 pm Operator: cps
Sample : 200 ng/ml, ALK PAH ICAL Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Jul 10 11:06 2007 Quant Results File: APAH0703.RES

Quant Method : C:\HPCHEM\1\METHODS\APAH0703.M (RTE Integrator)
Title : PAHs by GC/MS
Last Update : Tue Jul 10 10:58:46 2007
Response via : Initial Calibration
DataAcq Meth : ALKPAH

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) Acenaphthene-d10	7.02	164	1330149	1875.00	ng/ml	0.00
15) Chrysene-d12	17.80	240	1221854	1875.00	ng/ml	0.00
32) Perylene-d12	21.85	264	957445	1875.00	ng/ml	0.00

System Monitoring Compounds	R.T.	QIon	Response	Conc	Units	Dev(Min)
25) p-Terphenyl	14.83	244	114690	193.91	ng/ml	0.00
Spiked Amount	500.000		Recovery	=	38.78%	

Target Compounds	R.T.	QIon	Response	Conc	Units	Qvalue
2) Naphthalene	4.45	128	286264	189.62	ng/ml	100
3) C1-Naphthalene	5.34	142	193680	211.67	ng/ml	100
4) C2-Naphthalene	6.76	156	146785	192.40	ng/ml	100
5) C3-Naphthalene	7.83	170	116794	187.54	ng/ml	100
6) C4-Naphthalene	8.22	184	71456	188.60	ng/ml	100
7) Acenaphthylene	6.73	152	274464	201.21	ng/ml	100
8) Acenaphthene	7.06	154	156098	204.28	ng/ml	100
9) Fluorene	8.06	166	164661	193.82	ng/ml	100
10) C1-Fluorene	9.39	180	95620	194.49	ng/ml	100
11) C2-Fluorene	10.77	194	43225	154.18	ng/ml	100
12) C3-Fluorene	12.23	208	414	1.95	ng/ml#	1
13) Phenanthrene	10.25	178	225450	200.72	ng/ml	100
14) Anthracene	10.36	178	185738	181.02	ng/ml	100
16) C1-Phenanthrene/Anthracene	11.89	192	168107	197.58	ng/ml	100
17) C2-Phenanthrene/Anthracene	14.33	206	23647	51.22	ng/ml	100
18) C3-Phenanthrene/Anthracene	14.60	220	67453	195.30	ng/ml	100
19) C4-Phenanthrene/Anthracene	14.52	234	48956	202.52	ng/ml	100
20) Fluoranthene	13.50	202	187318	188.46	ng/ml	100
21) Pyrene	14.12	202	193771	189.04	ng/ml	100
22) C1-Fluoranthene/Pyrene	15.83	216	111222	190.15	ng/ml	100
23) C2-Fluoranthene/Pyrene	16.66	230	263	0.62	ng/ml#	100
24) C3-Fluoranthene/Pyrene	0.00	244	0	N.D.		
26) Benzo(a)anthracene	17.75	228	137387	194.71	ng/ml	100
27) C1-Benz(a)anthracene/Chrys	18.95	242	104615	191.87	ng/ml	100
28) C2-Benz(a)anthracene/Chrys	20.26	256	124221	190.65	ng/ml	100
29) C3-Benz(a)anthracene/Chrys	21.73	270	68565	183.91	ng/ml	100
30) C4-Benz(a)anthracene/Chrys	22.91	284	477	2.09	ng/ml#	1
31) Chrysene	17.87	228	127278	188.49	ng/ml	100
33) Benzo(b)fluoranthene	20.85	252	121501	183.81	ng/ml	100
34) Benzo(k)fluoranthene	20.92	252	103764	196.43	ng/ml	100
35) Benzo(e)pyrene	21.56	252	112009	186.97	ng/ml	100
36) Benzo(a)pyrene	21.68	252	102120	198.26	ng/ml	100
37) Dibenzo(ah)anthracene	24.39	278	64107	184.38	ng/ml	100
38) Indeno(1,2,3-cd)pyrene	24.31	276	73485	181.59	ng/ml	100
39) Benzo(ghi)perylene	24.76	276	99993	196.44	ng/ml	100



FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

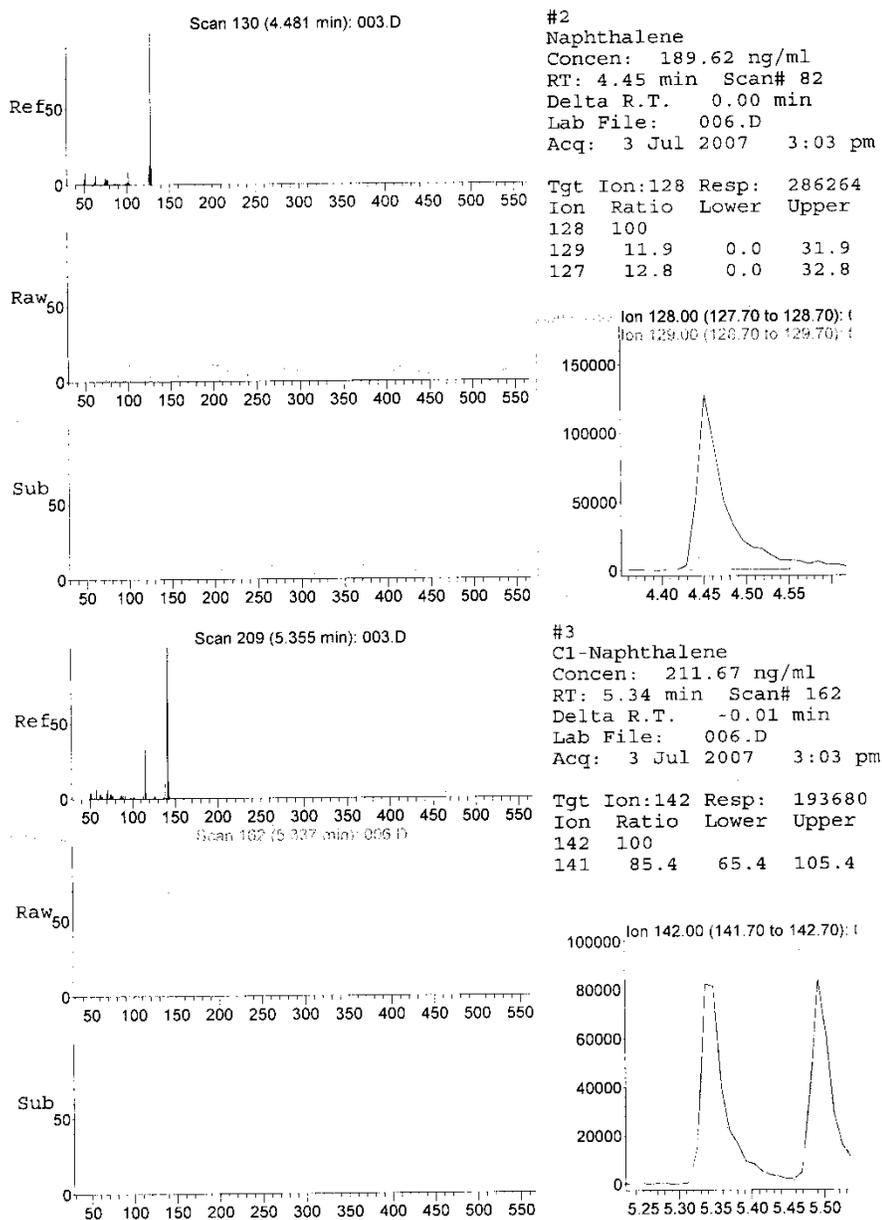




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

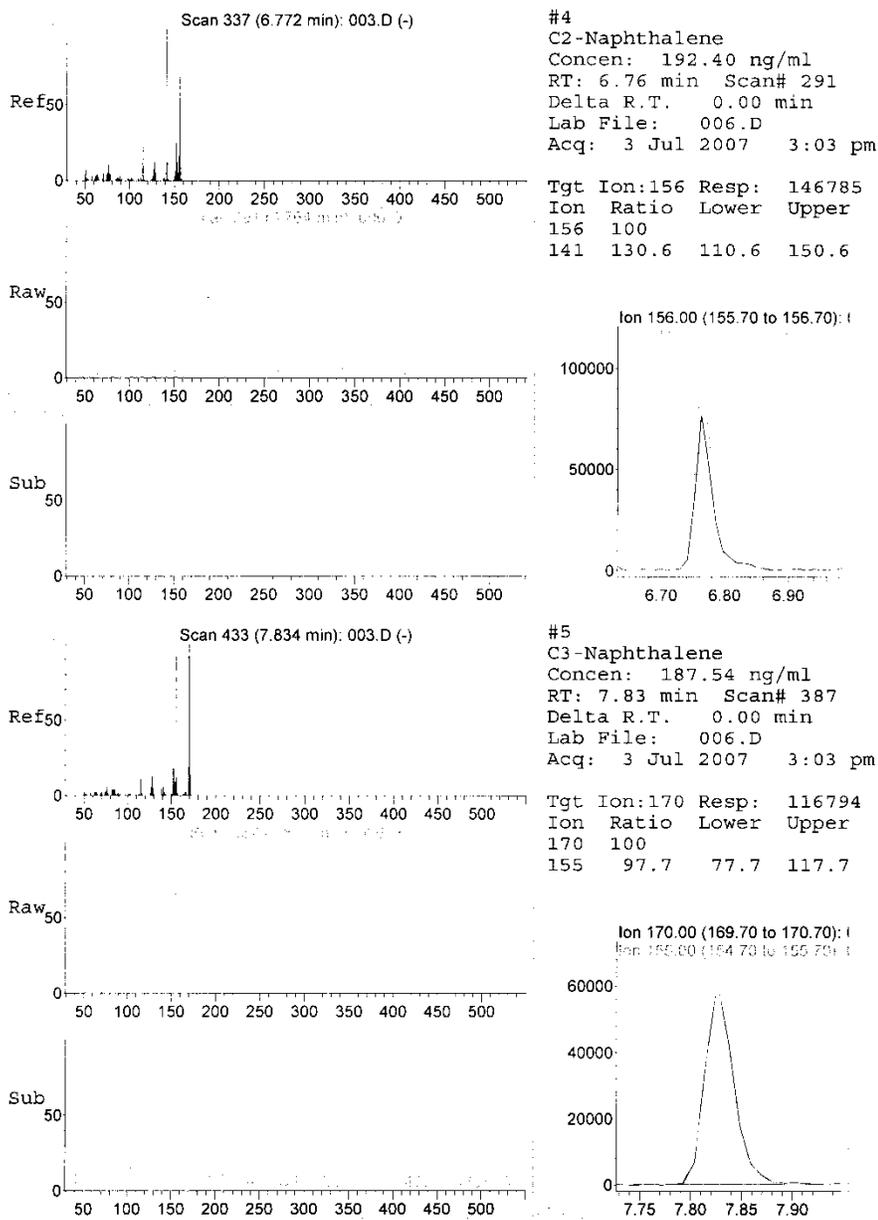




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

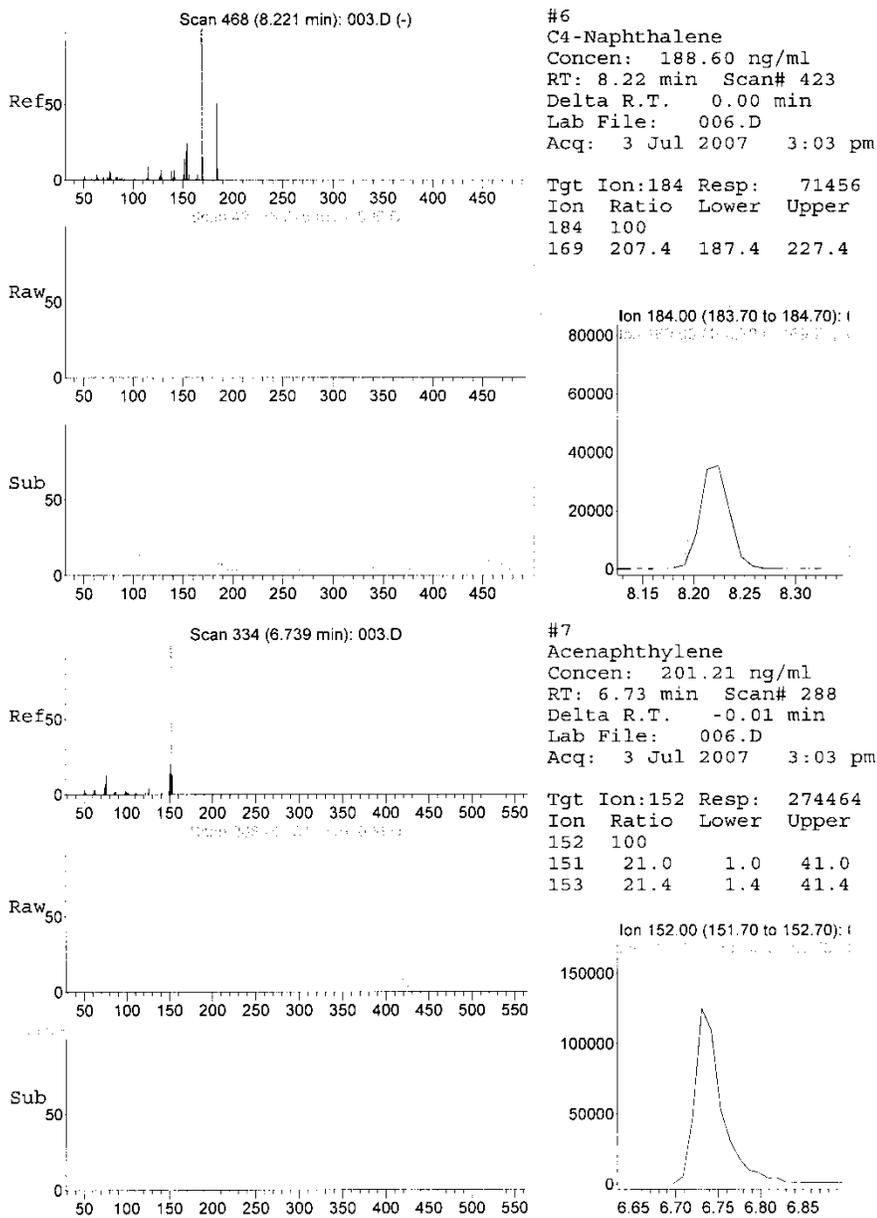




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

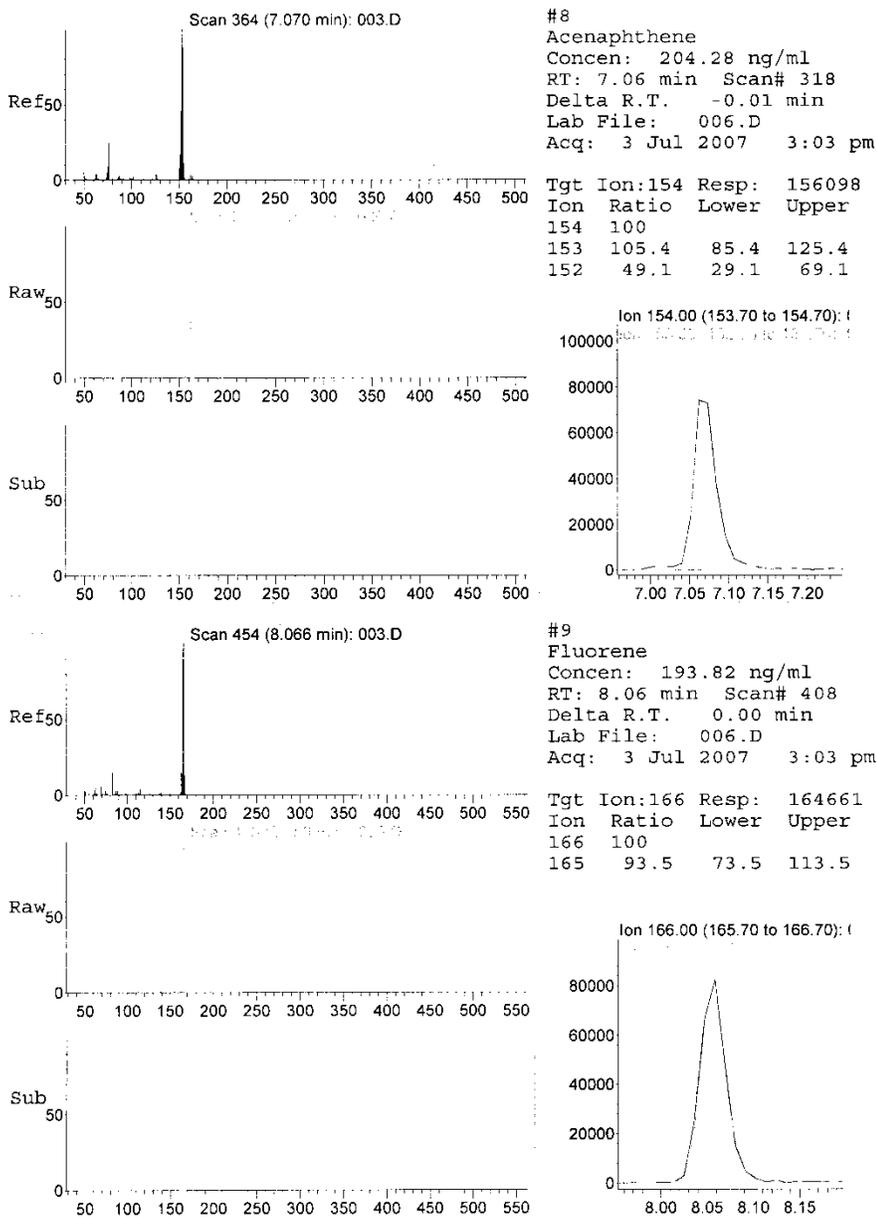




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

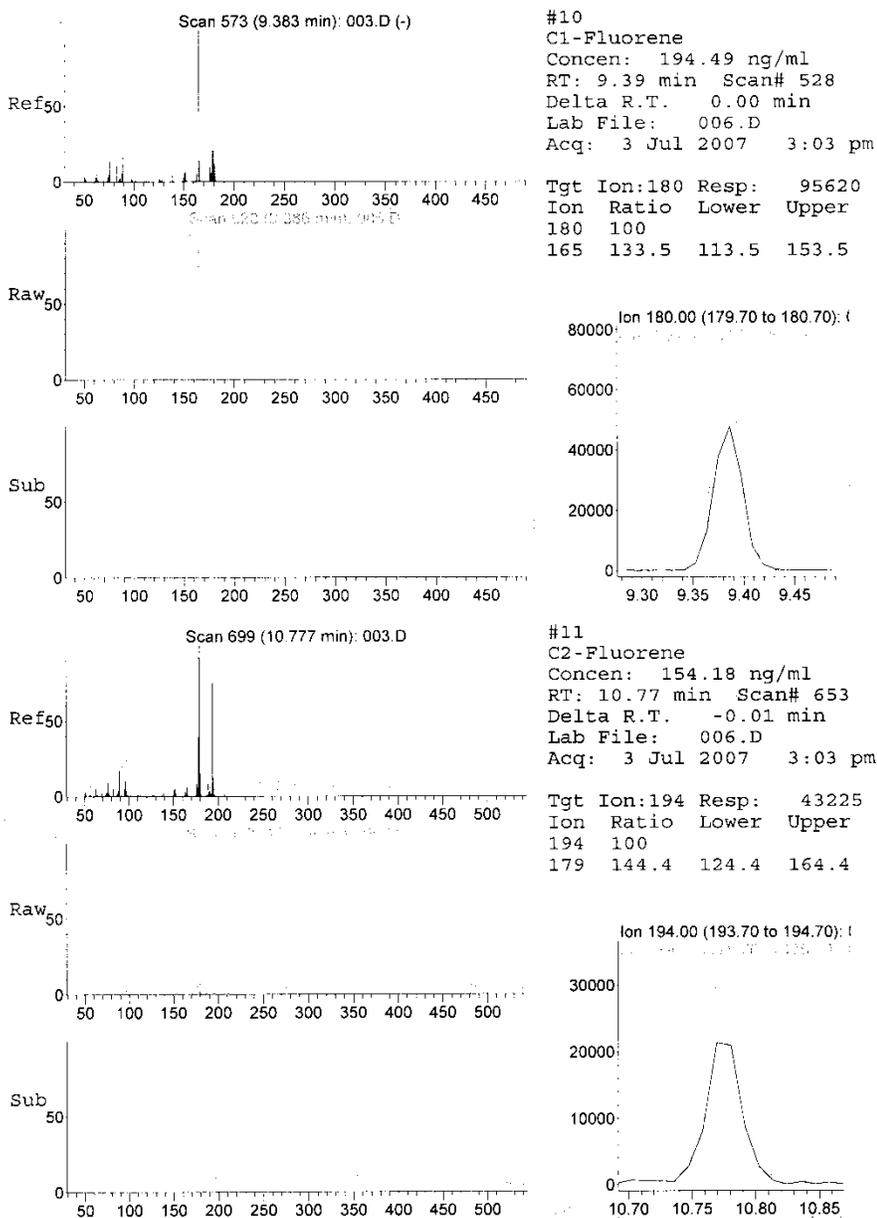




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

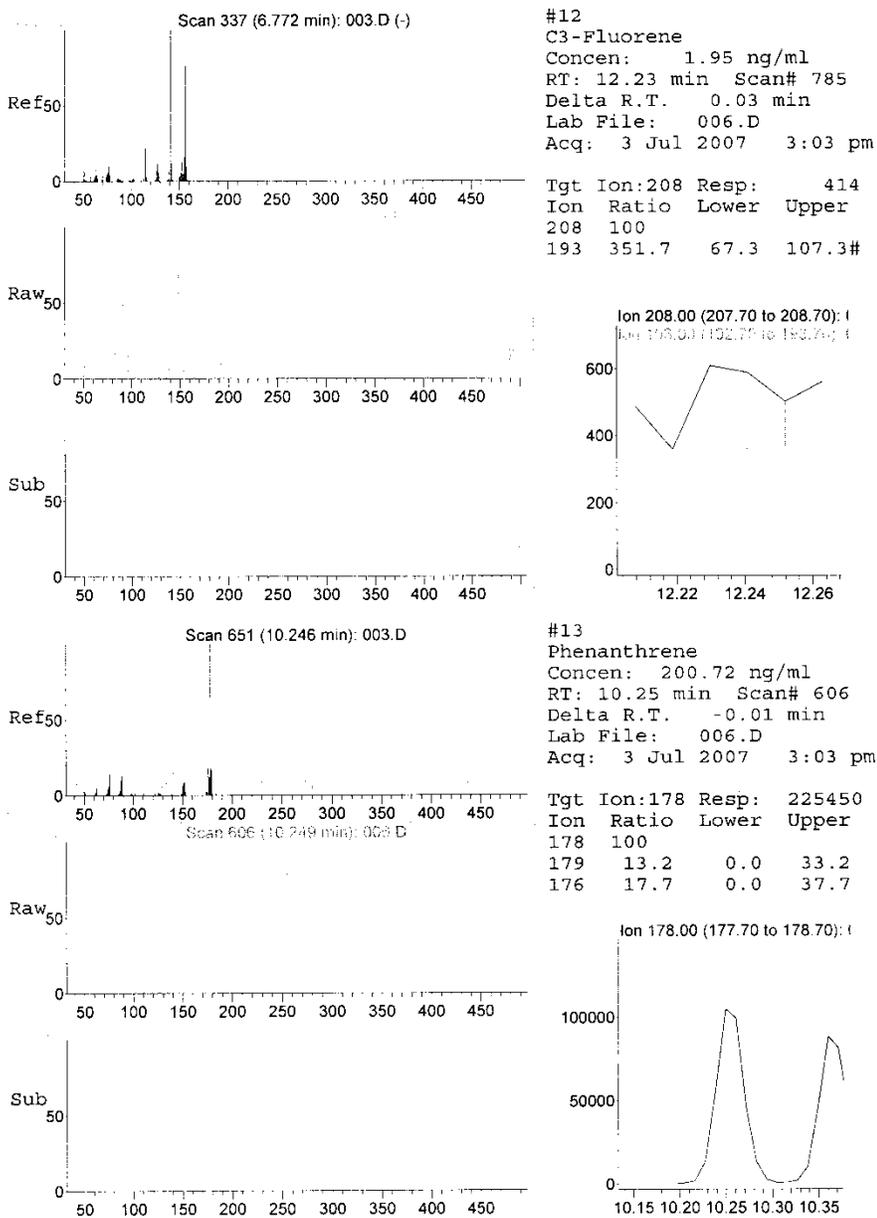




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

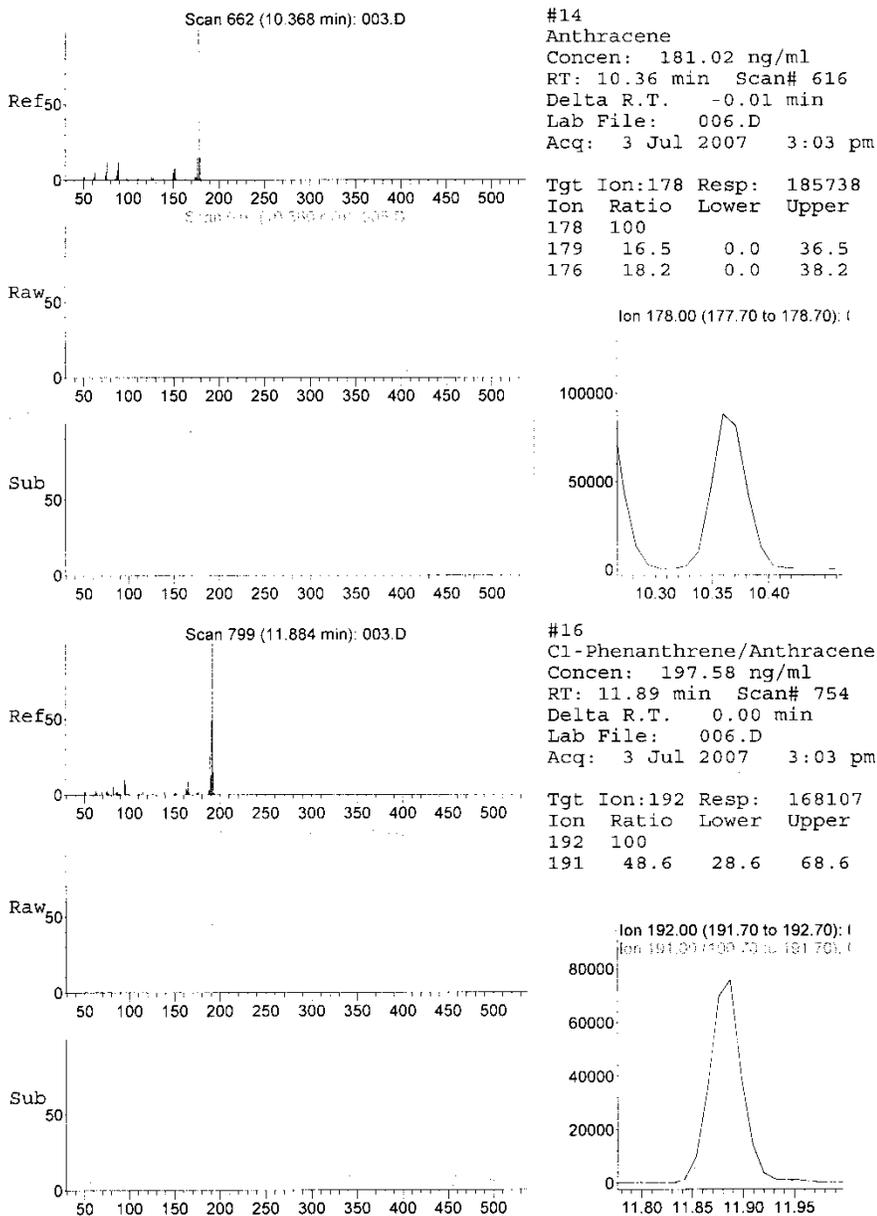




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

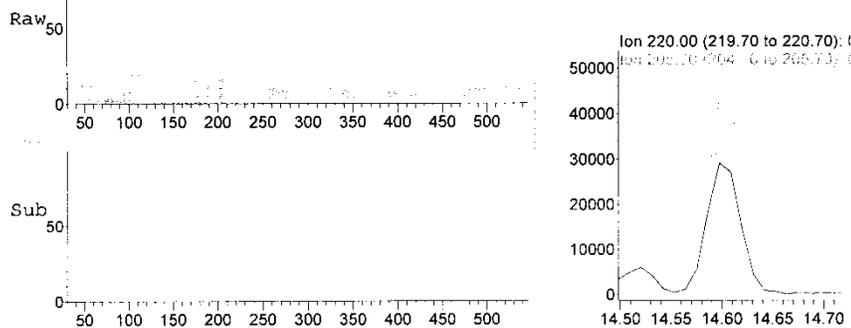
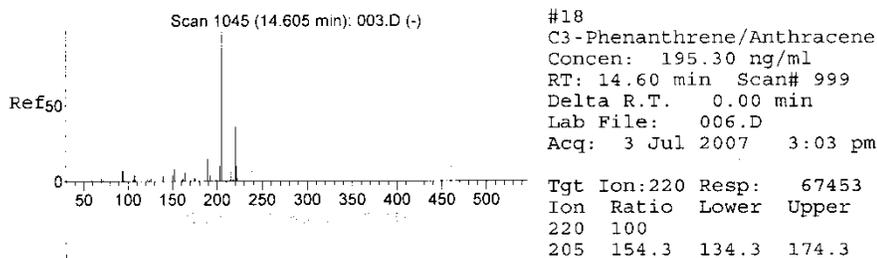
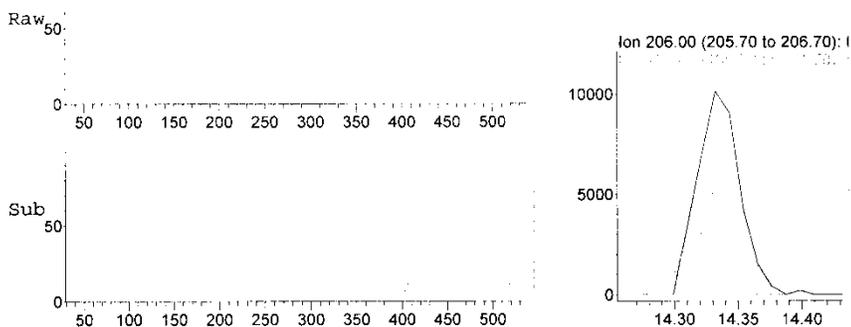
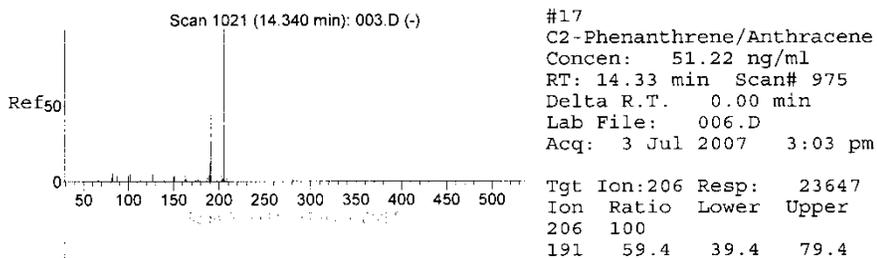




