REMEDIAL ACTION CONTRACT 2 FOR REMEDIAL, ENFORCEMENT, OVERSIGHT, AND NON-TIME-CRITICAL REMOVAL ACTIVITIES IN REGION 5

SAMPLING AND ANALYSIS PLAN FOR TECHNICAL ASSISTANCE FOR CBS MULTI-SITES MONROE COUNTY, INDIANA

Prepared for
U.S. Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, Illinois 60604-3590

Work Assignment No.	:	313-TATA-05ZZ
EPA Region	:	5
Date Prepared	:	October 11, 2018
Contract No.	:	EP-S5-06-02
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SAMPLING AND ANALYSIS PLAN FOR TECHNICAL ASSISTANCE FOR CBS MULTI-SITES MONROE COUNTY, INDIANA

SulTRAC has prepared this sampling and analysis plan (SAP) to meet the requirements specified in the U.S. Environmental Protection Agency (EPA) statement of work (SOW) Revision No. 2.1 dated January 2014, for the CBS Multi-Sites (CBS sites) in Bloomington, Monroe County, Indiana. SulTRAC, a joint venture between Tetra Tech and SCST Engineering, has prepared this SAP under the Remedial Action Contract 2 for Region 5, Contract No. EP-S5-06-02, Work Assignment No. 313-TATA-05ZZ. This SAP describes sampling and analysis activities to be conducted by SulTRAC during whole fish and fish tissue sampling for areas associated with the CBS sites including Neal's Landfill and Bennett's Dump. This SAP describes sampling activities to be conducted by SulTRAC to ensure that the sites are adequately monitored since the responsible party, CBS, has limited monitoring requirements pursuant to the Consent Decree and Consent Decree Amendment.

SulTRAC's SAP consists of the field sampling plan (FSP) (Appendix A) and the previously approved quality assurance project plan (QAPP) (Appendix B), which remains unchanged from the prior submittal. Field sampling activities and procedures presented in this revised FSP are limited to the scope of the fish tissue sampling proposed for this field sampling event. The laboratory (TestAmerica) and test methods are unchanged from prior submittals. Quality assurance (QA) and quality control (QC) protocols associated with the sampling and analysis activities are presented in the QAPP.

REMEDIAL ACTION CONTRACT 2 FOR REMEDIAL, ENFORCEMENT, OVERSIGHT, AND NON-TIME-CRITICAL REMOVAL ACTIVITIES IN REGION 5

APPENDIX A

FIELD SAMPLING PLAN FOR TECHNICAL ASSISTANCE FOR CBS MULTI-SITES MONROE COUNTY, INDIANA

Prepared for
U.S. Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, Illinois 60604-3590

Work Assignment No.	:	313-TATA-05ZZ
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ABBREVIATIONS, ACRONYMS, AND SYMBOLS

AECOM	AECOM Technical Services, Inc.
^{o}C	Degrees Celsius
CBS	CBS Corporation
CBS sites	CBS Multi-Sites
COC	Chain of custody
DOT	U.S. Department of Transportation
EPA	U.S. Environmental Protection Agency
FSP	Field sampling plan
GPS	Global Positioning System
HASP	Health and safety plan
ICS	Illinois Central Spring
IDEM	Indiana Department of Environmental Management
IDW	Investigation-derived waste
mg/kg	Milligrams per kilogram
mL	Milliliter
MS	Matrix spike
MSD	Matrix spike duplicate
N/A	Not applicable
OU	Operable Unit
PCB	Polychlorinated biphenyl
ppm	Part per million
QA	Quality assurance
QAPP	Quality Assurance Project Plan
QC	Quality control
RAC	Remedial Action Contract
SAP	Sampling and analysis plan
SOP	Standard operating procedure
SOW	Statement of work
Viacom	Viacom, Inc.
WA	Work assignment
Westinghouse	Westinghouse Electric Corporation

1.0 INTRODUCTION

SulTRAC has prepared this field sampling plan (FSP) as part of the sampling and analysis plan (SAP) for the CBS Corporation (CBS) Multi-Sites (CBS sites) in Bloomington, Monroe County, Indiana, under the U.S. Environmental Protection Agency (EPA) Remedial Action Contract (RAC) 2 for Region 5, Contract No. EP-S5-06-02, Work Assignment (WA) No. 313-TATA-05ZZ. The WA was revised to add fish sampling (whole/fillet), groundwater and spring (surface water) sampling, and sediment sampling within areas associated with CBS sites that include Lemon Lane Landfill, Neal's Landfill, and Bennett's Dump. This SAP is focused on collecting and analyzing fish tissue samples at three stream locations associated with Neal's Landfill and Bennett's Dump. Fish tissue sampling associated with the Lemon Lane Landfill is being conducted by CBS and is not addressed in this SAP.

The SAP consists of this FSP (Appendix A) and the Quality Assurance Project Plan (QAPP) (Appendix B), which are among the site-specific plans to be prepared under the WA in accordance with Subtask 1.2 of the EPA Statement of Work (SOW) (EPA 2013, 2014). This FSP describes sampling activities that SulTRAC will conduct to ensure that the CBS sites are adequately monitored since the responsible party, CBS, has limited monitoring requirements pursuant to the Consent Decree and Consent Decree Amendment.

The FSP consists of 12 sections, including this introduction (Section 1.0). Section 2.0 briefly describes and recounts the history of the CBS sites. Section 3.0 specifies project objectives. Section 4.0 describes proposed field sampling activities. Section 5.0 presents proposed field sampling procedures. Section 6.0 describes laboratory analytical methods. Section 7.0 presents decontamination procedures. Section 8.0 describes sample handling and analysis procedures. Section 9.0 presents the tentative schedule for field activities. Section 10.0 discusses disposal of investigation-derived waste (IDW). Section 11.0 specifies health and safety procedures. Section 12.0 describes quality assurance (QA) requirements. Sources referenced to prepare the FSP are listed after Section 12.0. Attachment A contains figures showing the CBS site locations in the area of Bloomington, Indiana. Attachment B presents SulTRAC's standard operating procedures (SOP). Attachment C presents SulTRAC's analytical subcontractor's (TestAmerica, Inc.) SOPs.

2.0 SITE DESCRIPTION AND HISTORY

The CBS sites were former industrial waste disposal sites for Westinghouse Electric Corporation (Westinghouse) (later known as CBS; Viacom, Inc. [Viacom]; and now CBS again). Among the industrial wastes were electrical capacitors, some of which contained polychlorinated biphenyls (PCB). In August 1985, EPA, the State of Indiana, Monroe County, the City of Bloomington, and Westinghouse entered into a consent decree. Under the Consent Decree, Westinghouse was to remediate six PCB-contaminated sites in the Bloomington area, which included Bennett's Dump, Neal's Landfill, and Lemon Lane Landfill. General site locations of Bennett's Dump, Neal's Landfill, and Lemon Lane Landfill are shown on Figure 1 (see Attachment A). Detailed sampling location maps of Bennett's Dump and Neal's Landfill are also provided in Attachment A as Figures 2 and 3, respectively. Details regarding the site history and site descriptions for Bennett's Dump, Neal's Landfill, and Lemon Lane Landfill are below.

BENNETT'S DUMP

The Bennett's Dump site, in Bloomington, Indiana, formerly was used as a dump. This site is part of a Consent Decree Amendment which was entered in 2009. CBS is the only responsible party. The site is located in the middle of a former quarry, and currently new industrial/commercial and residential development is underway surrounding the site. Capacitors containing PCBs and other PCB containing waste material were disposed of at the dump site. The remedial action for the source control operable unit (OU) 1 was completed in 1999 and the September 2006 Record of Decision and 2009 Consent Decree Amendment describe in detail the approach to be used in the implementation of the groundwater and sediment operable units. Two phases of work were planned and Phase 1 was completed in 2010. Phase 1 included the implementation of a passive quarry drain for the Wedge Quarry Complex and a subsequent investigation to determine the effects of the passive quarry drain on groundwater flow and PCB concentrations in groundwater. Phase II, which included the installation of a treatment system, was completed in 2014.

NEAL'S LANDFILL

Neal's Landfill is located in Bloomington, Indiana and is a former municipal landfill which accepted both municipal and industrial waste. This site is part of a Consent Decree Amendment which was entered in 2009. CBS is the only responsible party. Industrial waste included capacitors containing PCBs and other PCB contaminated material associated with the production of capacitors. The remedial action for the source control operable unit was completed in 1999 and the groundwater and sediment operable units were described in the September 2007 Record of Decision and 2009 Consent Decree Amendment. The remedial action for groundwater and sediment consisted of installing an additional collection system to capture water from South Spring, North Spring, North Spring Bypass and the Overflow Springs. The additional collection system works in conjunction with the current collection systems for South Spring, North Spring and Overflow Springs. In addition to the new collection system, PCB contaminated sediment was removed and disposed of in an off-site landfill. Sediment in-stream and within the banks was remediated to 1 ppm PCB on average and floodplain sediment has been remediated to 5 parts per million (ppm) on average. A remedy reopener is also part of the monitoring plan based on the cleanup of fish tissue. If the PCB levels in fish do not decrease, additional remedial measures may be necessary. The site construction is complete, but additional sampling by EPA will assist in evaluating the requirements to determine if the water treatment plant will require expansion (to capture all storm water flows that bypass treatment above 450 gallons per minute at the site).

LEMON LANE LANDFILL

The Lemon Lane Landfill is located in Bloomington, Indiana and is a former municipal landfill which accepted both municipal and industrial waste. This site is part of a Consent Decree Amendment which was entered in 2009. CBS is the only responsible party. Industrial waste included capacitors containing PCBs and other PCB contaminated material associated with the production of capacitors. The remedial action for the source control operable unit was completed in 2000 and a 1,000 gallon per minute water treatment plant with 1.2 million gallons of storage capacity was also completed in 2000. The Record of Decision for OU2, signed in September 2006 and also described in the 2009 Consent Decree Amendment, called for the installation of a new effluent line to carry treated effluent, sediment/soils cleanup at the swallow hole/Illinois Central Spring (ICS) emergence/quarry springs area, and installation of a storm water storage tank treatment system. This work is complete and phase 2 will consist of evaluating both Quarry B Spring and Rinker Spring to determine if the springs need to be treated at the existing plant. The site construction is complete, but CBS continues to evaluate the Quarry and Rinker Spring system to determine if the small springs will require treatment. CBS is not required to evaluate fish tissue post remediation.