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

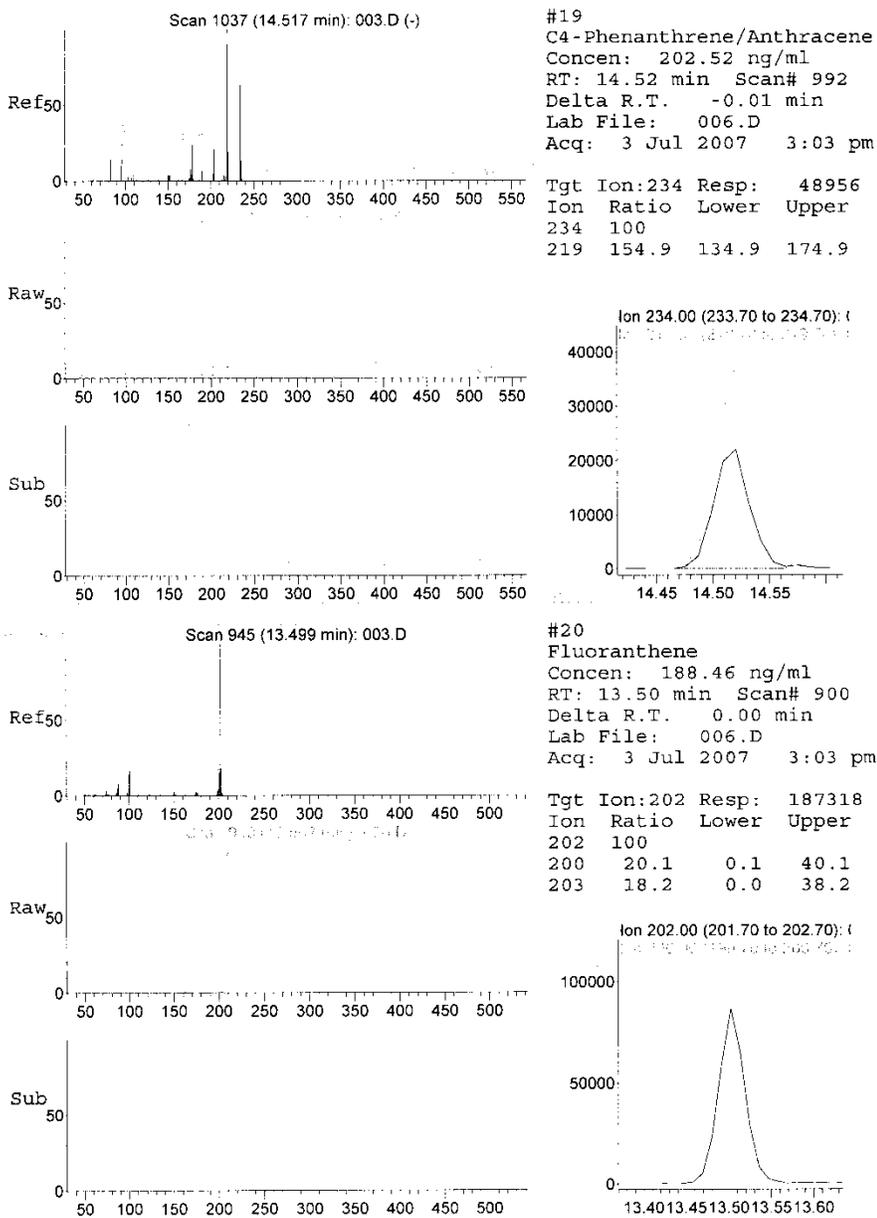




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

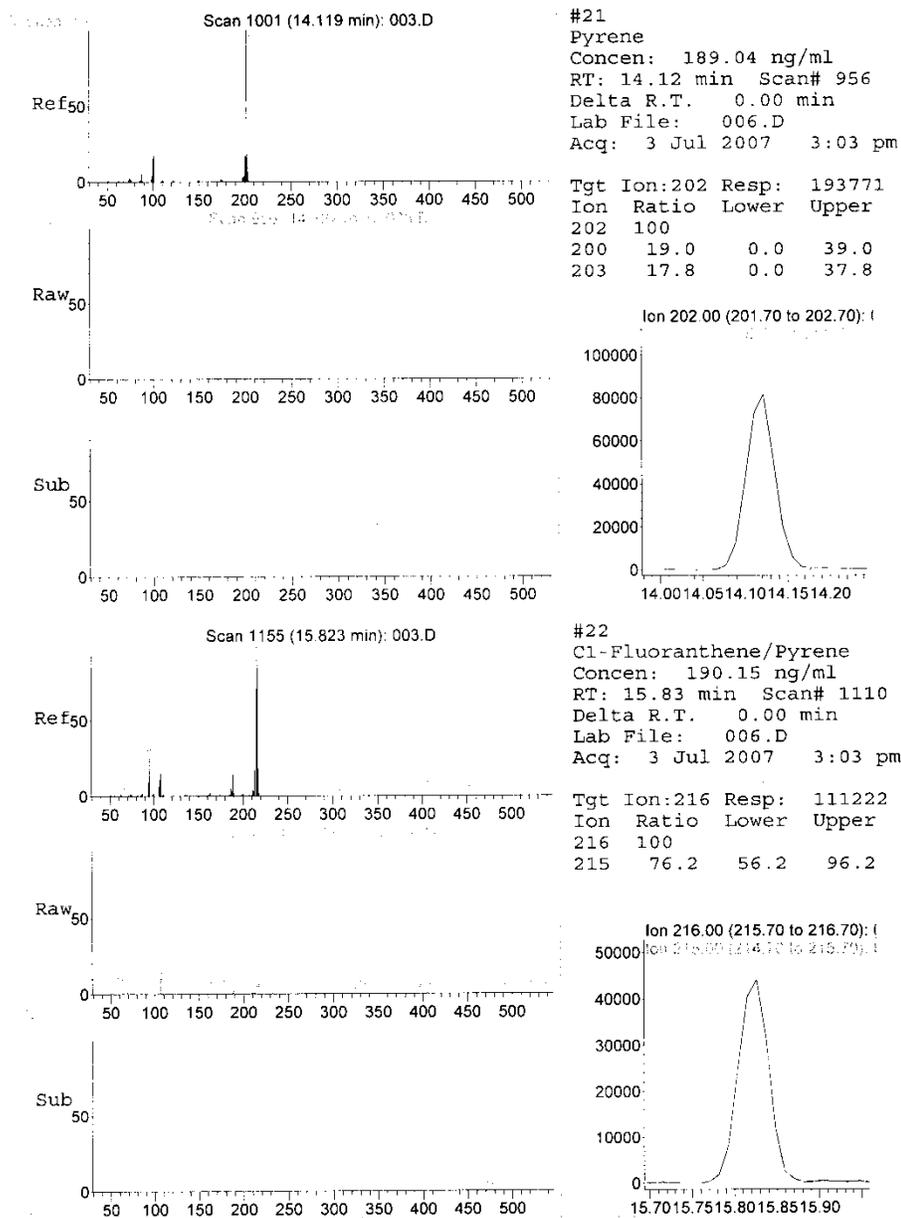




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

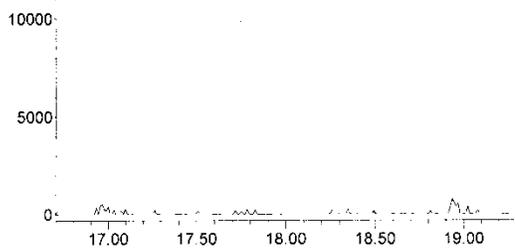
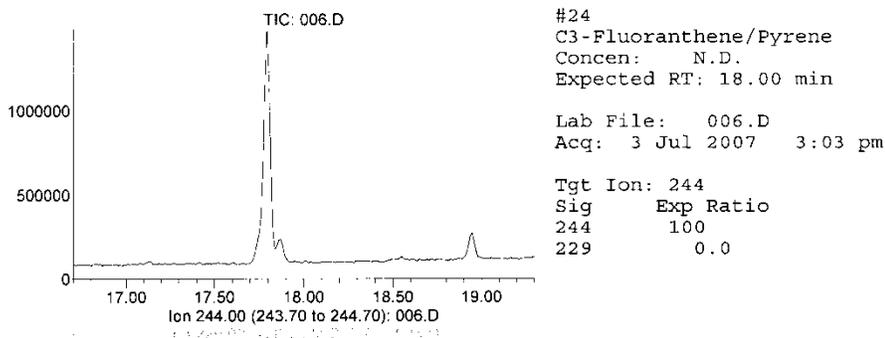
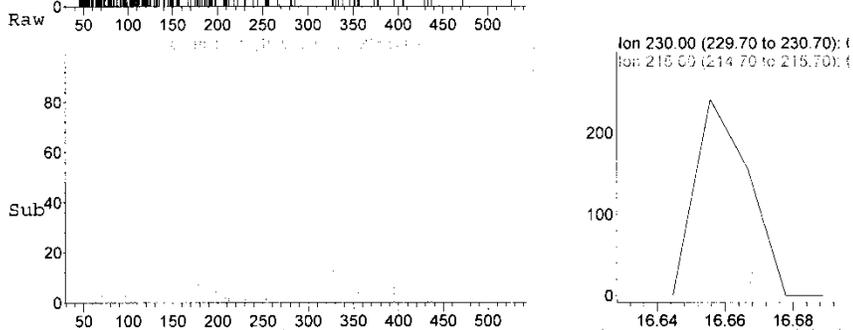
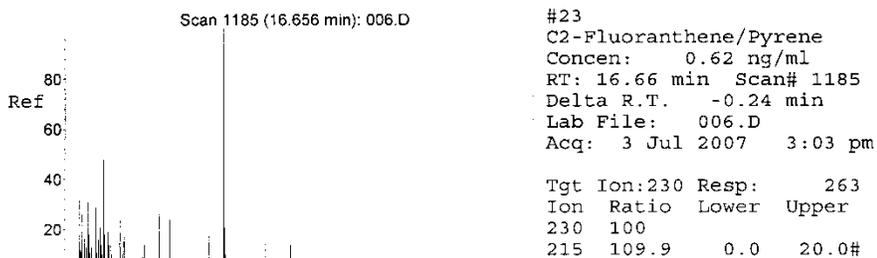
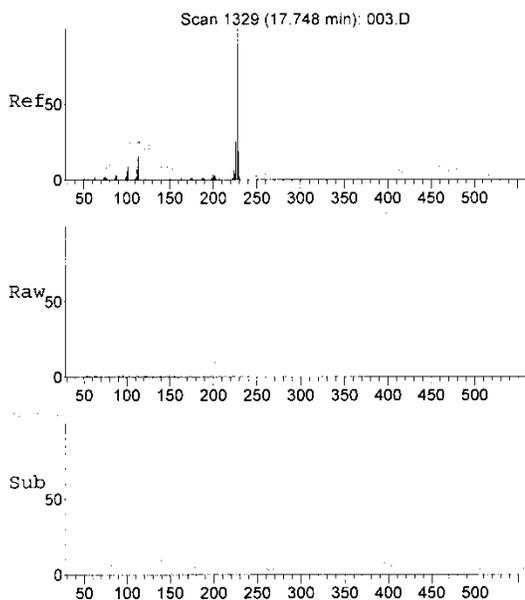




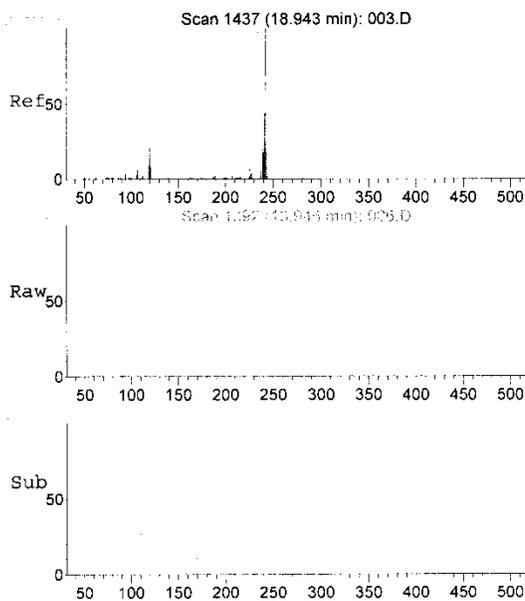
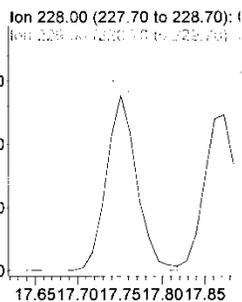
FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV



#26
Benzo(a)anthracene
Concen: 194.71 ng/ml
RT: 17.75 min Scan# 1284
Delta R.T. 0.00 min
Lab File: 006.D
Acq: 3 Jul 2007 3:03 pm

Tgt Ion	Resp	Lower	Upper
228	137387	100	
229	19.9	0.0	39.9
226	24.0	4.0	44.0



#27
Cl-Benz(a)anthracene/Chrysene
Concen: 191.87 ng/ml
RT: 18.95 min Scan# 1392
Delta R.T. 0.00 min
Lab File: 006.D
Acq: 3 Jul 2007 3:03 pm