3.0 PROJECT OBJECTIVES

The purpose of this FSP is to conduct fish sampling at areas associated with two of the CBS sites including Neal’s Landfill and Bennett’s Dump. Fish tissue sampling at the Lemon Lane Landfill is being conducted by CBS and is not included in this FSP. Sampling and evaluation of data are intended to evaluate current PCB concentrations in fish tissue and to compare results with prior sampling results using trend analysis. The fish sample data will be reviewed, and a statistical comparison of these data to each site’s historical fish sample data will occur to determine if any statistically significant trends in fish tissue PCB concentrations at these sites are evident as a result of CBS remedial activities. For Neal’s

Landfill only, the Consent Decree Amendment entered in 2009 includes a clause for EPA to modify the remedy for groundwater, surface water, and sediment contamination if EPA concludes that the remedy has not achieved and will not achieve target concentrations in fish within 10 years. Target mean PCB concentrations in whole fish to protect ecological receptors are 2.3 milligrams per kilogram (mg/kg) for "Location B – lower reach of Conard's Branch above Vernal Pike Bridge," and 0.9 mg/kg for "Location D – Richland Creek at the Vernal Pike Bridge." These are the Indiana Department of Environmental Management's (IDEM) historical fish sampling locations. IDEM Location B corresponds to location NL-1, and IDEM Location D corresponds to NL-2 in this FSP. The target mean PCB concentration in whole fish to protect human receptors is 0.2 mg/kg for "Location F – Richland creek at State Route 43 Bridge in Owen County." IDEM Location F corresponds to location NL-3 in this FSP.

4.0 FIELD SAMPLING ACTIVITIES

Proposed field investigation activities and the rationale for conducting these activities appear in this section. A detailed discussion of sample collection procedures appears in Section 5.0. SulTRAC will conduct one fish sampling event in the following creeks associated with the CBS sites: Conard's Branch (Neal's Landfill), and Stout's Creek (Bennett's Dump). SulTRAC assumes it will collect 40 fish samples plus field duplicates and quality control (QC) samples during the sampling event for PCB analysis by a subcontractor laboratory (four samples plus QC samples to be analyzed by the PCB congener method, and 36 samples plus QC samples to be analyzed by the PCB Aroclor method). All fish samples also will also be analyzed for percent lipids. Fish samples will be collected at locations most proximate to each landfill including one location associated with Neil's Landfill (Conard's Branch at Vernal Pike) and two locations downstream of Bennett's Dump (Stout's Creek at Hunter Valley Road and Stout's Creek at Acuff Road). The fish sampling locations at each site are shown on Figures 2 and 3 in Attachment 1. Tables 1 and 2 summarize each activity and its purpose.

Table 1
Summary of Field Investigation Activities

Investigation Area	Objective	Proposed Sampling Activities
Conard's Branch – Neal's Landfill[a]	Evaluate effectiveness of remediation	• Collect 8 fish samples plus QC samples • Analyze 4 fish samples plus QC samples for PCB Congeners • Analyze 4 fish samples plus QC samples for PCB Aroclors • Analyze all samples for percent lipids
Stout's Creek – Bennett's Dump[a]	Evaluate effectiveness of remediation	• Collect 32 fish samples plus QC samples • Analyze 32 fish samples plus QC samples for PCB Aroclors • Analyze all samples for percent lipids

Notes:

[a] The number of samples listed is an estimate for sampling events associated with both Neal's Landfill and Bennett's Dump, and may change depending on conditions experienced in the field. The number of samples will include investigative and QC samples. SulTRAC will collect field duplicate and MS/MSD samples for QC analysis. The fish samples will be analyzed as either whole bodies or fillets.

PCB	Polychlorinated biphenyl
MS/MSD	Matrix spike/matrix spike duplicate
QC	Quality control

Table 2
Sample Collection Summary

Matrix	Laboratory Parameters	Number of Samples[a]
Neal's Landfill		
Fish Tissue	PCB Congeners EPA Method 1668B	4
Fish Tissue	PCB Aroclors EPA SW-846 Method 8082	4
Fish Tissue	Percent Lipids	8
Bennett's Dump		
Fish Tissue	PCB Congeners EPA Method 1668B	0
Fish Tissue	PCB Aroclors EPA SW-846 Method 8082	32
Fish Tissue	Percent Lipids	32
Quality Control-Field Duplicate Samples		
Fish Tissue	PCB Congeners EPA Method 1668B	1
Fish Tissue	PCB Aroclors EPA SW-846 Method 8082	4
Fish Tissue	Percent Lipids	4
Quality Control-MS/MSD Samples[b]		
Fish Tissue	PCB Congeners EPA Method 1668B	1
Fish Tissue	PCB Aroclors EPA SW-846 Method 8082	2
Fish Tissue	Percent Lipids	2

Notes:

[a] The number of samples listed is an estimate for sampling events and may change depending on conditions experienced in the field. SulTRAC will collect field duplicate samples at a rate of 1 per every 10 samples submitted collectively and MS/MSD samples for QC analysis will be submitted at a rate of 1 per 20 samples or 1 per batch (if less than 20 samples) per analytical method.

[b] MS/MSD samples consist of extra sample volume and 1 MS/MSD sample pair counts as 2 sample analyses.

PCB	Polychlorinated biphenyl
MS/MSD	Matrix spike/matrix spike duplicate
QC	Quality control

5.0 FIELD SAMPLING PROCEDURES

This section describes the procedures to be used to collect the types of samples described in Section 4.0. Specifically, this section details the procedures and methods that will be used to collect fish tissue. Additional details regarding procedures for sample collection, analyses, and data management appear in SulTRAC's project QAPP (SulTRAC 2014b). All SulTRAC field activities will be conducted in accordance with the procedures described in this section of the FSP.

5.1 Fish Tissue Sampling

SulTRAC will conduct one fish sampling event in two of the creeks associated with the CBS sites. SulTRAC anticipates assistance from SulTRAC's local subcontractor, AECOM Technical Services, Inc. (AECOM). AECOM will provide personnel to support SulTRAC personnel. SulTRAC will rent electrofishing equipment to be utilized to conduct sampling at the shallower locations near the sites; it is anticipated that all sampling locations can be accessed on foot, using waterproof waders.

SulTRAC will use a backpack-mounted electroshocking unit. Fish samples will be collected at three locations from Conard's Branch and Stout's Creek (Figures 2 and 3). All personnel involved in electroshocking will be familiar with standard electrofishing operational and safety procedures. Fish sampling and sample preparation procedures will be consistent with the IDEM *Standard Operating Procedures for Fish Collections*, "Section 4: Use of Seines, Electrofishers, and Sample Processing" (IDEM 1992) (see Attachment D). Table 3 summarizes the targeted fish species and weights for each location. Targeted species were selected to represent three feeding guilds: (1) benthic, (2) omnivorous, and (3) pelagic. To obtain numbers and sizes of target species at each sampling site, the sampling team may move upstream or downstream a few hundred feet from the specified location. If the target species are not found after several passes at the location and upstream or downstream in the creeks, SulTRAC may return to try again the next day or before completion of the sampling event.

Fish collected for analysis must be shipped to the laboratory in a manner preventing decomposition or contamination. Packaged fish are to be placed immediately on wet ice and chilled to 4 degrees Celsius (°C) for transport back to the laboratory. Samples shipped on wet ice should reach the processing laboratory within 24 hours after collection to allow sufficient time for processing. Samples to be filleted should be processed no later than 48 hours after collection. If samples cannot be processed within this time frame, they should be frozen as whole fish, delivered to the laboratory as soon as possible, and stored at -20°C until they can be processed. Freezing samples should be avoided whenever possible because of the possibility that internal organs would rupture and contaminate fillet tissue. If fish are frozen, they should not be allowed to thaw during transport. Before they are processed, frozen fish samples should be only partially thawed before they are filleted. (Ice crystals should still be visible in the fillet tissue.)

Table 3
Target Fish Species Per Location

Creek	Sample locations	ID	Targets	Type of Sample	Length and Targeted Weight[a,b]	Feeding Guild
Conard's Branch	Conard's Branch at Vernal Pike bridge	B	Creek Chub	Whole body	Minimum 4.3 inches to Maximum 5.8 inches 15.3 to 35.7 grams	Omnivorous
Stout's Creek	South of N. Hunter Valley Road	BD-1	Creek Chub	Whole body	Minimum 5.4 inches to Maximum 6.5 inches 29.4 to 49.2 grams	Omnivorous
			Green Sunfish	Whole body	Minimum 4.2 inches Maximum 4.7 inches 23 to 32.6 grams	Top Predator
			Green Sunfish	Fillet	Minimum 3 inches to Maximum 5.7 inches 8.4 to 60.6 grams	Top Predator
			White Suckers	Whole body	Minimum 5.1 inches Maximum 7.4 inches 23.7 to 72.3 grams	Bottom Feeder
	North of Acuff Road	BD-2	Creek Chub or Stoneroller	Whole body	Minimum 4.5 inches to Maximum 6.8 inches 16.9 to 57.3 grams	Omnivorous
			Green Sunfish or Bluegill	Whole body	Minimum 3.7 inches to Maximum 4.9inches 16.1 to 37.3 grams	Top Predator
			Green Sunfish	Fillet	Minimum 145 inches to Maximum 145 inches 61.6 to 61.6 grams	Top Predator
			White Sucker	Whole body	Minimum 6 inches to Maximum 9.3 inches 37.8 to gr149 grams	Bottom Feeder

Notes:

a - Weights provided are from available records and are for whole fish collected.

b - Each fish sample should weigh at a minimum 35 grams. Creek Chubs and any other species that does not meet the minimum weight requirement will be composited to meet the weight requirement. Samples for duplicate analyses should be at least 50 grams as 10 grams would be lost by laboratory homogenization,but should target 70 grams if possible to provide a safety factor for adequate sample material. MS/MSD samples will be specified on the Chain of Custody and should weigh triple the required minimum sample mass (between 100 – 200 grams).

6.0 LABORATORY ANALYTICAL METHODS

Table 4 summarizes the laboratory methods used to analyze samples collected by SulTRAC during the site investigation. Samples to undergo chemical analyses for PCB Aroclors and PCB congeners will be sent to SulTRAC's subcontractor laboratory, TestAmerica, Inc. (TestAmerica) in Pittsburgh, Pennsylvania and Knoxville, Tennessee, respectively. In addition, TestAmerica's SOPs for determining the requested analytical parameters are presented in Attachment C of this FSP.

Table 4

Analytical Methods Summary

Parameter	Analytical Method
Fish Tissue	
PCB Aroclors	EPA SW-846 Method 8082A
PCB Congeners	EPA Method 1668B
Percent Lipids	According to TestAmerica SOP No. PT-OP-011, Rev. 5

Notes:

EPA	U.S. Environmental Protection Agency
PCB	Polychlorinated biphenyl
SOP	Standard operating procedure

7.0 DECONTAMINATION PROCEDURES

SulTRAC will implement decontamination procedures during fish sampling as outlined below.

7.1 Decontamination of Equipment Used for Fish Sampling

Non-disposable equipment used for fish sampling that comes into contact with fish tissue will be washed following the general sampling equipment decontamination procedures described in Section 2.5 of SOP 002. Specifically, per the guidelines established in SOP 002, stainless steel sampling equipment will be decontaminated using a three-tier process including an Alconox or Liquinox wash, a potable water rinse, and a distilled water rinse. SulTRAC does not anticipate need to use chemical solvents in decontaminating this type of equipment; however, solvents may be used as described in SOP 002 if field conditions indicate presence of gross contamination.

8.0 SAMPLE HANDLING AND ANALYSIS PROCEDURES

SulTRAC will collect fish tissue samples; prepare the samples for shipment; and complete all necessary paperwork such as COC and overnight shipping airbill labels. Sample containers, sample preservation, sample identification and documentation, sample COC, and sample packaging and shipping are discussed below. The SulTRAC QAPP (SulTRAC 2014b) provides detailed information regarding sample procedures that will be followed during the sampling investigation.

8.1 Sample Containers, Preservatives, and Holding Times

The samples will be preserved on ice and will be shipped to the appropriate laboratory on the day of collection via an overnight delivery service. Sample preservation methods and holding times are summarized in Table 5 below and in Worksheet No. 19 of the QAPP (Appendix B of the SAP).

Table 5

Sample Containers, Preservation Methods, and Holding times

Analytical Parameter	Matrix	No. of Containers Per Sample	Container Type	Preservation Method	Holding Time
PCB Congeners, Percent Lipids	Fish Tissue	One	Wrap each fish in extra-heavy-duty aluminum foil; place in waterproof plastic bag	Cool with ice (maximum shipping time 24 hours) OR Freeze with dry ice (maximum shipping time 48 hours)	Extract within 14 days (up to one year if frozen), and analyze within 40 days after extraction
PCB Aroclors, Percent Lipids	Fish Tissue	One	Wrap each fish in extra-heavy-duty aluminum foil, place in waterproof plastic bag	Cool with ice (maximum shipping time 24 hours) OR Freeze with dry ice (maximum shipping time 48 hours)	Extract within 14 days (up to one year if frozen), and analyze within 40 days after extraction

Notes:

mL	Milliliter
N/A	Not applicable
PCB	Polychlorinated biphenyl
°C	Degrees Celsius

8.2 Sample Identification

Each sample will be identified using an alphanumeric system (see Table 6 below) that identifies the project, general sampling location, and sample type. Field duplicate samples will be designated by "D." MS/MSD samples will be identified in the site logbooks and will be clearly designated on COC forms rather than by sample identification numbers.

Table 6
Generalized Sample Identification Scheme

Investigation Area	Sample Location	Matrix	Sequential Sample Number	Example Identification
Neal's Landfill – NL	B – Lower reach of Conard's Branch above Vernal Pike bridge	Fish Tissue Fillet – FTF Fish Whole Body – FWB	01	NL-1-FTF-01-XXXX* NL-1-FWB-01-XXXX*
Bennett's Dump – BD	1 – Hunter Valley Rd 2 – Acuff Rd	Fish Tissue Fillet – FTF Fish Whole Body – FWB	01	BD-1-FTF-01-XXXX* BD-1-FWB-01-XXXX*

Notes:

*	Month/Year format
FTF	Fish tissue fillet
FWB	Fish whole body

8.3 Sample Labels

Samples analyzed by the subcontracted laboratory will be identified using the nomenclature described in Table 7. Each label for these samples will be completed with the following information:

- Project number
- Sample collection date and time
- Preservative
- Sample collector's initials
- Analysis.

After it has been labeled, each sample will be preserved if and as required (see Table 5).

8.4 Sample Documentation

Sampling activities will be documented in a bound logbook using a ballpoint pen in accordance with SulTRAC field SOP No. 024 (see Attachment 1 to this FSP). The time of collection, identification number, sampling location, field observations (including measurements of pH and other field parameters), sampler's name, and analyses will be recorded in the logbook for each sample. Each page of the logbook will be dated, numbered, and signed by a SulTRAC staff member. Field data records will be maintained in accordance with Multi-Media Investigation Manual and Procedures (EPA 1992).

8.5 Sample Chain of Custody

All samples will be collected and handled according to proper COC procedures. When collecting samples for laboratory analysis, SulTRAC field personnel will complete the paperwork used for tracking samples, including the following items:

- COC forms
- Sample tags
- Custody seals.

SulTRAC will follow the procedures in the EPA Region 5 Central Regional Laboratory's *SARA/Superfund Sample Handling Manual* (EPA 1989) to complete the documentation listed above.

SulTRAC will appoint one of its field technical staff to serve as the sample custodian. Upon completion of all required documents, the sample custodian will sign and date the documents, and list the time of sample collection. The custodian will also confirm completeness of all descriptive information on COC forms, which will be included with each shipping container. The sample custodian will retain the pink copies of all COC forms for the project files.

8.6 Sample Packaging and Shipping

The following procedures will be implemented when collected samples are shipped:

- The cooler will be filled with sample bottles and sample bags, ice, and packing material. Sufficient packing material will be used to prevent sample containers from breaking during shipment. Enough ice will be added to maintain a sample temperature of 4 ± 2 degrees Celsius (°C).
- The COC forms will be placed inside a plastic bag, which will be sealed and taped to the inside of the cooler lid. The air bill, if required, will be completed before the samples are relinquished to the carrier. The laboratory will be notified if the sampler suspects that the sample contains any substance that would require laboratory personnel to take safety precautions.
- The cooler will be closed and taped shut with clear packaging tape around both ends. If the cooler has a drain, it will be taped shut both inside and outside of the cooler.
- A signed and dated custody seal will be placed on the front of each cooler. Wide clear tape will be placed over the seals to prevent accidental breakage.
- The COC record will be transported within the tape-sealed cooler. When the cooler is received at the analytical laboratory, laboratory personnel will open the cooler and sign the COC record to document transfer of samples.

All shipping containers will be labeled as required by DOT. After packaging, the samples will be shipped to the subcontractor laboratory.

9.0 TENTATIVE SCHEDULE FOR FIELD ACTIVITIES

The sampling event will occur in early November 2018.

10.0 DISPOSAL OF INVESTIGATION-DERIVED WASTE

Any generation of IDW by SulTRAC will occur during sampling to determine the nature and extent of contamination. All IDW requiring disposal will be containerized and disposed of at Neal's Landfill and/or Illinois Central Spring Water Treatment facilities.

11.0 HEALTH AND SAFETY PROCEDURES

All field activities will be conducted in accordance with SulTRAC's health and safety plan (HASP) (SulTRAC 2018), which is among the site-specific plans prepared for this WA. Prior to initiation of field activities, all SulTRAC field personnel will read and sign the HASP, indicating that they understand the plan and agree to operate in accordance with its requirements. A complete copy of the site-specific plans, including the HASP, will be maintained by the field sampling team at the site.

12.0 QUALITY ASSURANCE REQUIREMENTS

All sampling activities will be conducted in accordance with the QAPP, which is Appendix B of the SAP. A copy of the QAPP will be maintained by the field sampling team for immediate use in resolving any QA issues that might arise during field activities.

QC samples for all samples sent to subcontracted laboratories will be collected at the following frequencies:

- Field Duplicate: One per 10 environmental samples, with a minimum of one per sample matrix.
- MS/MSD Samples: One per 20 environmental samples per matrix. Due to the extended time frame of sampling, collection of additional MS/MSD samples may be necessary to close out a sample event.
- Rinsate Blank Samples: One per day of sampling to verify effectiveness of procedures applied to decontaminate non-disposable equipment (if necessary).

Field duplicate samples will consist of one sample which will be designated for homogenization in the laboratory and duplicate analysis; this sample should be ideally double the weight of a single sample (70 grams), but no less than 50 grams. A rinsate blank sample is collected by collecting analyte-free water that has been run over/through nondisposable or nondedicated sample collection equipment. Analytical results from these samples will indicate if contaminants have been introduced by contact of the sample medium with sampling equipment. MS/MSD is an environmental sample divided into two separate aliquots, each of which is spiked by the laboratory with known concentrations of target aliquots. The two spiked aliquots, in addition to an unspiked sample aliquot, are analyzed separately, and the results are compared to determine the effects of the matrix on precision and accuracy of the analysis. For fish samples, the MS/MSD should be collected at three times the minimum weight requirement of 35 grams (ideally 100 – 200 grams). All samples should be identified as MS/MSD for the laboratory.