Tgt Ion	Resp	Lower	Upper
242	104615	100	
241	44.4	24.4	64.4

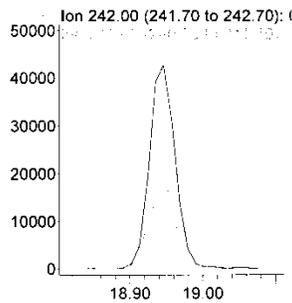




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

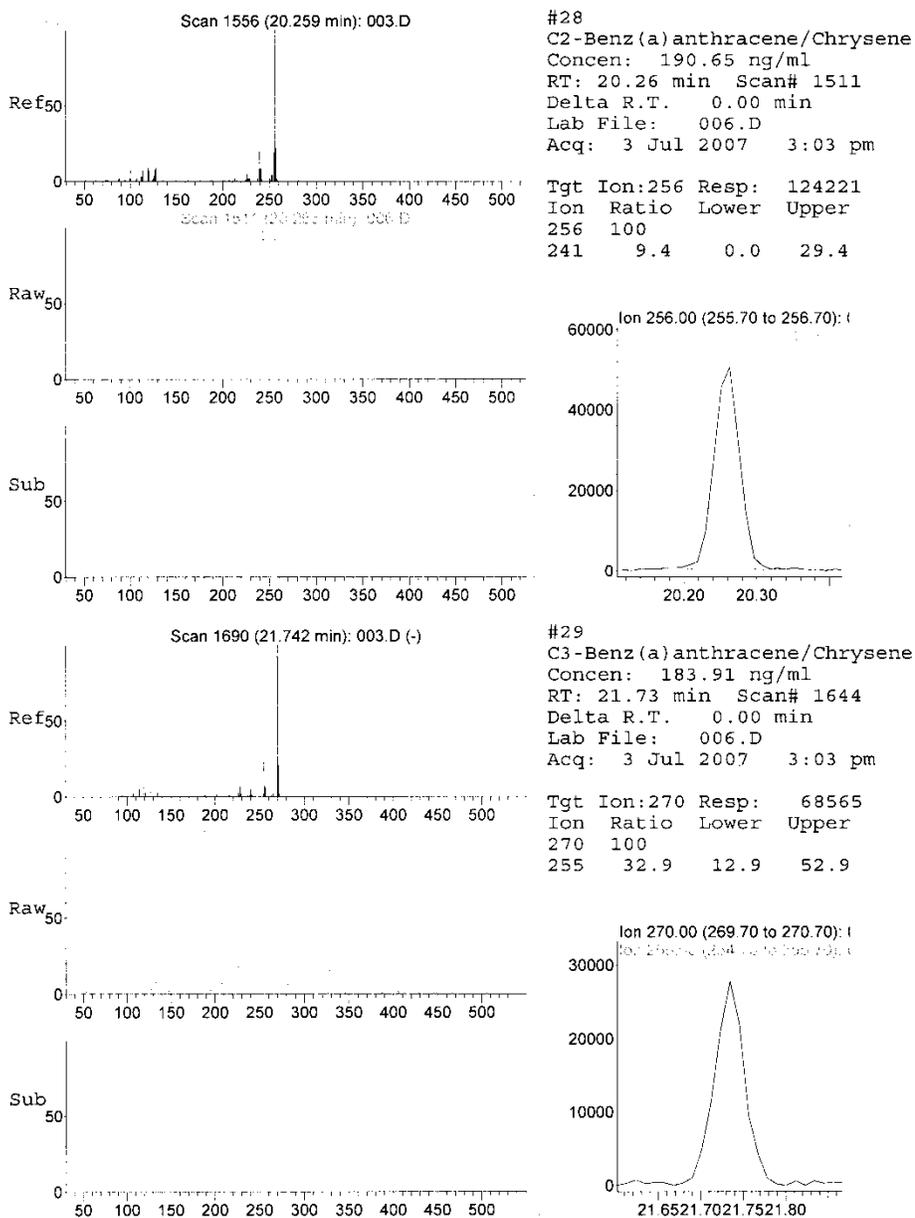




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

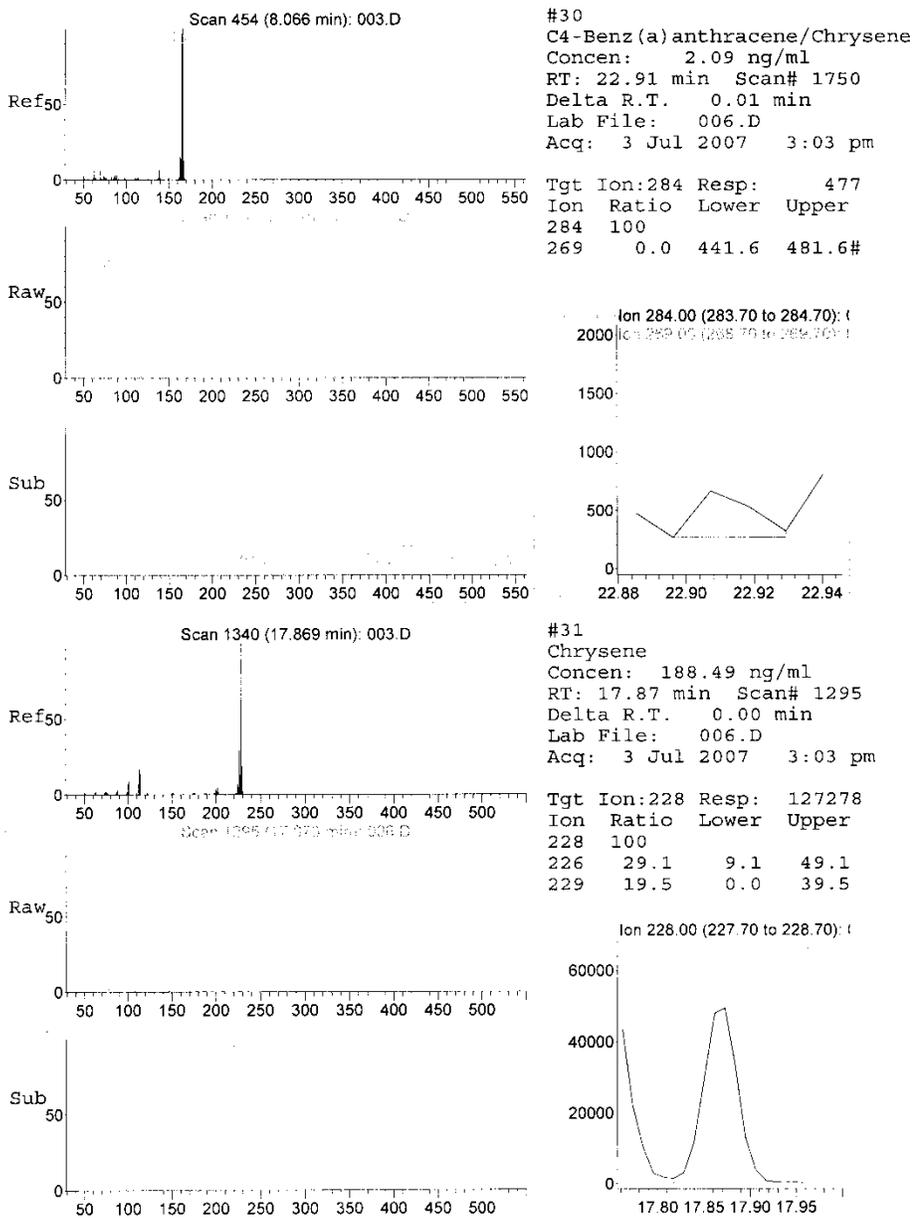




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

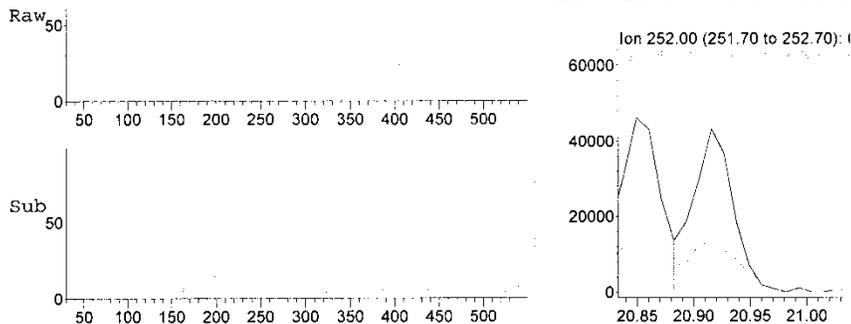
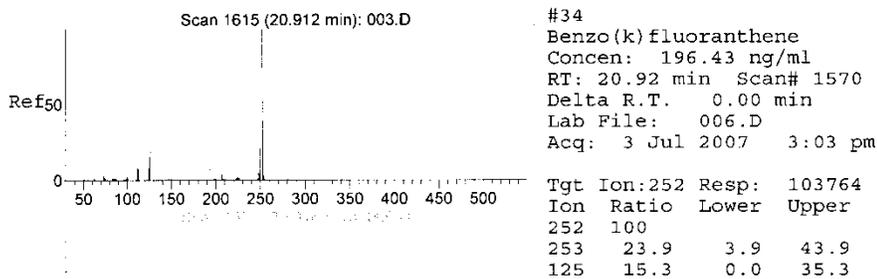
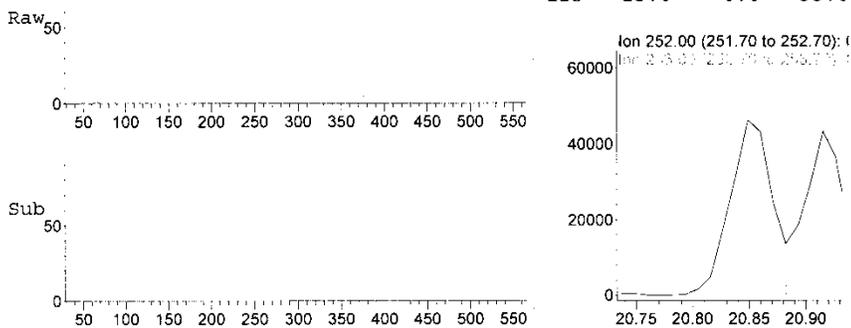
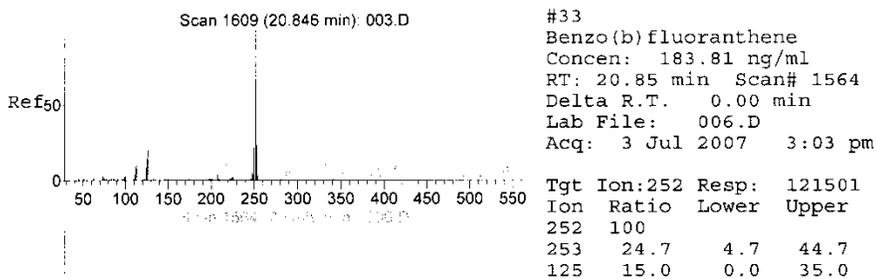
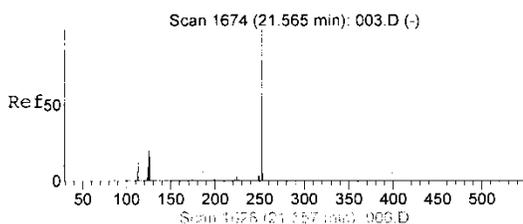




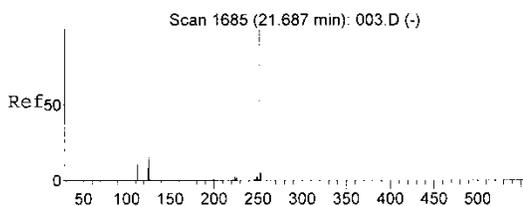
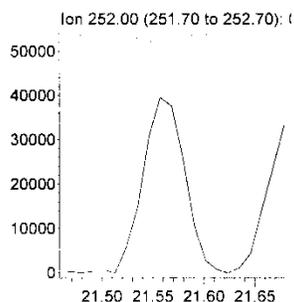
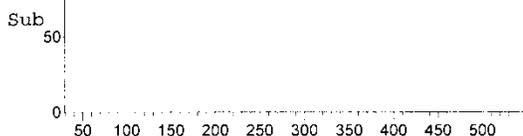
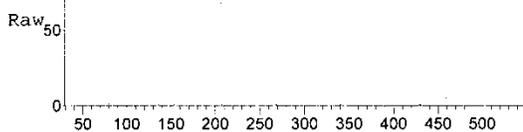
FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV



#35
Benzo(e)pyrene
Concen: 186.97 ng/ml
RT: 21.56 min Scan# 1628
Delta R.T. -0.01 min
Lab File: 006.D
Acq: 3 Jul 2007 3:03 pm

Tgt Ion	Resp	Lower	Upper
252	112009		
252	100		
253	33.5	13.5	53.5
125	19.8	0.0	39.8



#36
Benzo(a)pyrene
Concen: 198.26 ng/ml
RT: 21.68 min Scan# 1639
Delta R.T. -0.01 min
Lab File: 006.D
Acq: 3 Jul 2007 3:03 pm

Tgt Ion	Resp	Lower	Upper
252	102120		
252	100		
253	16.0	0.0	36.0
125	14.5	0.0	34.5

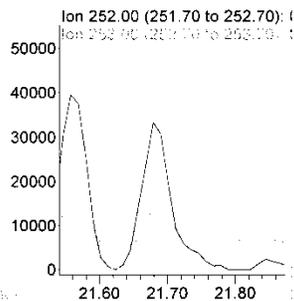
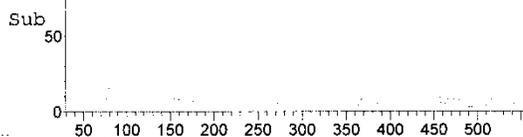
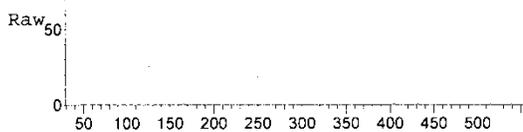




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

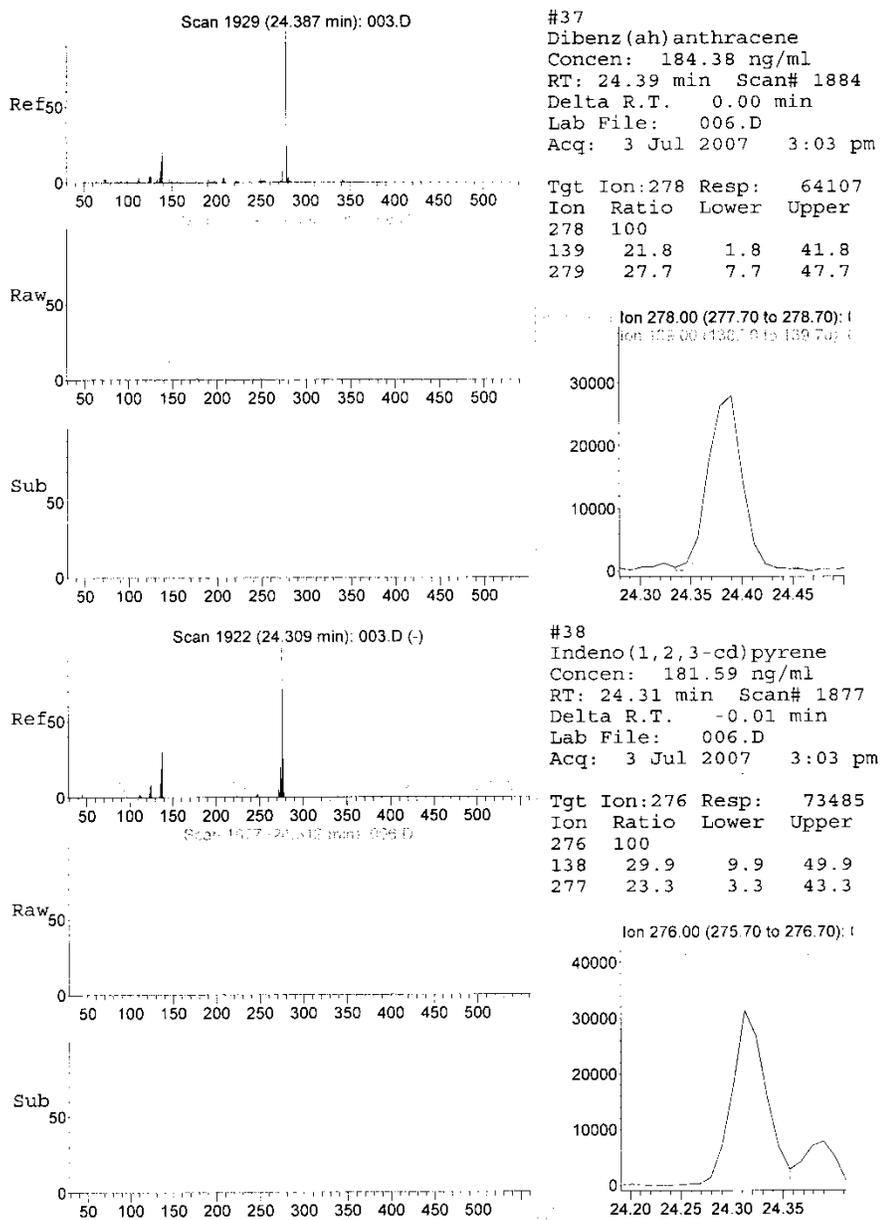




FIGURE 2 (CONTINUED)

TOTAL ION CHROMATOGRAM OF A CCV

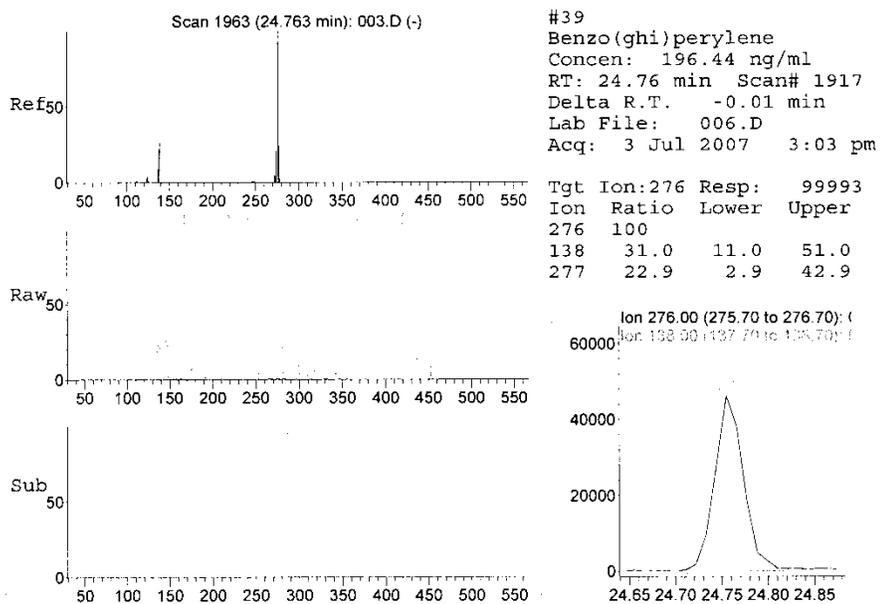




FIGURE 3

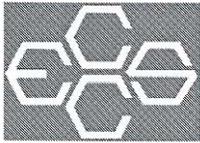
DATA SYSTEM SUMMARY OF %RSD AND AVERAGE RESPONSE FACTORS

Response Factor Report GC/MS Ins

Method : K:\HPCHEM\1\METHODS\APAH0703.M (RTE Integrator)
Title : PAHs by GC/MS
Last Update : Tue Jul 10 13:05:55 2007
Response via : Initial Calibration

Calibration Files
1 =009.D 2 =008.D 3 =007.D
4 =006.D 5 =005.D 6 =004.D

Compound	1	2	3	4	5	6	Avg	%RSD
1) Acenaphthene-d10	-----ISTD-----							
2) Naphthalene	2.313	2.007	2.096	2.016	2.099	2.123	2.128	5.30
3) C1-Naphthalene	1.165	1.295	1.279	1.364	1.246	1.308	1.290	5.49
4) C2-Naphthalene	1.143	1.073	1.115	1.034	1.042	1.032	1.075	4.00
5) C3-Naphthalene	0.936	0.844	0.875	0.831	0.872	0.887	0.878	3.98
6) C4-Naphthalene	0.549	0.503	0.505	0.503	0.554	0.543	0.534	5.73
7) Acenaphthylene	1.911	1.824	1.813	1.933	1.940	1.951	1.923	4.76
8) Acenaphthene	1.118	1.052	1.002	1.097	1.087	1.061	1.077	3.95
9) Fluorene	1.164	1.125	1.266	1.160	1.164	1.228	1.198	4.91
10) C1-Fluorene	0.717	0.674	0.619	0.673	0.692	0.714	0.693	6.43
11) C2-Fluorene	0.452	0.383	0.367	0.352	0.402	0.397	0.395	8.33
12) C3-Fluorene	0.300						0.300	0.00
13) Phenanthrene	1.572	1.551	1.485	1.588	1.591	1.628	1.583	3.64
14) Anthracene	1.391	1.309	1.485	1.307	1.422	1.564	1.446	8.81
15) Chrysene-d12	-----ISTD-----							
16) C1-Phenanthrene/Ant	1.334	1.313	1.287	1.290	1.295	1.321	1.306	1.35
17) C2-Phenanthrene/Ant	0.699	0.673	0.685	0.756	0.719	0.718	0.708	3.78
18) C3-Phenanthrene/Ant	0.579	0.550	0.489	0.518	0.548	0.519	0.530	5.77
19) C4-Phenanthrene/Ant	0.350	0.367	0.383	0.376	0.363	0.381	0.371	3.17
20) Fluoranthene	1.773	1.469	1.525	1.437	1.508	1.498	1.525	7.41
21) Pyrene	1.739	1.659	1.543	1.487	1.533	1.523	1.573	5.77
22) C1-Fluoranthene/Pyr	1.086	0.838	0.849	0.853	0.883	0.882	0.898	9.54
23) C2-Fluoranthene/Pyr	0.650						0.650	0.00
24) C3-Fluoranthene/Pyr	0.350						0.350	0.00
25) S p-Terphenyl	0.938	0.909	0.910	0.880	0.920	0.913	0.908	2.24
26) Benzo(a)anthracene	1.155	1.036	1.063	1.054	1.062	1.065	1.083	4.29
27) C1-Benz(a)anthracen	0.804	0.788	0.800	0.803	0.865	0.855	0.837	6.57
28) C2-Benz(a)anthracen	0.908	0.851	0.905	0.970	1.059	1.075	1.000	13.10
29) C3-Benz(a)anthracen	0.628	0.527	0.525	0.526	0.562	0.578	0.572	9.43
30) C4-Benz(a)anthracen	0.350						0.350	0.00
31) Chrysene	1.169	0.937	0.975	0.988	1.041	1.029	1.036	7.86
32) Perylene-d12	-----ISTD-----							
33) Benzo(b)fluoranthen	1.620	1.183	1.190	1.190	1.274	1.273	1.294	11.91
34) Benzo(k)fluoranthen	0.912	0.944	1.018	1.016	1.048	1.075	1.034	9.93
35) Benzo(e)pyrene	1.179	1.169	1.170	1.097	1.207	1.147	1.173	3.92
36) Benzo(a)pyrene	0.934	0.947	0.854	1.000	1.022	1.076	1.009	11.85
37) Dibenz(ah)anthracen	0.615	0.578	0.606	0.645	0.668	0.702	0.681	18.62
38) Indeno(1,2,3-cd)pyr	0.886	0.667	0.692	0.720	0.759	0.773	0.792	16.97
39) Benzo(ghi)perylene	0.942	0.937	0.904	0.979	0.996	0.993	0.997	10.69



The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

Reviewed By:

Richard Johnson, Operations Manager

01/01/08

Date

Nick Nigro, President

07/01/08

Date

Approved By:

Gregory J. Graf, Quality Manager

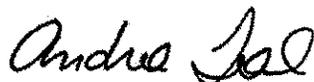
7/1/2008

Date

DIGESTION PROCEDURES FOR SOLIDS FOR ICP & ICP/MS

(Method: EPA 3050B)

Approvals (Signature/Date):



September 13, 2007

Andrea Teal
Quality Assurance Manager

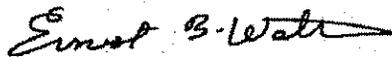
Date



September 13, 2007

Benjamin Gulizia
Laboratory Director/Lead Technical Director

Date



September 20, 2007

Ernest Walton
Health & Safety Manager / Coordinator

Date

Note: Any reference within this document to Severn Trent Laboratories, Inc. or STL should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.).

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Facility Distribution No. 1

Distributed To: QA Navigator

SOP REVISION / REVIEW SUMMARY

SOP Number: ME51:09.20.07:4

Review Date: September 20, 2007

Effective Date: October 15, 2007

Review / Revision Type:

Analytical/procedural revisions

- Revision # change. Completed SOP Training Forms are required.

Summary of Changes:

- Minor grammatical and editorial changes.
- Changed Laboratory name from STL Savannah to TestAmerica Savannah.
- Removed reference to SOP AN99: *Definitions, Terms, and Acronyms*. This SOP is now obsolete. Definitions are now contained in the glossary in the Laboratory Quality Manual.
- Revised all referenced SOP titles to be consistent with current revisions.
- Revised safety information to include location of electronic copies of the MSDS. Section 3.0.
- SOP CA70 is obsolete. Removed reference to this SOP and added reference to CSM for waste disposal information. Sections 3 and 15.
- Included information on preventative maintenance from SOP QA09, Section 14
- Removed the option to keep waste and oils at room temperature prior to preparation. All waste and oil samples must be kept at <6°C but not frozen. Section 5.0
- Removed references to hot plates and beakers.
- Changed the requirement to record the water temperature of the digestion block with each batch instead of daily. Section 6.1
- Updated the Apparatus and Materials list. - Section 6.0
- Revised reagent expiration dates to follow manufacturer's expiration dates. Section 7.
- Included the requirement to use a blank matrix for the method blank. Section 9.0

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used to digest soil, sediment, waste, biological tissue, and oil samples for the determination of total metals prior to analysis by ICP (SOP ME70: *Elements by ICP*) or ICP/MS (SOP ME74: *Elements by ICP/MS*).

The routine target analytes, reporting limits (RL), method detection limits (MDL), and the accuracy and precision limits associated with this procedure are listed in the LIMS Method Limit Groups (MLGs).

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 A known weight (approximately 1g) of the well-mixed sample is transferred to a suitable digestion vessel. The sample is digested with aliquots of nitric acid and hydrogen peroxide to break down the organics present in the sample. After the sample has been digested, as evidenced by a clear, pale yellow digestate, HCl is added to give an approximate acid concentration of 1% HCl and 5% HNO₃. Then the sample digest is diluted to 100mL with reagent water.

A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits.

2.2 Definitions – Refer to the Glossary Section of the Laboratory Quality Manual (LQM) for a complete listing of applicable definitions.

2.3 This SOP is based on the guidance in SW-846 Method 3050B.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual (CSM), the TestAmerica Savannah Addendum to the CSM, and this document.

3.1 Specific Safety Concerns or Requirements

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

3.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the procedure. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in this procedure can be found in the Reagents and

Materials section of this SOP. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Electronic copies of MSDS can be located on the EH&S page on Oasis.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns or permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2ppm-TWA 4ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Method blanks must be analyzed as a check on contamination due to the sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

The following table lists the routine sample containers, preservatives, and storage and holding time information:

MATRIX	ROUTINE CONTAINER	PRESERVATIVE	SAMPLE STORAGE	SAMPLE HOLDING TIME	DIGEST STORAGE
Soils and Sediments	500-mL plastic	None	<6°C but not frozen	6 months from date of collection to complete preparation and analysis (except for Hg which is 28 days from date of collection to complete prep and analysis)	Ambient
Wastes and Oils	500-mL plastic or glass*	None	<6°C but not frozen	6 months from date of collection to complete preparation and analysis (except for Hg which is 28 days from date of collection to complete prep and analysis)	Ambient
Biological Tissues	500-mL plastic or glass	None	Frozen until the time of sample preparation	6 months from date of thawing to complete preparation and analysis (except for Hg which is 28 days from date of thawing to complete prep and analysis)	Ambient

*Some organic wastes may destroy plastic containers

6.0 APPARATUS AND MATERIALS

- 6.1 Digestion block capable of maintaining a sample temperature of $95 \pm 5^\circ\text{C}$: The temperature of the digestion block must be monitored and recorded for each batch. The temperature is measured in a beaker or digestion vessel containing reagent water.
- 6.2 Digestion vessels, appropriate volume for use with digestion block - verify for volume accuracy per lot in accordance with SOP AN30: *Pipette and Volumetric Container Calibration Verification*.
- 6.3 Volumetric flasks - appropriate volumes, verify in accordance with SOP AN30: *Pipette and Volumetric Container Calibration Verification*
- 6.4 Graduated cylinder – appropriate volumes, verify in accordance with SOP AN30: *Pipette and Volumetric Container Calibration Verification*
- 6.5 Pipettes – appropriate volumes, verify in accordance with SOP AN30: *Pipette and Volumetric Container Calibration Verification*.
- 6.6 Top-loading balance – calibrate in accordance with SOP AN10: *Balance Calibration and Use*
- 6.7 Blank Matrix – Ottawa sand, boiling stones, or Teflon chips are to be used as the blank matrix for method blank, LCS/LCSD, and MDLs. These materials must be shown to be free from contaminants prior to use.

7.0 REAGENTS

Reagents must be tracked in accordance with SOP AN41: *Reagent and Standard Materials Traceability*.

7.1 Reagent water-lab generated deionized water. ASTM Type I. The conductivity must be checked daily in accordance with SOP AN35: *Conductivity Checks for Laboratory Deionized Water*.

7.2 Nitric acid (HNO₃) - reagent grade. The manufacturer's certification sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals. The expiration date for this type of reagent is based on the manufacturer's expiration date. Store this reagent at room temperature in the acid cabinet.

7.3 Nitric acid solution (1:1) - Measure 500mL of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly, add 500mL of concentrated nitric acid (HNO₃) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled container suitable for storing acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed. The expiration date for this type of reagent is based on the manufacturer's expiration date. Store this reagent at room temperature.

CAUTION: HEAT WILL EVOLVE AS THE NITRIC ACID MIXES WITH THE WATER. THIS SOLUTION WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING.

7.4 Hydrochloric acid (HCl) - reagent grade. The manufacturer's certification sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals. The expiration date for this type of reagent is based on the manufacturer's expiration date. Store this reagent at room temperature in the acid cabinet.

7.5 Hydrochloric acid solution (1:1) - Measure 500mL of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly, add 500mL of concentrated hydrochloric acid (HCl) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled storage container suitable for acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed. The expiration date for this type of reagent is based on the manufacturer's expiration date. Store this reagent at room temperature.

CAUTION: HEAT WILL EVOLVE AS THE HYDROCHLORIC ACID MIXES WITH THE WATER. HYDROCHLORIC ACID HAS A SUFFOCATING ODOR AND MUST BE USED UNDER THE HOOD. THIS SOLUTION WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING. PREPARE THIS SOLUTION UNDER A HOOD.

7.6 Hydrogen peroxide (H₂O₂), 30% - reagent grade. The expiration date for this type of reagent is based on the manufacturer's expiration date. Store this reagent in the refrigerator.

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Reagent and Standard Materials Traceability*. General guidance on the preparation of standards is also given in SOP AN41.

The lab should purchase certified solutions from TestAmerica-approved vendors, if available. The lab

should prepare standards from neat materials only if a certified solution is not available. See SOP AN41 for guidance for standard preparation.

8.1 ICP Spiking Solution 1 is a purchased solution containing the following elements: aluminum, arsenic, barium, selenium, thallium at 200mg/L; iron at 100mg/l; cobalt, magnesium, nickel, lead, strontium, vanadium, antimony at 50mg/L; copper at 25mg/L; chromium at 20mg/L; and silver, beryllium, cadmium at 5mg/L. Store this solution at room temperature. This standard must be used prior to the manufacturer's expiration date.

8.2 Preparation of the ICP Spiking Solution 2

Add 20-30mL reagent water to a clean 100-mL volumetric flask. Add 1mL of concentrated nitric acid and 5mL of hydrochloric acid to the volumetric flask. The standard will have an acid concentration of 1% HNO₃ and 5% HCl when diluted to volume.

Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	Vol. of Stock (mL)	Final Vol. (mL)	Conc. of std. (mg/L)
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0	100	500
Magnesium (Mg)	10000	5.0	100	500
Molybdenum (Mo)	1000	5.0	100	50
Potassium (K)	10000	5.0	100	500
Sodium (Na)	10000	5.0	100	500
Strontium (Sr)	1000	5.0	100	50
Tin (Sn)	1000	10	100	100
Titanium (Ti)	1000	10	100	100

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used within 6 months of preparation or sooner based on the manufacturer's expiration date.

8.3 Preparation of the ICP/MS Spiking Solution 1

Element	Ci (mg/L)	Vi (mL)	Vf (mL)	Cf (mg/L)
Aluminum (Al)	10000	1	100	100
Antimony (Sb)	1000	1		10
Arsenic (As)	1000	0.4		4
Barium (Ba)	1000	2		20
Beryllium (Be)	1000	0.5		5
Boron (B)	1000	2		20
Cadmium (Cd)	1000	0.5		5
Calcium (Ca)	10000	1		100
Chromium (Cr)	1000	2		20
Cobalt (Co)	1000	2		20
Copper (Cu)	1000	2.5		25
Iron (Fe)	10000	1		100
Lead (Pb)	1000	0.5		5
Magnesium (Mg)	10000	1		100
Manganese (Mn)	1000	5		50
Molybdenum (Mo)	1000	2		20
Nickel (Ni)	1000	2		20
Potassium (K)	10000	1		100
Selenium (Se)	1000	0.5		5
Sodium (Na)	10000	1		100
Strontium (Sr)	1000	2		20
Thallium (Tl)	1000	0.5		5
Tin (Sn)	1000	2		20
Titanium (Ti)	1000	2		20
Vanadium (V)	1000	2		20
Zinc (Zn)	1000	2		20
Zirconium (Zr)	1000	0.5		5

- Add 2mL of nitric acid and 0.5mL of hydrochloric acid to a 100-mL volumetric flask containing about 50mL of reagent water.
- Add the appropriate volume of each element stock standard to the flask. Dilute to volume with reagent water, and mix thoroughly.
- Store this solution at room temperature. This standard must be used within 6 months of preparation or sooner based on the manufacturer's expiration date.

8.4 Preparation of the ICP/MS Spiking Solution 2

Element	Ci (mg/L)	Vi (mL)	Vf (mL)	Cf (mg/L)
Mercury (Hg)	1000	0.05	100	0.5
Silver (Ag)	1000	0.5		5

- Add 10mL of hydrochloric acid to a 100-mL volumetric flask containing about 50mL of reagent water.
- Add the appropriate volume of each element stock standard to the flask, dilute to volume with reagent water, and mix thoroughly.
- Store this solution at room temperature. This standard must be used within 28 days of preparation or sooner based on the manufacturer's expiration date.

9.0 SAMPLE PREPARATION

The digestion batch consists of twenty or fewer field samples and the associated QC items. A digestion batch is not to exceed 20 field samples. Every digestion batch will have a method blank (MB), a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD and batch precision is required, then the LCS is performed in duplicate. Some clients may require a sample duplicate (SD) in addition to or instead of an MSD. The MB and LCS are performed using 1g aliquots of blank matrix.