REFERENCES

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ATTACHMENT A

FIGURES

(Three Sheets)

CBS MULTI-SITES
SITE LOCATIONS
MONROE COUNTY, INDIANA

FIGURE 1
SITE LOCATIONS

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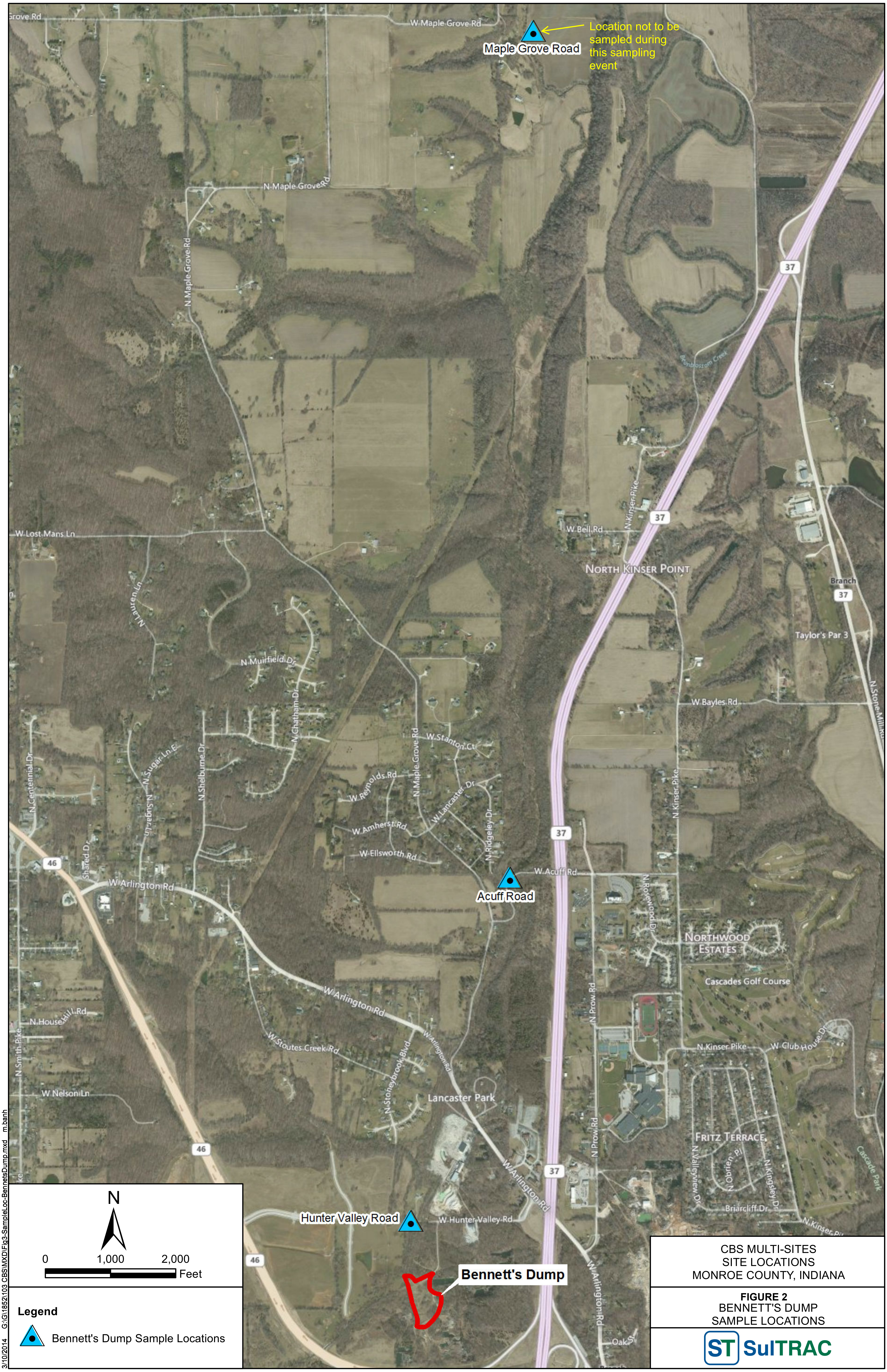

Legend

▲ Bennett's Dump Sample Locations

CBS MULTI-SITES
SITE LOCATIONS
MONROE COUNTY, INDIANA

FIGURE 2
BENNETT'S DUMP
SAMPLE LOCATIONS

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Legend

▲ Neal's Landfill Sample Locations

N

0 1,000 2,000 Feet

CBS MULTI-SITES
SITE LOCATIONS
MONROE COUNTY, INDIANA

FIGURE 3
NEAL'S LANDFILL
SAMPLE LOCATIONS

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ATTACHMENT B

SulTRAC STANDARD OPERATING PROCEDURES

(81 Sheets)

SOP APPROVAL FORM

ST SulTRAC

PROJECT-SPECIFIC

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

GENERAL EQUIPMENT DECONTAMINATION

SOP NO. 002

REVISION NO. 3

Last Reviewed: December 2013

John Diego

Quality Assurance Approved

04 December 2013

Date

1.0 BACKGROUND

All nondisposable field equipment must be decontaminated before and after each use at each sampling location to obtain representative samples and to reduce the possibility of cross-contamination.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for decontaminating equipment in the field.

1.2 SCOPE

This SOP applies to decontaminating general nondisposable field equipment. To prevent contamination of samples, all sampling equipment must be thoroughly cleaned prior to each use.

1.3 DEFINITIONS

Alconox: Nonphosphate soap, obtained in powder detergent form and dissolved in water
Liquinox: Nonphosphate soap, obtained in liquid form for mixing with water

1.4 REFERENCES

U.S. Environmental Protection Agency (EPA). 1992a. "Guide to Management of Investigation-Derived Wastes." Office of Solid Waste and Emergency Response. Washington D.C. EPA 9345.3-03FS. January.

EPA. 1992b. "RCRA Ground-Water Monitoring: Draft Technical Guidance." Office of Solid Waste. Washington, DC. EPA/530-R-93-001. November.

EPA. 1994. "Sampling Equipment Decontamination." Environmental Response Team SOP #2006 (Rev. #0.0, 08/11/94). http://www.ert.org/products/2006.PDF

1.5 REQUIREMENTS AND RESOURCES

The equipment required to conduct decontamination is as follows:

- Scrub brushes
- Large wash tubs or buckets
- Squirt bottles
- Alconox or Liquinox
- Tap water
- Distilled water
- Plastic sheeting
- Aluminum foil
- Methanol or hexane
- Isopropanol (pesticide grade)
- Dilute (0.1 N) nitric acid

2.0 PROCEDURE

The procedures below discuss decontamination of personal protective equipment (PPE), drilling and monitoring well installation equipment, borehole soil sampling equipment, water level measurement equipment, general sampling equipment, and groundwater sampling equipment.

2.1 PERSONAL PROTECTIVE EQUIPMENT DECONTAMINATION

Personnel working in the field are required to follow specific procedures for decontamination prior to leaving the work area so that contamination is not spread off site or to clean areas. All used disposable protective clothing, such as Tyvek coveralls, gloves, and booties, will be containerized for later disposal. Decontamination water will be containerized in 55-gallon drums (refer to Section 3.0).

Personnel decontamination procedures will be as follows:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.

3. Wash neoprene boots (or neoprene boots with disposable booties) with Liquinox or Alconox solution and rinse with clean water. Remove booties and retain boots for subsequent reuse.

4. Wash outer gloves in Liquinox or Alconox solution and rinse in clean water. Remove outer gloves and place into plastic bag for disposal.

5. Remove Tyvek or coveralls. Containerize Tyvek for disposal and place coveralls in plastic bag for reuse.

6. Remove air purifying respirator (APR), if used, and place the spent filters into a plastic bag for disposal. Filters should be changed daily or sooner depending on use and application. Place respirator into a separate plastic bag after cleaning and disinfecting.

7. Remove disposable gloves and place them in plastic bag for disposal.

8. Thoroughly wash hands and face in clean water and soap.

2.2 DRILLING AND MONITORING WELL INSTALLATION EQUIPMENT DECONTAMINATION

All drilling equipment should be decontaminated at a designated location on site before drilling operations begin, between borings, and at completion of the project. Decontamination may be conducted on a temporary decontamination pad constructed at satellite locations within the site area in support of temporary work areas. The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Decontamination pads may be constructed of concrete, wood, or plastic sheeting, depending on the site-specific needs and plans. Wash waters and contaminated soil generated during decontamination activities should be considered contaminated and thus, should be collected and containerized for proper disposal.

Monitoring well casing, screens, and fittings are assumed to be delivered to the site in a clean condition. However, they should be steam cleaned and placed on polyethylene sheeting on-site prior to placement downhole. The drilling subcontractor will typically furnish the steam cleaner and water.

The drilling auger, bits, drill pipe, any portion of drill rig that is over the borehole, temporary casing, surface casing, and other equipment used in or near the borehole should be decontaminated by the drilling subcontractor as follows:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.

2. Maintain the same level of personal protection as was used for sampling.

3. Remove loose soil using shovels, scrapers, wire brush, etc.

4. Steam clean or pressure wash to remove all visible dirt.

5. If equipment has directly or indirectly contacted contaminated media and is known or suspected of being contaminated with oil, grease, polynuclear aromatic hydrocarbons (PAH), polychlorinated biphenyls (PCB), or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol.

6. To the extent possible, allow components to air dry.

7. Wrap or cover equipment in clear plastic until it is time to be used.

8. All wastewater from decontamination procedures should be containerized.

2.3 BOREHOLE SOIL SAMPLING DOWNHOLE EQUIPMENT DECONTAMINATION

All soil sampling downhole equipment should be decontaminated before use and after each sample as follows:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.

2. Maintain the same level of personal protection as was used for sampling.

3. Prior to sampling, scrub the split-barrel sampler and sampling tools in a wash bucket or tub using a stiff, long bristle brush and Liquinox or Alconox solution.

4. After sampling, steam clean the sampling equipment over the rinsate tub and allow to air dry.

5. Place cleaned equipment in a clean area on plastic sheeting and wrap with aluminum foil.

6. Containerize all water and rinsate; disposable single-use sampling equipment should also be containerized.

7. Decontaminate all equipment placed down the hole as described for drilling equipment.

2.4 WATER LEVEL MEASUREMENT EQUIPMENT DECONTAMINATION

Field personnel should decontaminate the well sounder and interface probe before inserting and after removing them from each well. The following decontamination procedures should be used:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.
3. Wipe the tape and probe with a disposable Alconox- or Liquinox-impregnated cloth or paper towel.
4. If immiscible layers are encountered, the interface probe may require steam cleaning or washing with pesticide-grade isopropanol.
5. Rinse with deionized water.

2.5 GENERAL SAMPLING EQUIPMENT DECONTAMINATION

All nondisposable sampling equipment should be decontaminated using the following procedures:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.
3. To decontaminate a piece of equipment, use an Alconox wash; a tap water wash; a solvent (isopropanol, methanol, or hexane) rinse, if applicable, or dilute (0.1 N) nitric acid rinse, if applicable; a distilled water rinse; and air drying. Use a solvent (isopropanol, methanol, or hexane) rinse for grossly contaminated equipment (for example, equipment that is not readily cleaned by the Alconox wash). The dilute nitric acid rinse may be used if metals are the analytes of concern.
4. Place cleaned equipment in a clean area on plastic sheeting and wrap with aluminum foil.
5. Containerize all water and rinsate.

2.6 GROUNDWATER SAMPLING EQUIPMENT

The following procedures are to be employed for the decontamination of equipment used for groundwater sampling. Decontamination is not necessary when using disposable (single-use) pump tubing or bailers. Bailer and downhole pumps and tubing decontamination procedures are described in the following sections.