- 9.1 To homogenize a soil sample, follow the procedures given in SOP QA15: *Homogenization, Compositing, and Segregation of Samples*. The sample may be vigorously stirred in the sample container or transferred to a plastic "baggie" and thoroughly mixed by kneading the container. After the sample is homogenized, return only enough sample to the original container to fill it three-fourths full. This will allow the sample to be stirred and homogenized if additional aliquots of the sample are required. Place the discarded sample in a containerized waste receptacle for disposal.
- 9.2 If the sample is a solid material, break up the solid in the baggie by hitting the sample with a hammer or other suitable crushing device. Contact the Department Manager if the matrix is difficult to break up or is difficult to mix.
- 9.3 Weigh 1.0-1.2g (wet weight) of each homogeneous sample into a 125-mL Teflon digestion vessel. The lab may weigh a larger aliquot equal to 1.0g of sample on a dry weight basis, if required.

NOTE: A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits; if 0.1g is digested, the final volume of the digestate must be 10mL. If the sample weight to final volume ratio is less than 1:100, the reporting limits will be higher than those listed in the LIMS MLGs. Adjust the volumes of spikes and reagents proportionately to compensate for the sample weight/final volume. For example, if 0.5g is digested, use ½ of the spike and reagent volumes listed above.

- 9.4 Weigh a 1-g aliquot of blank matrix into a labeled digestion vessel to serve as the method blank.
- 9.5 Weigh a 1-g aliquot of blank matrix into two labeled digestion vessel to serve as the LCS and LCSD (if required). Add 1.0mL of each spiking solution (ICP Spike I and ICP Spike II, or ICP/MS Spike I and ICP/MS Spike II) to the designated laboratory control spikes.
- 9.6 Add 1.0mL of each spiking solution (ICP Spike I and ICP Spike II, or ICP/MS Spike I and ICP/MS Spike II) to the designated matrix spike samples. The MS/MSD are prepared by weighing 1.0-1.2g of the sample chosen for the matrix spike into labeled digestion vessels.
- 9.7 Add 5mL of reagent water and 5mL of concentrated HNO₃ to each digestion vessel and swirl the vessel to mix the contents.
- 9.8 Place the digestion vessels on the digestion block. The water in the digestion block must be 95°C +/-

5°C. Carefully heat the vessel until a gentle reflux is achieved. The sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Do not allow the samples to boil. Reflux for 10-15 minutes.

- 9.9 Remove the digestion vessels from the digestion block, and allow the beakers to cool to room temperature. Add 5mL of concentrated HNO₃ to each sample. Return the digestion vessels to the digestion block. Carefully heat the digestion vessels until a gentle reflux is achieved. Reflux the samples for 30 minutes. Do not allow the samples to boil.
- 9.10 Repeat the procedure in Section 9.9 with a second 5-mL portion of concentrated HNO₃ if brown fumes are given off. Repeat Section 9.9 until no brown fumes are given off.
- 9.11 Evaporate the sample digestate to approximately 10mL. Do not allow the bottom of the digestion vessels to go dry during the evaporation. Remove the samples from the digestion block, and allow the samples to cool to room temperature before continuing onto the next step.

NOTE: If the sample is still warm when the 30% H₂O₂ (hydrogen peroxide) is added in the next step, the sample may "boil over" and the entire process must be started over.

- 9.10 Add 2mL of reagent water to each beaker. Slowly and carefully add 3mL of 30% H₂O₂ to each beaker. It is very important to add the hydrogen peroxide slowly to prevent loss of sample due to vigorous effervescence. Return the digestion vessels to the digestion block, and heat until the effervescence subsides. Cool the digestion vessels after the effervescence subsides.
- 9.11 Continue to add 30% H₂O₂ in 1-3mL aliquots to the sample digestate until the effervescence is minimal or until the general appearance of the digestate is unchanged. Warm the sample digestate after each addition of H₂O₂ on the digestion block.

NOTE: Do not add more than 10mL of hydrogen peroxide to each sample.

- 9.12 After the last addition of peroxide, reduce the volume of the digestate to 5-10mL without boiling and without allowing the bottom of the digestion vessel to go dry. Add 10mL of concentrated HCl to each sample digestate. Return the beakers to the digestion block, and reflux the sample digestates for 10-15 minutes.
- 9.13 Wash down the inside of the digestion vessels with reagent water. Dilute the sample digestate to 100mL with reagent water.

10.0 ANALYTICAL PROCEDURES

The analytical procedures associated with this digestion procedure are given in SOP ME70: *Elements by ICP* or SOP ME74: *Elements by ICP/MS*.

11.0 DATA ANALYSIS AND CALCULATIONS

Calculations for the determination of metals by ICP and ICP/MS are given in the associated analytical SOPs (SOP ME70 or SOP ME74).

12.0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 Analytical Batching

The analytical batch consists of up to twenty client samples and the associated quality control items. The minimum quality control items associated with this procedure are: a method blank (MB), a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

If insufficient sample is available for the MS/MSD, this situation must be annotated on the batch log. If batch precision data is required by the client or regulatory program, then the LCS is extracted in duplicate.

Refer to SOP QA17: *Analytical Batching and Evaluation of QC Data*, the analytical SOPs, and the LQM for guidance on the evaluation of the QC in an analytical batch.

12.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP QA05: *Preventive and Corrective Action Procedures*. SOP QA05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

13.0 METHOD PERFORMANCE

The reporting limits (RL), method detection limits (MDL), and accuracy and precision limits associated with these methods are given in the LIMS Method Limit Groups (MLGs).

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP QA06: *Training Procedures*.

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per analyte by the analysis of four IDOCs that meet the method criteria for accuracy and precision. The IDOC must be from a second source than that used to prepare the calibration standards. The IDOC must be documented on the IDOC Form shown in SOP QA06 with documentation routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per analyte. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), or via acceptable results on a PT study.

13.2 Method Detection Limit

The method detection limit must be determined annually for each analyte in accordance with SOP QA07: *Determination of Detection Limits (MDLs and IDLs)*.

14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

Refer to SOP QA09: *Maintenance Procedures for Laboratory Instrumentation* for routine preventive maintenance and the manufacturer's guides for trouble-shooting items.

The temperature of the hot plate or digestion block must be monitored with each batch. If the temperature required for sample preparation cannot be maintained, the heating device must be removed from service and repaired or replaced.

All non-operational equipment must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

15.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Follow the guidance for disposal in the TestAmerica Savannah Addendum to the CSM. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

Excess samples, reagents, and standards must be disposed in accordance with the TestAmerica Savannah Addendum to the CSM.

15.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

Excess soil samples from homogenization procedure - Transfer to TCLP container for characterization in hazardous waste department.

Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

Acidic sample digestions – Neutralize before disposal into drain/sewer system.

Excess oil samples – Transfer to waste department for storage/disposal.

16.0 REFERENCES

TestAmerica Savannah's *Laboratory Quality Manual (LQM)*, current revision

TestAmerica Laboratories' *Quality Management Plan (QMP)*, current revision

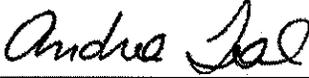
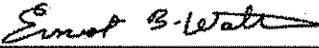
Test Methods for Evaluating Solid Waste, Third Edition, SW-846; EPA Office of Solid Waste and Emergency Response: Washington, DC. (including Update III)

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

There are no tables, diagrams, or validation data associated with this SOP.

ELEMENTS BY ICP

(Methods: EPA 200.7 and 6010B)

Approvals (Signature/Date):			
	07/08/08		07/08/08
Andrea Teal	Date	Benjamin Gulizia	Date
Quality Assurance Manager		Laboratory Director/Lead Technical Director	
	07/08/08		07/08/08
Ernest Walton	Date	Chris Kana	Date
EH&S Coordinator / Technical Manager		Department Manager	

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1.0 Scope and Application

This SOP gives the procedures for the determination of metals (elements) in water, soil, wipe, leachate, tissue, filter, and waste samples by inductively coupled plasma (ICP) atomic emission spectroscopy.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Prior to analysis by ICP, the sample must be digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

Note: Drinking water samples (EPA 200.7) only require digestion if the determination of silver (Ag) is requested or if the turbidity is greater than or equal to 1.0 NTU.

This SOP is based on the following methods: EPA Method 200.7 and SW-846 Method 6010B.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual (QAM)* for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in

accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

- 4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.
- 4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.
- 4.1.5 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample digestate, using a peristaltic pump, using the method of standard additions (MSA), or using an internal standard.

4.2 Matrix Interferences

- 4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences.
- 4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.
- 4.2.3 Spectral interferences are caused by the overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous phenomena, and stray light from the line emissions of highly concentrated elements.
 - 4.2.3.1 Spectral overlap may be compensated for by the use of inter-element correction factors.
 - 4.2.3.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the

responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

The plasma generates high temperatures. Ensure that all equipment is shut down and cooled off before performing maintenance and troubleshooting in the plasma area.

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Electronic copies of MSDS can be found using the "MSDS Online" button on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
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Hydrochloric Acid ⁽²⁾	Corrosive Poison	5ppm - Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid ⁽²⁾	Corrosive Oxidizer Poison	2ppm - TWA 4ppm - STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			
2 – Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Thermo Jarrell Ash TJA ICAP61E-trace, Varian 730 ES, or other suitable inductively coupled plasma emission spectrometer with data system

Top-loading Balance – Verify in accordance with SOP AN10: *Balance Calibration and Use*

6.2 Lab Supplies

Argon gas supply and appropriate fittings

Cooling water supply

Peristaltic pump

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP AN30: *Pipette and Volumetric Container Calibration Verification*

Pump-style Pipettes – various sizes. Verify in accordance with SOP AN30: *Pipette and Volumetric Container Calibration Verification*

Disposable Graduated Pipettes – various sizes. Verify in accordance with SOP AN30: *Pipette and Volumetric Container Calibration Verification*

pH paper – provides a quick and easy way to approximate the pH of a sample to determine if a sample has been properly preserved or if the pH of a sample is in the

proper range for a preparation step. pH paper should be checked upon receipt, as follows, to make sure that it is functioning properly.

- Examine the pH paper. If the paper is discolored or looks worn, it may be defective.
- Place a piece of pH paper on a watch glass or other suitable surface and add a few drops of a certified buffer solution onto the paper.
- Compare the color of the pH paper to the reference colors. If the colors match, the paper can be used. If not, acquire new paper.

Detergent – used for washing non-disposable labware.

6.3 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine containers provided by the laboratory are as follows:

Liquids: 250mL Plastic – purchased with Certificate of Analysis attesting to purity.

Liquids: 8oz Plastic Jar – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it; that is, the "children may not outlive the parents".

Unless listed elsewhere in this SOP, the expiration dates given below apply.

- 7.1.1 The expiration date for unopened standards and reagents is the manufacturer's expiration date.
- 7.1.2 The expiration date for opened stock reagents is the manufacturer's expiration date or 5 years from the date opened, whichever is sooner.
- 7.1.3 The expiration date for opened stock standards is the manufacturer's expiration date.
- 7.1.4 The expiration date for prepared reagents is 6 months from the date prepared or the expiration date of the parent reagent, whichever is sooner.
- 7.1.5 The expiration date for prepared standards is 6 months from the date prepared or the expiration date of the parent standard, whichever is sooner.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP AN41: *Reagent and Standard Materials Traceability*.

Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Laboratory Reagent Water – ASTM Type I. The conductivity must be monitored in accordance with SOP AN35: *Conductivity Checks for DI Water*.

7.2.2 Nitric acid (HNO₃) – reagent grade. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.

7.2.3 Hydrochloric acid (HCl) – reagent grade. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.

7.3 Standards

Standards must be prepared and documented in accordance with SOP AN41: *Reagent and Standard Materials Traceability*. Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

Refer to Attachment 5 for standard preparation information.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in 250-mL plastic containers containing 1.5 mL 1:1 nitric acid preservative or 500-mL plastic containers containing 3.0 mL 1:1 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

If dissolved metals are requested the sample must be filtered prior to the addition of the preservative. If the sample is to be filtered by the laboratory, the sample must be collected in a 250-mL plastic container without preservative. The samples are acidified with 1.25mL concentrated nitric acid to a pH <2 after filtration.

Samples are stored at room temperature until the time of digestion. Samples must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and analyzed within 6 months of collection.

8.2 Soil Samples

Soil samples are routinely collected in 8-oz plastic containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of digestion. Samples must be digested within 6 months of collection. Digestates must be stored at room temperature until the time of analysis and analyzed within 6 months of collection.

8.3 Waste (Oil) Samples

Waste (oil) samples are routinely collected in 500-mL plastic containers.

Samples are stored at room temperature until the time of digestion. Samples must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and analyzed within 6 months of collection.

8.4 Tissue Samples

Tissue samples are routinely collected in plastic containers with the size dependent upon the type of tissue being collected. Plastic jars or plastic baggies can be used.

Upon receipt, samples must be placed in the freezer at -10° to -20°C if digestion cannot be completed that day. Samples must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and analyzed within 6 months of collection.

8.5 Wipe Samples

Wipe samples are routinely collected in 40-mL VOA vials. Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until time of digestion. The sample must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and analyzed within 6 months of collection.

Refer to SOP PS25: *Wipe Tests: Sampling and Analysis* for additional information on wipe procedures.

8.6 TCLP/SPLP Leachate Samples

Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a 500mL plastic container and preserved with 1.0mL nitric acid to a pH <2. TCLP/SPLP leachates are stored at room temperature until the time of digestion. The leachate sample must be digested within 6 months of completion of the TCLP/SPLP extraction. Digestates are stored at room temperature until the time of analysis and analyzed within 6 months of completion of the TCLP/SPLP extraction.

8.7 Filter Samples

Filter samples are non-routine. Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until time of digestion. The sample must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and analyzed within 6 months of collection.

9.0 Quality Control

SOP QA17: *Analytical Batching and Evaluation of QC Data* and the SOP Summary in Attachment 4 provide requirements for evaluating QC data.

9.1 Batch QC

A digestion batch consists of up to 20 environmental samples and the associated QC items. The minimum QC items required for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS) and either a matrix spike duplicate (MSD) or a sample duplicate (SD). If there is insufficient sample to perform the MS/MSD or sample duplicate, this situation must be noted in the Batch Information section of the digestion log.

Refer to the following SOPs for specifics on the preparation process:

MATRIX	SOP
Aqueous samples	ME50
Leachate samples	ME50
Soil samples	ME51
Waste (oil) samples	ME51
Tissue Samples	ME51
Wipe Samples	ME51
Filter Samples	ME51

Batch QC must meet the criteria given in Attachment 4 of this SOP.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP QA16: *Evaluation of Calibration Curves*. This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of at least one standard (the high standard) and a blank.

Refer to Attachment 5 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP AN41.

If a multi-point curve is requested the calibration curve consists of a minimum of 3 standards and a blank. The initial calibration standard concentrations currently in use in the laboratory for a multi-point curve included in Attachment 5.

Tabulate the concentrations and corresponding responses for each analyte. Establish a calibration curve by plotting the concentration along the x-axis and the corresponding response along the y-axis.

The correlation coefficient (r) of the regression curve must be greater than 0.995 for the initial calibration curve to be acceptable.

If any calibration regression fit, other than linear, is utilized for the calibration of the ICP (i.e., Curvilinear or Full Fit), the upper limit of the linear range is the concentration of the High Standard.

9.2.2 Highest Standard

The highest concentration calibration standard is reanalyzed as an "unknown" after the instrument is calibrated. The results for the re-analysis of the highest concentration calibration standard must be within $\pm 5\%$ of the true value for each target analyte. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.

9.2.3 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP QA16 with a standard obtained from a second source.

The ICV must be within 10% of the true value to be acceptable for SW-846 Method 6010 and 5% of the true value with a %RSD $\leq 3\%$ to be acceptable for EPA Method 200.7.

Note: for the Thermo Trace instrument, 2 replicates are analyzed. For the Varian 730 ES instrument, 3 replicates are analyzed.

The initial calibration verification standard concentration currently in use in the laboratory is given in Attachment 5. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP AN41.

9.2.4 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis on calibration blanks. Initial calibration blanks are analyzed immediately following the initial calibration. Continuing calibration blanks are analyzed immediately following each continuing calibration verification (CCV).