2.6.1 Bailers

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.
3. Evacuate any purge water in the bailer.
4. Scrub using soap and water and/or steam clean the outside of the bailer.
5. Insert the bailer into a clean container of soapy water. Thoroughly rinse the interior of the bailer with the soapy water. If possible, scrub the inside of the bailer with a scrub brush.
6. Remove the bailer from the container of soapy water.
7. Rinse the interior and exterior of the bailer using tap water.
8. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol.
9. Rinse the bailer interior and exterior with deionized water to rinse off the tap water and solvent residue, as applicable.
10. Drain residual deionized water to the extent possible.
11. Allow components to air dry.
12. Wrap the bailer in aluminum foil or a clean plastic bag for storage.
13. Containerize the decontamination wash waters for proper disposal.

2.6.2 Downhole Pumps and Tubing

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.

2. Maintain the same level of personal protection as was used for sampling.

3. Evacuate any purge water in the pump and tubing.

4. Scrub using soap and water and/or steam clean the outside of the pump and, if applicable, the pump tubing.

5. Insert the pump and tubing into a clean container of soapy water. Pump/run a sufficient amount of soapy water to flush out any residual well water. After the pump and tubing are flushed, circulate soapy water through the pump and tubing to ensure that the internal components are thoroughly flushed.

6. Remove the pump and tubing from the container.

7. Rinse external pump components using tap water.

8. Insert the pump and tubing into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).

9. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the pump and tubing with pesticide-grade isopropanol.

10. Rinse the pump and tubing with deionized water to flush out the tap water and solvent residue, as applicable.

11. Drain residual deionized water to the extent possible.

12. Allow components to air dry.

13. For submersible bladder pumps, disassemble the pump and wash the internal components with soap and water, rinse with tap water, isopropanol (if necessary), and deionized water, and allow to air dry.

14. Wrap pump and tubing in aluminum foil or a clean plastic bag for storage.

15. Containerize the decontamination wash waters for proper disposal.

3.0 INVESTIGATION-DERIVED WASTE

Investigation-derived waste (IDW) can include disposable single-use PPE and sampling equipment, soil cuttings, and decontamination wash waters and sediments. Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage will be provided in project-specific documents, or separate direction will be provided by the project manager. The following guidelines are provided for general use:

1. Assume that all IDW generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label IDW storage containers with the facility name and address, date, contents, company generating the waste, and an emergency contact name and phone number.

4. Temporarily store the IDW in a protected area that provides access to the containers and allows for spill/leak monitoring, sampling of containers, and removal following determination of the disposal method.

SOP APPROVAL FORM

SulTRAC

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

SEDIMENT AND SLUDGE SAMPLING

SOP NO. 006

REVISION NO. 4

Last Reviewed: December 2013

John Diego

Quality Assurance Approved

19 December 2013

Date

1.0 BACKGROUND

Sediments generally are materials deposited in surface impoundments or in natural waterways such as lakes, streams, rivers, oceans, and sloughs, as well as particulate matter deposited on the marsh or wetland surface.

Sludges are semisolid materials ranging from dewatered solids to high-viscosity liquids. Sludges generally accumulate as residuals of water-bearing waste treatment or industrial process systems. Sludges typically accumulate in tanks, drums, impoundments, or other types of containment systems.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for sampling sediment in lakes, streams, and rivers as well as sludge in open drums and shallow tanks (3 feet deep or less) or surface impoundments that are largely accessible. Sludge sampling of drums or other containers with smaller or less accessible openings is described in SOP 008.

1.2 SCOPE

This SOP applies to collection of sediment and sludge samples. It provides detailed procedures for gathering such samples with specific equipment.

1.3 DEFINITIONS

Bottom Dredge Grab Sampler: A clamshell-type metal scoop activated by a counter-lever latching system.

Composite Sample: A sample comprised of multiple grab samples representing a physical average of the total number of grab samples.

Discrete Sample: A sample comprised of a portion of material representing conditions present at a single unit of space and time.

Electronic Vibration Corer: A type of sampler that uses an electrically powered vibrating head, which vibrates vertically along the axis of the sampler to penetrate the sediment.

Gravity Corer: Metal tube with a tapered nosepiece on the bottom and a check valve on the top. The nosepiece reduces core disturbance during penetration. The check valve allows air and water to pass through the sampler during deployment and prevents sample loss (washout) during retrieval.

Hand Corer: Thin-wall tube or core barrel sampler. Some models include a tapered nosepiece, a "T" handle to facilitate sampler deployment and retrieval, and a check valve on top. Some models are available with extension handles, core barrel liners, core catchers, and sample extruders.

Transect Sampling: A series of samples collected at several locations oriented in a straight line.

1.4 REFERENCES

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U.S. Environmental Protection Agency (EPA). 1984. "Characterization of Hazardous Waste Sites -- A Methods Manual. Volume II -- Available Sampling Methods." Second Edition. EPA-600/A-84-076. December.

EPA. 1994. "Sediment Sampling." Environmental Response Team SOP #2016 (Rev. #0.0, 11/17/94). On-Line Address: http://www.ert.org/products/2016.PDF

EPA. 2001. "Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual." Office of Water. Washington, D.C. EPA/823/B-01/002. October.

AScI Corporation. 2002 "Collecting Sediment Samples by Vibracoring - Standard Operating Procedure." On-line Address: http://www.vibracoring.com/files/CoreSOP.pdf

1.5 REQUIREMENTS AND RESOURCES

The selection of sampling equipment and procedures should be based on project objectives and site-specific conditions such as the type and volume of sediment or sludge to be sampled, sampling depth, and the type of sample required (disturbed or undisturbed). The selected sampling equipment should be constructed of inert materials that will not react with the sediment or sludge being sampled.

The following equipment may be required to sample sediment or sludge:

- Plastic sheeting
- Field logbook
- Stainless steel or disposable spoons or spatulas
- Stainless-steel or disposable scoop or trowel
- Stainless-steel bowls
- Telescoping or fixed-length pole/handle for scoop or trowel
- Hip waders, chest waders, or high rubber boots (depending on water depth)
- Boat (depending on water depth)
- Gravity corer
- Hand corer
- Sludge sampler
- Electronic vibration corer
- Bottom dredge sampler
- Stainless-steel or Teflon® tray
- Nylon rope
- Sample containers and labels
- Chain-of-custody and shipping materials
- Decontamination materials

2.0 PROCEDURES

This section provides general procedures for sampling sediment and sludge. Sections 2.1 through 2.5 specify the methods and equipment to be used for such sampling.

Sediment Sampling

Sediment can be sampled using a stainless-steel scoop or trowel (see Section 2.1), a hand corer (see Section 2.2), a gravity corer (see Section 2.3), an electronic vibration corer (see Section 2.4), or a bottom dredge grab sampler such as a Ponar grab sampler (see Section 2.5). Soil core catchers and brass, polycarbonate plastic, or Teflon® liners are often used with core samplers. A number of factors must be considered when selecting the type of sampler to be used. In streams, lakes, and impoundments, for instance, sediment is likely to demonstrate significant variations in composition.

For stream sediment sampling, the sampling location farthest downstream should be sampled first to avoid cross-contamination. Sediment samples collected in upstream and downstream locations should be obtained in similar depositional environments and, whenever possible, should be obtained from slow-moving pools. In addition, a sediment sample should be collected at approximately the same location as an associated aqueous sample. Aqueous samples should be obtained first to avoid collecting suspended particles that may result from sediment sampling. To avoid disturbing an area to be sampled, sampling locations in streams should always be approached from the downstream side.

Sediment samples collected from lakes and impoundments should also be collected at approximately the same locations as associated aqueous samples. As in stream sampling, aqueous samples should be collected first to avoid collecting suspended particles that may result from sediment sampling. Downgradient and background samples should be collected from similar depositional environments.

Typically, fine-grained sediments should be targeted as they have greater surface area available for adsorption of contaminants. In streams or rivers, low-velocity depositional areas (bends, areas behind obstructions, pooling areas) are preferred sampling locations. High velocity turbulent areas should be avoided, if possible. Samples may be collected at a single location, along a transect line, or as composite samples consisting of material from multiple grab samples.

Often times the top 10 centimeters (approximately top six inches) of sediment is targeted as the sample interval because most benthic organisms are found in this horizon. Sampling multiple locations can provide information on the horizontal distribution of contaminants in a given water body. A column of sediment can provide information on the vertical distribution of contaminants with depth. Sampling locations, the number of samples to be collected, sampling rationale, and sampling approach (grab, transect, composite, etc.) should be discussed in the project-specific plans.

Exact sampling locations should be documented in field logbooks or on data sheets with respect to fixed reference points or located using global positioning satellite (GPS) technology. In addition, the presence of rocks, debris, or organic material in the sludge or sediment to be sampled may preclude use of some types of samplers or require modification of sampling equipment.

Sludge Sampling

Sludge can often be sampled using a stainless-steel scoop or trowel (see Section 2.1). Frequently sludge forms when components with higher densities settle out of a liquid. When this happens, the sludge may still have an upper liquid layer above the denser components. When the liquid layer is sufficiently shallow, the sludge may be sampled using a hand corer (see Section 2.2). Use of the hand corer is preferred because it results in less sample disturbance. The hand corer also allows for the collection of an aliquot of the overlying liquid. This prevents drying or excessive oxidation of a sample before analysis. The hand corer may also be adapted to hold a brass, polycarbonate plastic, or Teflon® liner.

A gravity corer may also be used to collect samples of most sludges and sediments (see Section 2.3). A gravity corer is capable of collecting an undisturbed sample that profiles the strata present in a sludge or sediment. Depending on the weight of the gravity corer and the density of the sludge or sediment, a gravity corer may penetrate the material up to 30 inches. If the layer is shallow (less than 1 foot), gravity corer and hand corer penetration may damage any underlying liner or confining layer. In such situations, a Ponar grab sampler may be used because it is generally capable of penetrating only a few inches (see Section 2.4).

The following subsections specify methods for sediment and sludge sampling with specific equipment.

2.1 SAMPLING WITH A SCOOP OR TROWEL

Sediment or sludge samples may be collected with a simple scoop or trowel. This method is more applicable to sludge but can be used for sediments provided that the water is very shallow (a few inches). However, using a scoop or trowel may disrupt the water-sediment interface and cause substantial sample alteration. This method provides a simple, quick means of collecting a disturbed sample of sludge or sediment.

The following procedure can be used for sampling sludge or sediment with a scoop or trowel:

1. Place all sampling equipment on plastic sheeting next to the sampling location Sample containers should be selected in accordance with the requirements in the project field sampling plan (FSP) or quality assurance project plan (QAPP).
2. Affix a completed sample container label to the appropriate sample container.
3. Carefully insert a pre-cleaned scoop or trowel into the sludge or sediment and remove the sample. In the case of sludge exposed to air, remove the first 2 to 4 inches of material before collecting the sample. In the case of collecting sediment with abundant debris or vegetation at the surface, carefully remove the debris (avoid disturbing the sediment to the extent possible) before collecting the sample.
4. When compositing a series of grab samples, combine the samples in a stainless-steel bowl or Teflon® tray. Record pertinent information in the field logbook (sample description, color, odor, texture, etc.) or in a field data collection form, if applicable. If samples are to be collected for VOC analysis, fill the appropriate VOC containers first before compositing the sample and logging the sample information.
5. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.
6. If required, preserve the sample in accordance with the requirements in the project FSP or QAPP.
7. Ensure that a Teflon® liner is present in the sample container cap, if required. Secure the cap tightly on the sample container.
8. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements. Samples are typically stored in coolers on ice before and during shipment.
9. Decontaminate all non-disposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

2.2 SAMPLING WITH A HAND CORER

The hand corer, sometimes called a hand auger (see Figure 1), is used in the same situations and for the same materials as those described for the use of a scoop or trowel (see Section 2.1). However, the hand corer may be used to collect an undisturbed sample that can profile any stratification resulting from changes in material deposition thus allowing for sampling of the specific layer or layers of interest.

The exact type of hand corer will vary depending on the manufacturer, but the operational procedures discussed below are applicable to most types of hand corers. For example, some hand corers can be fitted with extension handles that allow collection of samples underlying a shallow layer of liquid. Most hand corers can be fitted with core catchers (Figure 1) to prevent sample loss upon retrieval and they can be adapted to hold liners, which are generally available in brass, polycarbonate plastic, or Teflon®. The type of hand corer and liner material should be chosen that will not compromise the intended analytical procedures.

The following procedure can be used for sampling sludge or sediment with a hand corer:

1. Place all sampling equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in the project FSP or QAPP.

2. Affix a completed sample container label to the appropriate sample container.

3. Position a pre-cleaned hand corer above the sampling location. If the sediment or sludge is non-cohesive, insert a core catcher into the end of the sampler. Carefully deploy the hand corer into the sludge or sediment using a smooth, continuous motion. Gently rotating the corer while it is being pushed may facilitate greater penetration and decrease core compaction.

4. When the hand corer is at the desired depth, rotate the “T” handle or the core barrel and retrieve the hand corer using a single, smooth motion. If the hand corer does not include a “T” handle, cap the top of the core barrel with your hand to provide additional suction during retrieval.

5. Remove the core catcher and nosepiece (if applicable) and extract the sample. Place the sample on a clean stainless-steel or Teflon® tray. When compositing a series of grab samples, combine the samples in a stainless-steel bowl or Teflon® tray. Record pertinent information in the field logbook (sample description, color, odor, etc.) or in a field data collection form, if applicable. If samples are to be collected for VOC analysis, fill the appropriate VOC containers first before compositing the sample and logging the sample information.

6. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

7. If required, preserve the sample in accordance with the requirements in the project FSP or QAPP.

8. Ensure that a Teflon® liner is present in the sample container cap, if required. Secure the cap tightly on the sample container.

9. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements. Samples are typically stored in coolers on ice before and during shipment.

10. Decontaminate all non-disposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

2.3 SAMPLING WITH A GRAVITY CORER

A gravity corer (see Figure 2) can collect essentially undisturbed samples to profile strata that develop in sediment and sludge during the deposition process. Depending on the sediment or sludge density and the gravity corer’s weight, the sampler typically can penetrate the sediment or sludge to a depth of 30 inches, using the weight of the sampler to assist penetration. A gravity corer is lowered into the sediment from a fixed support (such as a boat or a portable tripod pole system).

Gravity corers should be used carefully in open drums, shallow tanks, or lagoons with liners. A gravity corer could penetrate beyond the sludge or sediment layer and damage the liner material.

The following procedure can be used for sampling sediment or sludge with a gravity corer:

1. Place all sampling equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in the project FSP or QAPP.

2. Affix a completed sample container label to the appropriate sample container.

3. Attach the required length of sample line to a pre-cleaned gravity corer. Braided, 3/16-inch nylon line is sufficient; however, 3/4-inch nylon line is easier to grasp during hoisting.

4. Secure the free end of the line to a fixed support to prevent accidental loss of the gravity corer.

5. Position the gravity corer above the sampling location. Allow the gravity corer to fall freely through the liquid and penetrate the sludge or sediment layer.

6. Retrieve the gravity corer with a smooth, continuous lifting motion. Do not bump the corer, as this may result in some sample loss.

7. Remove the core catcher and nosepiece (if applicable) from the gravity corer. Slide the sample out of the corer into a stainless-steel or Teflon® pan. When compositing a series of grab samples, combine the samples in a stainless-steel bowl or Teflon® tray. Record pertinent information in the field logbook (sample description, color, odor, etc.) or in a field data collection form, if applicable. If samples are to be collected for VOC analysis, fill the appropriate VOC containers first before compositing the sample and logging the sample information.

8. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

9. If required, preserve the sample in accordance with the requirements in the project FSP or QAPP.

10. Ensure that a Teflon® liner is present in the sample container cap, if required. Secure the cap tightly on the sample container.

11. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements. Samples are typically stored in coolers on ice before and during shipment.

12. Decontaminate all non-disposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

2.4 SAMPLING WITH AN ELECTRONIC VIBRATION CORE SAMPLER

Electronic vibration corers are commonly used core samplers because they can retrieve deep core samples in most types of sediment. For example, electronic vibration corers can be used from a boat to collect a sample from the shallow sea floor. Vibration corers typically are not appropriate for sludge sampling as they could penetrate beyond the sludge layer and damage the liner material of a container.

Vibration samplers have an electrically powered vibrating head, which vibrates vertically along the axis of the sampler to penetrate the sediment (Figure 3). The core barrel and liner are inserted into the head of the vibrator and the entire assembly is lowered into the water. A vibrating core sampler can penetrate compact sediments and collect core samples up to 10 meters long depending on the type of system used, the horsepower of the vibrating head, and weight of the sampler.

The two general types of vibrating core systems typically used include larger boat-deployed systems and portable pole systems. Larger vibrating core samplers require the use of an appropriately sized boat to maintain balance and provide adequate lift to break the head of the corer out of the sediment for sample retrieval (EPA 2001).

The following procedure can be used for sampling sediment with a submersible boat-deployed vibration corer:

1. Place all sampling equipment on plastic sheeting next to the sampling location if working next to the shore or stream bank. Sample containers should be selected in accordance with the requirements in the project FSP or QAPP.

2. Affix a completed sample container label to the appropriate sample container.

3. Securely attach the vibrating corer assembly to the winch cable.

4. Position the vibrating corer above the sampling location. Using the winch, suspend and lower the corer until the core tube contacts the bottom.

5. Begin vibration and continue penetration until the core tube is fully buried or refusal occurs. Care should be taken to keep the cable taught ensuring that the core tube is vertical. Turn off vibration when coring is completed.

6. Retrieve the vibration corer with the winch, using vibration only if extraction is difficult. Keep the core tube in a near vertical position once retrieved.

7. Remove the core catcher, nosepiece, and liner from the core tube. Cap the ends of the liner, label the liner to identify the location, sample number, time of collection, and date. Transfer the sample on-shore for further processing. Record pertinent information in the field logbook (sample description, color, odor, etc.) or on a field data collection form.

8. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

9. If required, preserve the sample in accordance with the requirements in the project FSP or QAPP.

10. Ensure that a Teflon® liner is present in the sample container cap, if required. Secure the cap tightly on the sample container.

11. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements. Samples are typically stored in coolers on ice before shipment.

12. Decontaminate all non-disposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

The following procedure can be used for sampling sediment with a portable pole system vibration corer:

1. Place all sampling equipment on plastic sheeting next to the sampling location if working next to the shore or stream bank. Sample containers should be selected in accordance with the requirements in the project FSP or QAPP.

2. Affix a completed sample container label to the appropriate sample container.

3. Securely attach the retrieval lines to the core tube mounting cap.

4. Insert liner tube (core catcher end down) into mounting clamp and hand-tighten wing nuts evenly.

5. Insert the desired length extension pole into mounting plate socket and secure with bolt and locknut. Slip flared lower end of the extension tube over the check-valve end of the core tube adapter while applying tension on the retrieval lines. Lower the system carefully until it contacts the bottom.

6. Press and vibrate the core tube until it is fully buried or refusal occurs. Note the depth of penetration by markings on the extension pole. Turn off vibration when coring is completed.

7. Disconnect the extension pole and manually retrieve the vibration corer with the retrieval lines or use a hand winch, if necessary. Keep the core tube in a near vertical position once retrieved.

8. Remove the core catcher, nosepiece, and liner from the core tube. Cap the ends of the liner, label the liner to identify the location, sample number, time of collection, and date. Transfer the sample on-shore for further processing. When compositing a series of grab samples, combine the samples in a stainless-steel bowl or Teflon® tray. Record pertinent information in the field logbook (sample description, color, odor, etc.) or on a field data collection form. If samples are to be collected for VOC analysis, fill the appropriate VOC containers first before compositing the sample and logging the sample information.

9. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

10. If required, preserve the sample in accordance with requirements in the project FSP or QAPP.

11. Ensure that a Teflon® liner is present in the sample container cap. Secure the cap tightly on the sample container.

12. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements. Samples are typically stored in coolers on ice before and during shipment.

13. Decontaminate all non-disposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

2.5 SAMPLING WITH A BOTTOM DREDGE SAMPLER

Bottom dredge samplers are typically used to sample sediments that cannot be easily collected using trowels, scoops, or coring devices or in cases where large quantities of sample are desired. Bottom dredge samples are particularly effective when sampling from a boat where there are several feet of water above the sediment surface. Several types and sizes of dredges exist including the Peterson, Eckman, and Ponar. Dredges can be activated upon contact with the sediment or by using a "messenger" to close the buckets. The procedures discussed below are applicable to sampling with a Ponar dredge sampler. A Ponar grab sampler (see Figure 4) can be used to sample most types of sludges and sediments. A Ponar grab sampler is used by holding the grab sampler above the area to be sampled and lowering the grab sampler until it makes contact with the sediment. Its penetration depth into the sediment usually does not exceed several inches. The Ponar grab sampler, like other grab samplers, cannot collect undisturbed samples; therefore, this sampler should be used only after all overlying water samples have been collected.