The absolute value of the initial and continuing calibration blanks must be less than 3x the IDL or less than the RL/CRDL, whichever is smaller, to be acceptable for SW-846 Method 6010. The absolute value of the initial and continuing calibration blanks must be less than the RL/CRDL to be acceptable for EPA Method 200.7.

9.2.5 Continuing Calibration Verification

The initial calibration curve must be verified every 10 samples with a mid-level standard.

The CCV must be within 10% of the true value to be acceptable.

The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to level 1/2 of the High Standard concentration. Refer to

Attachment 5 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP AN41.

9.2.6 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. Yttrium is used as the internal standard for the Thermo Trace ICP (ICP-D) and the Varian 730ES ICP (ICP-E). Scandium may be used as an alternate ISTD for the Varian 730ES ICP.

The internal standard must be added to all standards, samples, and QC items prior to analysis. This is accomplished by means of an additional channel on the peristaltic pump and connected to the sample line with a 'T' or 'Y' connector fitting. This ensures constant concentration of the internal standard and eliminates the possibility of human spiking error. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. A concentration of 7mg/L is used for Yttrium.

Any sample containing ISTD recoveries greater than 120% should be diluted and re-analyzed. Although ISTD recoveries less than 50% are extremely rare, the analyst should consider further dilution if this situation occurs.

9.2.6.1 Ionization Suppressants

High concentrations of some elements, such as Na, K, Ca, and Mg, can produce interferences from their ionization effects. The introduction of an Ionization Suppressant in abundance, such as Lithium, will minimize this interference. Therefore, Lithium is added to the internal standard solution, and is continually pumped at a constant rate.

See Attachment 5 for details on the preparation of the internal standard solution.

9.2.7 Reporting Limit Check Standard

When a single point calibration is utilized a reporting limit check standard must be analyzed to demonstrate that the ICP is capable of detecting the target compounds at or below the reporting limit (RL). The element concentrations in the RL check standard must be at levels that are less than or equal to the reporting limit for the samples being analyzed. The determined concentration must be detected within $\pm 50\%$ of the true concentration. If a multi-point calibration curve is used that includes a standard at or below the RL, the RL check standard is not required.

9.2.8 Interference Check Standards

The purpose of the Interferent Check Standard is to prove that the instrument software is adequately correcting for common interferences through the use of interelement correction factors. The concentrations of the target analytes must be within 20% of the true concentrations. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

9.2.9 Serial Dilution

A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present. Compare the results of the diluted and un-diluted aliquots of

sample digestate for analytes that are present in the native sample at a concentration $\geq 50 \times$ IDL.

If the results of the dilution are within $\pm 10\%$ of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than $\pm 10\%$, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike.

9.2.10 Post-Digestion Spike

A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. This should be the same sample selected for serial dilution.

The post-digestion spike must be within 25% of the true value to be acceptable.

Result of Post-Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample
<75% recovery but >50% recovery	1) Dilute and re-spike. Elevate RL accordingly (for all associated samples). 2) Spike and evaluate all associated samples. 3) Spike and evaluate all associated samples by single point MSA 4) Qualify all associated samples
<50% recovery	Dilute digestate and repeat spike. Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples) If recoveries are not 75-125%, analyze all associated samples by single point MSA. Note – high level of target analytes may inhibit spike recovery. Consult the supervisor in events where high levels of targets appear to be interfering

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high if corrected for spike recovery.

9.2.11 Single Point Method of Standard Additions

Two identical aliquots of the sample digest are taken. One aliquot is spiked with a solution of known concentration. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The concentrations of both aliquots are measured and the sample concentration is calculated.

Note: The post-digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgement when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyzed to determine the extent of the matrix interferences.

9.2.12 % RSD of Multiple Exposures

The ICV must be within 10% of the true value to be acceptable for SW-846 Method 6010 and 5% of the true value with a %RSD $\leq 3\%$ to be acceptable for EPA Method 200.7.

The Thermo Trace instrument performs 2 replicates of each sample. The Varian 730 ES instrument performs 3 replicates of each sample. To be acceptable, the %RSD must be $< 30\%$ for samples with concentrations above the RL.

9.2.13 Interelement Correction Factors (IEC)

Interelement correction factors (IEC) for all elements must be determined annually using the manufacturer's guidance.

The lab may combine the linearity study with the IEC study, thereby eliminating redundancy. (Single element linearity check solutions for each analyte should be closely evaluated for all non-spiked elements, and applicable correction factors should be applied according to the instrument's software. The analyst must be careful not to correct for any contamination which may be present in the actual solution.)

The IECs must be verified at the beginning and end of each analytical sequence through the analysis of interferent check solutions ICSA and ICSAB.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP QA05: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 4. SOP QA05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

The sample preparation procedures are given in the following SOPs:

Matrix	SOP #
Aqueous samples	ME50
Leachate samples	ME50
Soil samples	ME51

Waste (oil) samples	ME51
Tissue Samples	ME51
Wipe Samples	ME51
Filter Samples	ME51

10.2 QC Sample Preparation

10.2.1 Serial Dilution

Dilute the digestate by a factor of 5 and analyze the dilution using the same procedures used for the un-diluted aliquot.

10.2.2 Post-Digestion Spike

Transfer 10mL of a digestate to a suitable vial. Spike the sample with 0.10mL of CLP Spike I and 0.10mL of Spike II. The theoretical concentration of the post digestion spike is the same as the LCS or MS if the volume of spiking solution is discounted.

10.3 Analysis

10.3.1 Instrument Operating Conditions

Turn the ICP on and allow it to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If the optics were turned off, allow 2 hours warm up time.

Run the "Automatic Profile" program. The "automatic profile" of the instrument should be checked twice a day to compensate for changes in air pressure, humidity, and temperature. If the environment of the instrument is such that daily changes in the instrument profile are extreme, the instrument should be "profiled" every few hours.

Instrument maintenance must be performed in accordance with Attachment 2 of this SOP.

10.3.2 Internal Standard (ISTD)

Prior to analysis, internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples.

10.3.3 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described in Section 9.2.1. Once the calibration has been established and verified with a high level standard and an ICV in accordance with Sections 9.2.2 and 9.2.3, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5.

10.3.4 Sample Analysis

Remove the digestates from the refrigerator and allow them to come to room temperature.

The sample digestate must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The default procedure is to include QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in the clock.

10.3.5 Example Analytical Sequence

An example analytical sequence is listed below.

Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Instrument Warm-up Profile	
Initial Calibration	
High Calibration Standard	Re-analyzed as a sample
ICV	Second Source
ICB	
Reporting Limit Check Standard	
ICP Interference Check Solution A (ICSA)	
ICP Interference Check Solution AB (ICSAB)	
CCV	10-injection clock begins with injection of the CCV
CCB	
Samples & Batch QC Items	Up to 10 injections, including QC.
CCV	10-injection clock begins with injection of the CCV
CCB	
Samples & Batch QC Items	Up to 10 injections, including QC.
CCV	10-injection clock begins with injection of the CCV
CCB	
Samples & Batch QC Items	Up to 10 injections, including QC.
CCV	10-injection clock begins with injection of the CCV
CCB	

Note: The analytical sequence must end with the analysis of the detection limit check standard, ICSA, ICSAB, CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

11.0 Calculations / Data Reduction

11.1.1 Dilutions

If the concentration of a sample is above the linear range of the ICP, as determined in Attachment 6, the sample digestate must be diluted and reanalyzed.

Dilutions must be prepared in reagent water containing 5% hydrochloric acid and 1% nitric acid by volume.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes to determine if historical data is available for review.

11.1.3 Chemical Relationships

The analyst must be aware of the following chemical relationships:

- Total Results should be \geq Dissolved results

11.1.4 MARRS

Savannah uses the Environmental Information Systems Corporation MARRS program for data reduction and reporting. The following is the procedure used:

An archive file is sent via the laboratory network to a second PC. The MARRS software uploads the archive file, and compares the data to the quality control parameters that are built into the software. These parameters may be customized to meet specific project requirements.

11.1.4.1 When the data file is uploaded, the analyst reviews the data to ensure that the QC types are correct. The QC types are: samples, calibration standards, ICV, ICB, CRI, ICSA, ICSAB, CCV, CCB, prep blanks (liquid, solid), LCS (liquid, solid), serial dilutions, post-digestion spikes, MS/MSD, DUP, etc. If any typographical errors are noted by the instrument analyst on the instrument's summary report, then these errors need to be corrected in the MARRS system.

11.1.4.2 The sample data are then compared to the tightest limits for the samples on the run. For example, most of the analysis run may include data that only require CCVs to be 90-110%, but part of the analysis run may need the CCVs to be 95-105%. In this instance, the entire run is compared to the 95-105% limits. There are tables set up with the tightest criteria required. These tables are used for the initial data evaluation.

11.1.4.3 After the results are processed, a data review report is printed that shows the samples and QC that exceed the acceptable limits. When the report shows that acceptable limits are exceeded, the analyst will determine if the element is required for the project. If the element is not required, this is noted on the data review report. If the element is required, a reanalysis is initiated for that sample and element.

11.1.4.4 A report is printed that shows that the correct number of CCV/CCBs were analyzed with the samples. If more than 10 samples are analyzed between CCV/CCBs, then all affected samples are reanalyzed.

11.1.4.5 When the data reduction is complete, all compliant data are reported to the LIMS system.

11.2 Calculations

- 11.2.1 The calculations associated with batch QC determinations are given in SOP QA17. Applicable calculations include accuracy (% recovery) and precision (%RPD).
- 11.2.2 The calculations associated with initial and continuing calibrations are given in SOP QA16. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.
- 11.2.3 The calculation to determine final concentration is given as follows:

$$Final\ Concentration = \frac{C_{ISTD}}{AREA_{ISTD}} \otimes AREA_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

C_{ISTD} = Concentration of the internal standard
 $AREA_{ISTD}$ = Total ion peak area of the internal standard
 $AREA_{Sample}$ = Total ion peak area of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor
dw = % Moisture decimal equivalent

Note: All dry weight corrections are performed automatically in LIMS.

11.2.4 Method of Standard Additions

The concentrations of both sample aliquots, Section 9.2.11, are measured and the sample concentration is calculated.

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

Where:

S_1 = Absorbance or concentration of the spiked aliquot
 S_2 = Absorbance or concentration of the un-spiked aliquot
 V_s = Volume of spike solution
 V_x = Volume of sample aliquots
 C_s = Spike solution concentration

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDL associated with this procedure is given in the Method Limit Group (MLG) in LIMS.

At a minimum, the MDL must be determined initially upon method set-up and annually thereafter, and verified annually in accordance with SOP QA07: *Determination of Detection Limits (MDLs and IDLs)*.

12.2 Determination of the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the concentration of analyte that can be statistically distinguished from the background noise of the instrument. The IDL limit must be determined annually, at a minimum, for each analyte in accordance with SOP QA07: *Determination of Detection Limits (MDL and IDL)*.

The IDL is defined as three times the average of the standard deviation of seven replicate analyses of the IDL solution performed over three non-consecutive days. The IDL may be elevated above the background noise (blank levels). The current IDL associated with this procedure is given in the Equipment Limit Group (ELG) in LIMS.

12.3 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP QA06: *Training Procedures*.

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The LCSs must be from a second source than that used to prepare the calibration standards. The IDOC must be documented on the IDOC Form shown in SOP QA06 with documentation routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.4 Training Requirements

All training must be performed and documented in accordance with SOP QA06: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.).

Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.
- Acidic sample digestions – Neutralize before disposal into drain/sewer system.

15.0 References / Cross-References

- SOP AN10: *Balance Calibration and Use*
- SOP AN30: *Pipette and Volumetric Container Calibration Verification*
- SOP AN41: *Reagent and Standard Materials Traceability*
- SOP QA02: *Data Review and Reporting*
- SOP QA05: *Preventive and Corrective Action Procedures*
- SOP QA06: *Training Procedures*
- SOP QA07: *Determination of Detection Limits (MDLs and IDLs)*
- SOP QA15: *Homogenization, Compositing, and Segregation of Samples*
- SOP QA16: *Evaluation of Calibration Curves*
- SOP QA17: *Analytical Batching and Evaluation of QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Methods for Chemical Analysis of Water and Waste*; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.
- *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986.
- *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of

Research and Development. Washington, DC.

16.0 Method Modifications

Wipe, waste, filter, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs and MDLs (converted for initial and final volumes, etc.), and soil QC limits to evaluate wipe, waste, filter, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. The soil blank matrix is used as the blank matrix for these matrices.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

- Attachment 1: Sample Collection, Preservation, and Holding Time Table
- Attachment 2: Preventative Maintenance and Troubleshooting
- Attachment 3: SOP Summary
- Attachment 4: QC Summary
- Attachment 5: Standard Preparation Tables
- Attachment 6: Linear Range Determination
- Attachment 7: Element Wavelengths

Attachment 1: Sample Collection, Preservation, and Holding Time Table

Listed below are the holding times and preservation requirements:

Matrix	Routine Sample Container	Minimum Sample Size	Preservation¹	Holding Time²
Water	250-mL or 500-mL plastic	50mL	Nitric Acid	6 months
Soil	8-oz plastic	10g	N/A	6 months
Tissue	Plastic, various sizes	1.0g	N/A	6 months
Leachate	250-mL plastic	50mL	Nitric Acid	6 months
Filter		1filter	N/A	6 months
Wipe	40-mL VOA vial	1wipe	N/A	6 months
Waste	500-mL plastic	10g	N/A	6 months

¹Samples requiring dissolved metals must be filtered prior to preservation.

²Inclusive of digestion and analysis.

Attachment 2: Preventative Maintenance and Troubleshooting

Preventive Maintenance

Refer to the instrument manufacturer's guides for trouble-shooting items.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
ICAP Service Schedule								
Pump Tubing	X							Change daily.
Nebulizer							X	Clean as needed.
Filters			X					Inspect monthly. Clean or replace as needed.
Chiller Water Filter					X			Clean or replace every six months
Injector Tip		X						Inspect weekly, clean or replace as needed
Tubing Connectors		X						Replace as needed

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Attachment 3: SOP Summary

Sample Preparation Summary

Prior to analysis by ICP, the sample must be digested using the sample preparation method appropriate to the matrix. Samples should be prepared according to the appropriate matrix-specific SOP.

Matrix	SOP #
Aqueous samples	ME50
Leachate samples	ME50
Soil samples	ME51
Waste (oil) samples	ME51
Tissue Samples	ME51
Wipe Samples	ME51
Filter Samples	ME51

Sample Analysis Summary

Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

Analytical Sequence

Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Instrument Warm-up Profile	
Initial Calibration	
High Calibration Standard	Re-analyzed as a sample
ICV	Second Source
ICB	
Reporting Limit Check Standard	
ICP Interference Check Solution A (ICSA)	
ICP Interference Check Solution AB (ICSAB)	
CCV	10-injection clock begins with injection of the CCV
CCB	
Samples & Batch QC Items	Up to 10 injections, including QC.
CCV	10-injection clock begins with injection of the CCV
CCB	
Samples & Batch QC Items	Up to 10 injections, including QC.
CCV	10-injection clock begins with injection of the CCV
CCB	
Samples & Batch QC Items	Up to 10 injections, including QC.
CCV	10-injection clock begins with injection of the CCV
CCB	

Note: The analytical sequence must end with the analysis of the detection limit check standard, ICSA, ICSAB, CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

Attachment 4: QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 std. and 1 blank	
Initial Calibration: Multi-point-minimum 3 stds and 1 blank	Daily	Correlation >0.995	Recalibrate
Highest Standard	Immediately after every calibration	Recoveries within $\pm 5\%$ of expected values	New initial calibration
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846: within $\pm 10\%$ 200.7: within $\pm 5\%$, $\leq 3\%$ RSD	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within $\pm 10\%$ of the true value	Terminate the analysis, fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	6010B: The absolute value of the ICB/CCB must be less than 3x the IDL or less than the RL/CRDL, whichever is smaller. 200.7: Absolute value of the calibration blank must be less than the RL/CRDL	Terminate the analysis, correct problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning and end of an analysis run	6010B & 200.7: Determined values must be within $\pm 20\%$ of the true values. Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Lab Control Sample	One per batch of twenty samples or less	6010B: MLG 200.7: 85-115%	Redigest and reanalyze batch
Method Blank	One per batch of twenty samples or less	6010B: $ \text{result} < \text{RL}$ or result $< 5\%$ of the analyte level in the sample	Redigest and reanalyze batch

QC Item	Frequency	Criteria	Corrective Action
MS/MSD or MS/SD	One set per batch of twenty samples or less	200.7: result <RL or result <10% of the analyte level in the sample	Flag and report data
Serial Dilution (1/5 Dilution)	One per batch of twenty samples or less	MLG 75-125% (200.7) <10%RPD (6010, 200.7) See Section 9.2.9	
Post Digestion Spike	One per batch of twenty samples or less	See Section 9.2.10	
Reporting Limit Check Solution	At the beginning and end of an analysis run	Recovery +/-50% of the true concentration (if the instrument is not calibrated at or below the RL).	Stop the analysis, fix the problem and reanalyze the affected samples.
%RSD (CV) of Multiple Exposures	Evaluate for all Calibration, QC, and samples	Conc. >= RL Warning: <=20% Acceptance <=30%	-if CV>20 but <=30, review data for possible interferences; -if interference present, reanalyze digest -if no interference present, report average -if CV>=30%, reanalyze digest, report the result that has the lower precision value or dilute the digestion and reanalyze
Linear Range of ICP	Determined at least annually in accordance with Attachment 6	Conc. < RL % difference </= 5%	-use professional judgement -reanalyze at a lower concentration
Interlelement correction factors (IEC)	Determined at least annually Verified at the beginning and end of an analysis run	See ICSA, ICSAB criteria	See ICSA, ICSAB corrective action

Attachment 5: Standard Preparation

Note: All standards must be stored at room temperature and have an expiration date of 6 months from date prepared.

Continuing Calibration Verification (CCV)
 Final Volume (mL) 2000
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(single element stocks)</i>				
Ag	1000	CPI	1.0	0.5
Al	10000	SPEX	1.0	5.0
B	1000	CPI	10.0	5.0
Fe	10000	CPI	1.0	5.0
Sr	1000	ABSOLUTE	5.0	2.5
Ti	1000	CPI	1.0	0.5
Na	10000	CPI	0.5	2.5
<i>(multi element mixes)</i>				
CAL STD 2		CPI		
As	50		20	0.5
Mo	50			0.5
Pb	50			0.5
Sb	50			0.5
Se	500			5.0
Tl	500			5.0
CAL STD 3		CPI		
Ba	500		20	5.00
Be	50			0.50
Cd	50			0.50
Co	50			0.50
Cr	500			5.00
Cu	500			5.00
Mn	500			5.00
Ni	250			2.50
Zn	250			2.50
CAL STD 5		CPI		
Ca	500		20	5.00
K	1000			10.00
Mg	500			5.00
Na	500			5.00
Sn	500			5.00
V	500			5.00

Internal Standard (ISTD)

Final Volume (mL) 10000
Solvent 30% HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Y	10000	CPI	7.0	7.0

Purchased Standards	Concentration (%)	Vendor *	Volume Used (g)	Final Concentration (mg/L)
Lithium Carbonate (powder)	18.78%	Mallinckrodt	20	375.6

MDL & IDL Intermediate
Final Volume
 (mL) 100
Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(single element stocks)</i>				
Ag	1000	CPI	0.04	0.4
Al	10000	SPEX	0.10	10.0
As	1000	CPI	0.20	2.0
B	1000	CPI	0.40	4.0
Ba	1000	CPI	0.02	0.2
Be	1000	CPI	0.0050	0.1
Ca	10000	SPEX	0.10	10.0
Cd	1000	CPI	0.04	0.4
Co	1000	CPI	0.10	1.0
Cr	1000	CPI	0.10	1.0
Cu	1000	CPI	0.10	1.0
Fe	10000	CPI	0.10	10.0
K	10000	CPI	0.20	20.0
Mg	10000	SPEX	0.10	10.0
Mn	1000	CPI	0.02	0.2
Mo	1000	CPI	0.10	1.0
Na	10000	CPI	2.04	204.0
Ni	1000	CPI	0.10	1.0
Pb	1000	CPI	0.10	1.0
Sb	1000	CPI	0.20	2.0
Se	1000	CPI	0.20	2.0
Sn	1000	CPI	0.20	2.0
Sr	1000	CPI	0.04	0.4
Ti	1000	CPI	0.05	0.5
Tl	1000	CPI	0.20	2.0
V	1000	CPI	0.06	0.6
Zn	1000	CPI	0.10	1.0

CLP SPIKE 1 - Post Spike, LCS/MS Spike
Final Volume
 (mL) 100
Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(multi element mixes)</i>				
CRDL STD		SPEX		
Ag	20		10.0	2.00
As	20			2.00
Be	10			1.00
Cd	10			1.00
Co	100			10.00
Cr	20			2.00
Cu	50			5.00
Mn	30			3.00
Ni	80			8.00
Pb	6			0.60
Sb	120			12.00
Se	10			1.00
Tl	20			2.00
V	100			10.00
Zn	40			4.00

SPIKE II - Post Spike, LCS/MS Spike

Final Volume (mL) 100

Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(single element stocks)</i>				
B	1000	cpi	10	100
Ca	10000	spex	5.0	500
K	10000	cpi	5.0	500
Mg	10000	spex	5.0	500
Mo	1000	cpi	5.0	50
Na	10000	cpi	5.0	500
Sn	1000	cpi	10	100
Sr	1000	cpi	5.0	50
Ti	1000	cpi	10	100

RL ICP INTERMEDIATE

Final Volume (mL) 100
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(single element stocks)</i>				
Ag	1000	cpi	0.1	1.0
Al	10000	spex	0.2	20.0
As	1000	cpi	0.1	1.0
B	1000	cpi	0.5	5.0
Ba	1000	cpi	0.1	1.0
Be	1000	cpi	0.04	0.4
Ca	10000	spex	0.5	50.0
Cd	1000	cpi	0.05	0.5
Co	1000	cpi	0.1	1.0
Cr	1000	cpi	0.1	1.0
Cu	1000	cpi	0.2	2.0
Fe	10000	cpi	0.05	5.0
K	10000	cpi	1.0	100.0
Mg	10000	spex	0.5	50.0
Mn	1000	cpi	0.1	1.0
Mo	1000	cpi	0.1	1.0
Na	10000	cpi	0.5	50.0
Ni	1000	cpi	0.4	4.0
Pb	1000	cpi	0.05	0.5
Sb	1000	cpi	0.2	2.0
Se	1000	cpi	0.1	1.0
Sn	1000	cpi	0.5	5.0
Sr	1000	cpi	0.1	1.0
Ti	1000	cpi	0.1	1.0
Tl	1000	cpi	0.25	2.5
V	1000	cpi	0.1	1.0
Zn	1000	cpi	0.2	2.0

RL ICP Working
 Final Volume (mL) 100
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(from RL ICP Intermediate)</i>				
Ag	1.0	cpi	1.0	0.010
Al	20	spex		0.20
As	1.0	cpi		0.010
B	5.0	cpi		0.050
Ba	1.0	cpi		0.010
Be	0.4	cpi		0.0040
Ca	50	spex		0.50
Cd	0.5	cpi		0.0050
Co	1.0	cpi		0.010
Cr	1.0	cpi		0.010
Cu	2.0	cpi		0.020
Fe	5.0	cpi		0.050
K	100	cpi		1.0
Mg	50	spex		0.50
Mn	1.0	cpi		0.010
Mo	1.0	cpi		0.010
Na	50	cpi		0.50
Ni	4.0	cpi		0.040
Pb	0.50	cpi		0.0050
Sb	2.0	cpi		0.020
Se	1.0	cpi		0.010
Sn	5.0	cpi		0.050
Sr	1.0	cpi		0.010
Ti	1.0	cpi		0.010
Tl	2.5	cpi		0.025
V	1.0	cpi		0.010
Zn	2.0	cpi		0.020

RL LOW INTERMEDIATE

Final Volume (mL) 100
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(single element stocks)</i>				
Ag	1000	cpi	0.1	1.0
Al	10000	spex	0.2	20.0
As	1000	cpi	0.1	1.0
Ba	1000	cpi	0.1	1.0
Be	1000	cpi	0.02	0.2
Ca	10000	spex	0.2	20.0
Cd	1000	cpi	0.02	0.2
Co	1000	cpi	0.1	1.0
Cr	1000	cpi	0.06	0.6
Cu	1000	cpi	0.1	1.0
K	10000	cpi	0.2	20.0
Mg	10000	spex	0.2	20.0
Mn	1000	cpi	0.1	1.0
Mo	1000	cpi	0.1	1.0
Ni	1000	cpi	0.1	1.0
Pb	1000	cpi	0.06	0.6
Sb	1000	cpi	0.12	1.2
Se	1000	cpi	0.1	1.0
Zn	1000	cpi	0.2	2.0

RL LOW INTERMEDIATE

Final Volume (mL) 100
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(single element stocks)</i>				
Ag	1	cpi	0.50	0.0050
Al	20	spex		0.10
As	1	cpi		0.0050
Ba	1	cpi		0.0050
Be	0.2	cpi		0.0010
Ca	20	spex		0.10
Cd	0.2	cpi		0.0010
Co	1	cpi		0.0050
Cr	0.6	cpi		0.0030
Cu	1	cpi		0.0050
K	20	cpi		0.10
Mg	20	spex		0.10
Mn	1	cpi		0.0050
Mo	1	cpi		0.0050
Ni	1	cpi		0.0050
Pb	0.6	cpi		0.0030
Sb	1.2	cpi		0.0060
Se	1	cpi		0.0050
Zn	2.0	cpi		0.010

ICV
 Final Volume (mL) 500
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(single element stocks)</i>				
K	1000	Element	5.0	10.0
Na	1000	Element	4.5	9.0
Sn	1000	Element	0.5	1.0
Sr	1000	Element	0.5	1.0
<i>(multi element stocks)</i>				
QC19		Element		
As	100			1.0
Be	100			1.0
Ca	100			1.0
Cd	100			1.0
Co	100			1.0
Cr	100			1.0
Cu	100			1.0
Fe	100			1.0
Mg	100			1.0
Mn	100			1.0
Mo	100			1.0
Ni	100			1.0
Pb	100			1.0
Sb	100			1.0
Se	100			1.0
Ti	100			1.0
V	100			1.0
Zn	100		5.0	1.0
QC7		Element		
Ag	100			1.0
Al	100			1.0
B	100			1.0
Ba	100			1.0
K	1000			10.0
Na	100			1.0
Si	50		5.0	0.5

ICSA
 Final Volume (mL) 1000
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(multi element mixes)</i>				
Interferents A		SPEX		
Al	5000		100	500.0
Ca	5000			500.0
Mg	5000			500.0
Fe	2000			200.0

ICSAB
 Final Volume (mL) 1000
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(multi element mixes)</i>				
Interferents A		SPEX		
Al	5000		100	500.0
Ca	5000			500.0
Mg	5000			500.0
Fe	2000			200.0
Altany C		Spectro Pure		
Ag	20		10	0.20
As	10			0.10
Ba	50			0.50
Be	50			0.50
Cd	100			1.00
Co	50			0.50
Cr	50			0.50
Cu	50			0.50
Mn	50			0.50
Ni	100			1.00
Pb	5			0.05
Sb	60			0.60
Se	5			0.05
Tl	10			0.10
V	50			0.50
Zn	100		1.00	
Trace AB				
Mo	100		10	1.0
Sn	100			1.0

Trace AB
Final Volume (mL) 100
Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(single element stocks)</i>				
Mo	1000	cpi	10	100.00
Sn	1000	cpi	10	100.00

High Std
 Final Volume (mL) 1000
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(single element stocks)</i>				
Ag	1000	CPI	1.0	1.0
Al	10000	SPEX	1.0	10.0
B	1000	CPI	10.0	10.0
Fe	10000	CPI	1.0	10.0
Sr	1000	ABSOLUTE	5.0	5.0
Ti	1000	CPI	1.0	1.0
<i>(multi element mixes)</i>				
CAL STD 2		CPI		
As	50		20	1.0
Mo	50			1.0
Pb	50			1.0
Sb	50			1.0
Se	500			10.0
Tl	500			10.0
CAL STD 3		CPI		
Ba	500		20	10.00
Be	50			1.00
Cd	50			1.00
Co	50			1.00
Cr	500			10.00
Cu	500			10.00
Mn	500			10.00
Ni	250			5.00
Zn	250			5.00
CAL STD 5		CPI		
Ca	500		20	10.00
K	1000			20.00
Mg	500			10.00
Na	500			10.00
Sn	500			10.00
V	500			10.00

CRI
 Final Volume (mL) 500
 Solvent 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<i>(multi element mixes)</i>				
CRDL STD		SPEX		
Ag	20		0.50	0.0200
As	20			0.0200
Be	10			0.0100
Cd	10			0.0100
Co	100			0.1000
Cr	20			0.0200
Cu	50			0.0500
Mn	30			0.0300
Ni	80			0.0800
Pb	6			0.0060
Sb	120			0.1200
Se	10			0.0100
Tl	20			0.0200
V	100			0.1000
Zn	40			0.0400

Attachment 6: Linear Range Determination

The linear range must be determined at a minimum of once per year for routine work. The linear range must be determined at a minimum of quarterly for the CLP and DOD QSM programs.

Profile and calibrate the ICP as described in Section 9.2.1.

Prepare individual standards at concentrations that are expected to define the linear range of the instrument. The calibration standards and the linear range standards must be matrix matched; that is, they must have the same percentage of hydrochloric and nitric acids.

Analyze the standards following the analytical sequence described in Section 10.3.5. Verify the calibration after every 10 analyses.

Compare the concentration of the linear range standard with its true concentration as follows:

$$\text{PercentDifference} = \left| \frac{C_{\text{cal}} - C_{\text{true}}}{C_{\text{true}}} \right| \otimes 100$$

Where:

C_{cal} = concentration determined from analysis

C_{true} = true concentration of the standard

If the percent difference is less than or equal to 5%, the linear range is confirmed at that concentration. If the percent difference is greater than 5%, repeat the analysis with a lower concentration.

The linear range may be extended by analyzing higher standards and evaluating the results against the 5% difference criterion. The linear range of the ICP for an analyte is the highest standard of that analyte that meets this criterion.

If any calibration regression fit, other than linear, is utilized for the calibration of the ICP (i.e., Curvilinear or Full Fit), the upper limit of the linear range is the concentration of the High Standard.

Attachment 7: Element Wavelengths

Element	Wavelength 1 (nm)	Wavelength 2 (nm)	Wavelength 3 (nm)
Aluminum (Al)	308.215		
Antimony (Sb)	206.838		
Arsenic (As)	189.042	193.696	
Barium (Ba)	493.409		
Beryllium (Be)	313.042		
Boron (B)	249.678		
Cadmium (Cd)	226.502	228.802	
Calcium (Ca)	317.933	315.887	
Chromium (Cr)	267.716		
Cobalt (Co)	228.616		
Copper (Cu)	324.754		
Iron (Fe)	259.940	271.441	
Lead (Pb)	220.353		
Magnesium (Mg)	279.079		
Manganese (Mn)	257.610		
Molybdenum (Mo)	202.030		
Nickel (Ni)	231.604		
Potassium (K)	766.491		
Selenium (Se)	196.026		
Silver (Ag)	328.068		
Sodium (Na)	588.995	330.231	
Strontium (Sr)	421.552	421.552	
Thallium (Tl)	189.042	190.801	377.572
Tin (Sn)	189.989		
Titanium (W)	334.941		
Vanadium (V)	292.402		
Zinc (Zn)	213.856	206.200+	

18.0 Revision History

Summary of Changes from Previous Revision:

- Updated to new TestAmerica SOP template. Significant formatting and content changes made.
- Revised the ICB/CCB criteria for SW-846 Method 6010.
- Updated EPA Method 200.7 requirements to evaluate NPDES samples using the more stringent Drinking Water criteria.
- Add standards preparation information.