The following procedure can be used for sampling sludge or sediment with a Ponar grab sampler:

1. Place all sampling equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in the project FSP or QAPP.

2. Affix a completed sample container label to the appropriate sample container.

3. Attach the required length of sample line to a pre-cleaned Ponar grab sampler. Braided, 3/4-inch nylon line is recommended for ease in hoisting.

4. Measure the distance from the water surface or other reference point to the top of the sludge or sediment. Mark this measurement on the sample line. To avoid unnecessary disturbance of the sludge or sediment from lowering the Ponar grab sampler too quickly, it is recommended that a second mark be made on the sample line to indicate the proximity of the reference mark.

5. Open the Ponar sampler's jaws until they are latched. The jaws will be triggered if the Ponar sampler comes in contact with or is supported by anything other than the sample line. Tie the free end of the sample line to a fixed support.

6. Position the Ponar grab sampler above the sampling location. Lower the sampler until the proximity mark is reached. Then, slowly lower the Ponar grab sampler until it touches and penetrates the sludge or sediment.

7. Allow the sample line to slacken a few inches to release the latching mechanism that closes the sampler's jaws. As the jaws close, they scoop the sludge or sediment up into the sampler. More slack may be required when sampling in surface waters with strong currents.

8. Retrieve the sampler, open the jaws carefully, and release its contents into a stainless-steel or Teflon® tray. When compositing a series of grab samples, combine the samples in a stainless-steel bowl or Teflon® tray. Record pertinent information in the field logbook (sample description, color, odor, etc.) or on a field data collection form. If samples are to be collected for VOC analysis, fill the appropriate VOC containers first before compositing the sample and logging the sample information.

9. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

10. If required, preserve the sample in accordance with the requirements in the project FSP or QAPP.

11. Ensure that a Teflon® liner is present in the sample container cap, if required. Secure the cap tightly on the sample container.

12. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements. Samples are typically stored in coolers on ice before and during shipment.

13. Decontaminate all non-disposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

FIGURE 1

HAND CORER

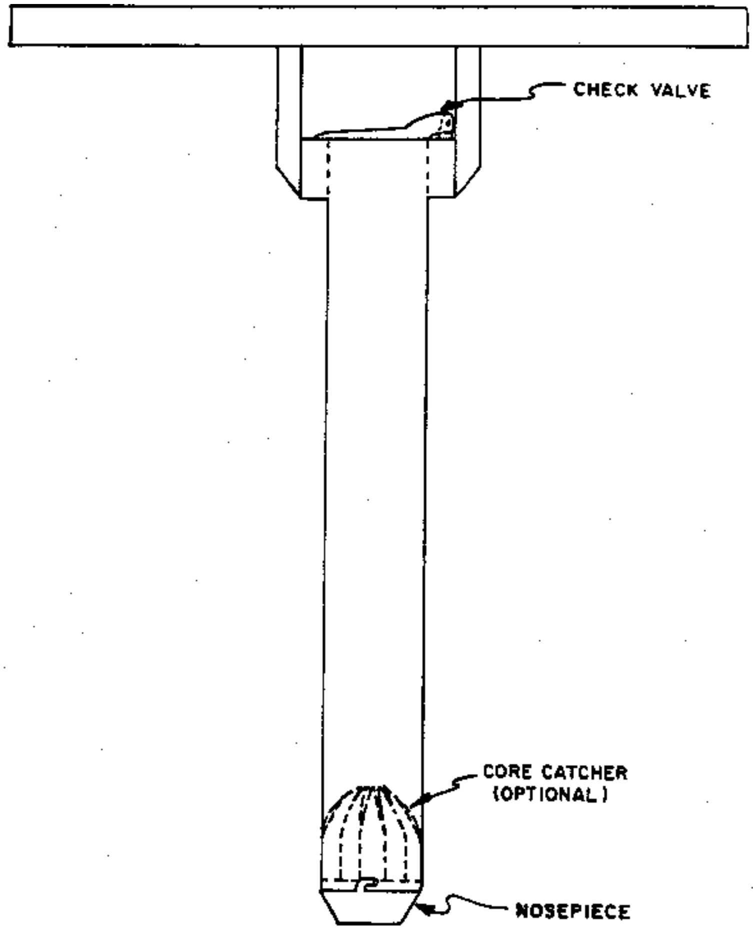


FIGURE 2

GRAVITY CORER

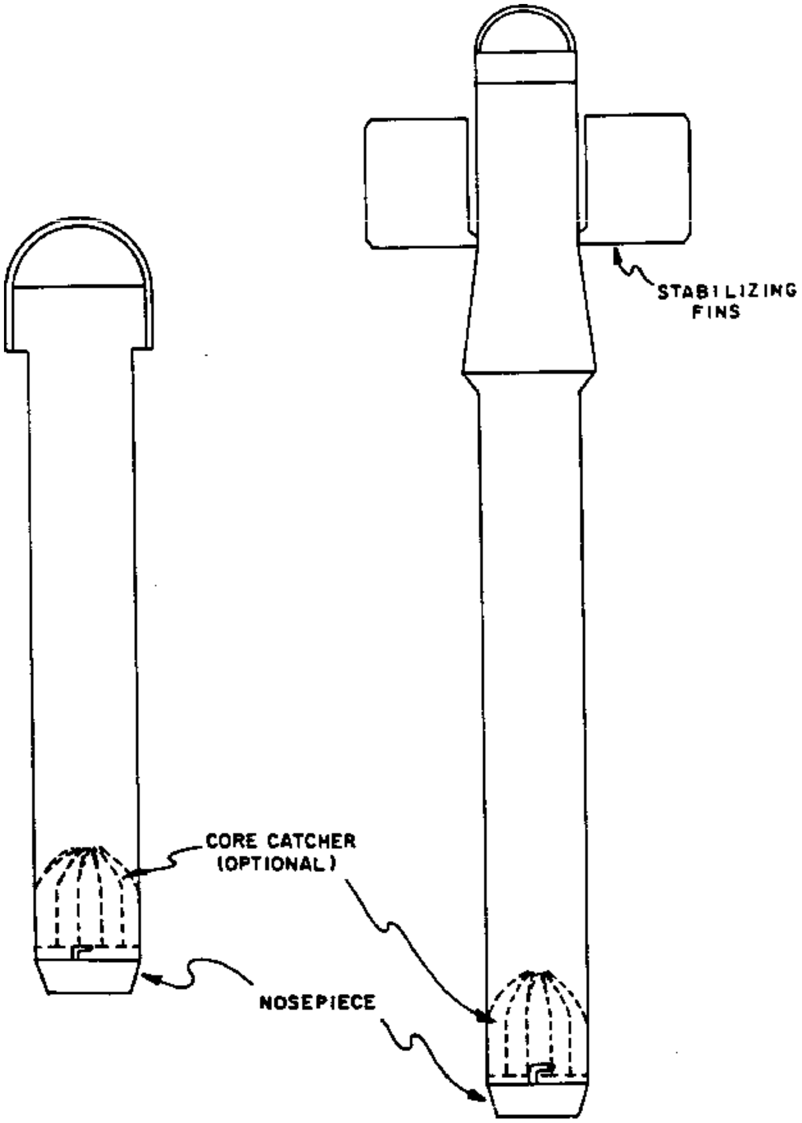


FIGURE 3

VIBRACORE SAMPLER

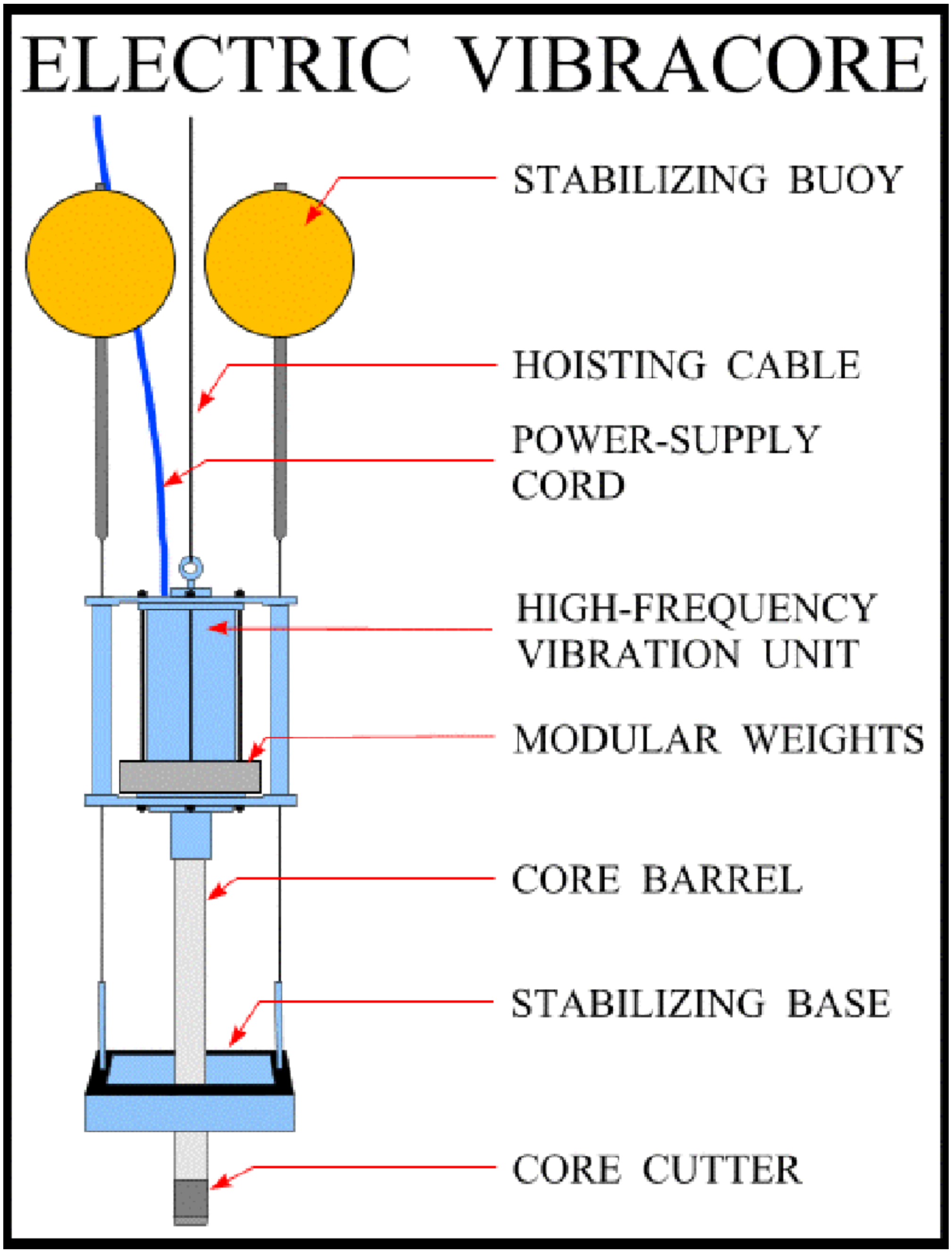
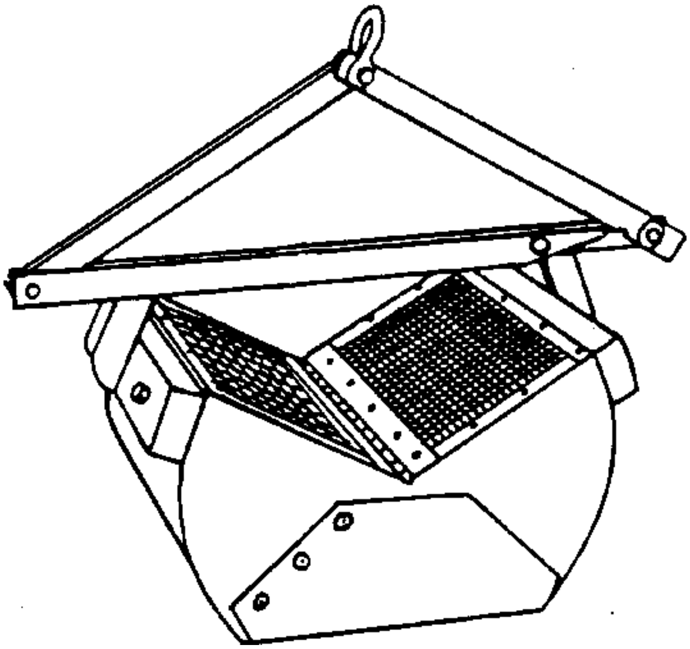


FIGURE 4

BOTTOM DREDGE GRAB SAMPLER



SOP APPROVAL FORM

ST SulTRAC

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

SURFACE WATER SAMPLING

SOP NO. 009

REVISION NO. 4

Last Reviewed: December 2013

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Date

1.0 BACKGROUND

Surface water sampling is conducted to determine the quality of surface water entering, leaving, or affected by a site. Surface water bodies that can be sampled include streams, rivers, lakes, ponds, lagoons, and surface impoundments. This standard operating procedure (SOP) discusses common methods of collecting grab samples that represent water quality in a water body at a particular point in time.

A series of grab samples also can be composited to represent water quality over a longer period of time. Composite samples can be flow proportional or time proportional. The details of compositing water samples are not included in this SOP.

1.1 PURPOSE

This SOP establishes the requirements and procedures for surface water sampling.

1.2 SCOPE

This SOP applies to surface water sampling and the instruments and methods used to collect the samples.

1.3 DEFINITIONS

Kemmerer Sampler: A messenger-activated water sampling device. Water flows through the device until the release mechanism is triggered to close the container.

Peristaltic Pump: A rotary, positive-displacement pumping device characterized by its low suction and rhythmic operation, and by the fact that the pump does not come into direct contact with the water being sampled.

Pond Sampler: A sampling device fabricated by using an adjustable beaker clamp to attach a beaker to a telescoping, heavy-duty aluminum pole.

1.4 REFERENCES

U.S. Environmental Protection Agency (EPA). 1977. "Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities." EPA-530/SW-611. August.

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1.5 REQUIREMENTS AND RESOURCES

Surface water sampling requires a variety of procedures and instruments. The choice of procedure should be determined by site-specific conditions, such as the type of surface water body, the sampling depth, and the sample location's distance from shore.

Samples can be collected from shallow depths by submerging the sample container. An intermediary disposable collection container or one constructed of a nonreactive material also may be used. A pond sampler, a peristaltic pump, or a Kemmerer sampler may be used to provide extended reach. The following equipment may be required to sample surface water:

- Decontamination materials
- Sample containers and labels
- Point-source bailer
- Dipper
- Boat
- Pond sampler
- Peristaltic pump with batteries or power source
- Silicone tubing
- Heavy-wall Teflon® tubing

- Kemmerer sampler
- Bucket
- Logbook or field data sheets
- Chain-of-custody documentation
- Shipping materials

2.0 PROCEDURES

Safe access, handling, and other physical limitations should be influential factors during surface water sampling. A site-specific sampling plan should delineate which of the procedures described below will be used. Any deviations from the sampling plan should be recorded in the site-specific field logbook.

The following subsections provide detailed procedures for surface water sampling using specific instruments and methods. In all cases, select a sampling location where the water quality will best represent the water chemistry of the water body. Avoid stagnant or fast-moving areas. Do not sample immediately downstream of incoming tributaries, because of the likelihood of incomplete mixing.

2.1 SURFACE WATER SAMPLING BY SUBMERGING SAMPLE CONTAINER

Samples from shallow depths should be collected by submerging the sample container. This method is advantageous when the sample might be significantly altered during transfer from a collection vessel into another container. This method should not be used for sampling lagoons or surface impoundments where contact with contaminants is a potential concern.

The following procedure can be used for sampling surface water by submerging the sample container:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements specified in the project-specific field work plan, field sampling plan, or quality assurance project plan (QAPP).

2. If required by the project, collect field parameter measurements using procedures in relevant specific Tetra Tech SOPs and project-specific field sampling plan. Record this information on the field sheet or in the logbook.

3. A visual check for visible surface material (pond scum or ice) should be performed before sampling. If present, surface water samples should be collected by directly

submerging the sample container (with lid still on) into the surface water at the specified sampling location. Avoid contacting the bottom of the water body with the sample container because this will disturb sediment that may interfere with the surface water sample. Once submerged, the lid should be removed to allow the container to fill with water below any visible material on the surface of the water. A visual check should be conducted during and after sample collection to ensure sample integrity. If no surface materials are present, sample as instructed below.

4. For stream sampling, sample the location farthest downstream first. In general, work from zones suspected of low contamination to zones of high contamination. Orient the mouth of the sample container facing upstream while standing downstream so as not to stir up any sediment that would contaminate the sample. Avoid contacting the bottom of the water body with the sample container because this will disturb sediment that may interfere with the surface water sample.

5. For a larger body of surface water, such as a lake, collect samples near the shore, unless boats are feasible and permitted. Collect samples from shallow depths by submerging the sample container. Avoid contacting the bottom of the water body with the sample container because this will disturb sediment that may interfere with the surface water sample. If sampling from a boat, collect the sample as far away as possible from the outboard engine to avoid possible fuel contamination.

6. If sediment samples are to be collected (using procedures in SOP No. 006 [Sludge and Sediment Sampling]) with surface water samples, collect surface water samples at each location before collecting sediment samples to avoid contaminating the water samples with excess suspended particles generated during sediment sampling.

7. Continue delivery of the sample until the container is almost full. If sampling for volatile organic compounds (VOC) or other analytical parameters requiring pre-preserved sample containers, the use of a transfer device is recommended so that the preservative is not displaced.

8. Preserve the sample in accordance with requirements specified in the project-specific field work plan, field sampling plan, or QAPP. Ensure that a Teflon® liner is present in the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

9. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

2.2 SURFACE WATER SAMPLING WITH TRANSFER DEVICE

A dipper, bailer, or other device made of inert material, such as stainless steel or Teflon®, can be used to transfer liquid samples from their source to a sample container. This prevents contamination of the outside of the sample container as a result of direct immersion in surface water. Depending on the sampling application, the transfer device may be either disposed of or reused. If reused, the device should be thoroughly rinsed and decontaminated in accordance with SOP 002 (General Equipment Decontamination), prior to sampling a different source.

A transfer device can be used in most sampling situations, and is preferred when (1) direct contact or physical access limitations pose a health and safety concern and (2) sample containers are pre-preserved. However, direct collection by submerging the sample container is the preferred method when possible.

The following procedure can be used for sampling surface water with a dipper, bailer, or other transfer device:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements specified in the project-specific field work plan, field sampling plan, or QAPP.
2. If required by the project, collect field parameter measurements using procedures in relevant specific Tetra Tech SOPs. Record this information on the field sheet or in the logbook.
3. With minimal surface water disturbance, submerge a precleaned dipper, bailer, or other transfer device.
4. Allow the device to fill slowly and continuously.
5. Retrieve the device from the surface water with minimal disturbance.
6. Remove the cap from the sample container. Slightly tilt the mouth of the container below the edge of the transfer device.
7. Empty the device slowly, allowing the sample to flow gently down the inside of the container with minimal entry turbulence. Continue delivery of the sample until the container is almost full. If sampling for VOCs, the container must be completely filled leaving no head space.
8. Preserve the sample in accordance with requirements specified in the project-specific field work plan, field sampling plan, or QAPP. Ensure that a Teflon® liner is present in

the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

9. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

10. Decontaminate the transfer device prior to reuse or storage using the procedures in SOP No. 002 (General Equipment Decontamination).

2.3 SURFACE WATER SAMPLING WITH POND SAMPLER

A pond sampler may be used to collect liquid samples from ponds, pits, and lagoons (see Figure 1). A pond sampler is easily and inexpensively fabricated. To construct a pond sampler, use an adjustable clamp to attach a sampling beaker to the end of a two- or three-piece telescoping aluminum tube. The telescoping tube serves as the handle. All nondisposable equipment should be cleaned before and after each use.

The following procedure can be used for sampling surface water with a pond sampler:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements specified in the project-specific field work plan, field sampling plan, or QAPP.

2. If required by the project, collect field parameter measurements using procedures in relevant specific Tetra Tech SOPs. Record this information on the field sheet or in the logbook.

3. Assemble the pond sampler. Ensure that the sampling beaker, bolts, and nuts securing the clamp to the pole are tightened properly.

4. Collect the sample by slowly submerging the precleaned beaker with minimal surface water disturbance.

5. Retrieve the pond sampler from the surface water with minimal disturbance.

6. Remove the cap from the sample container. Slightly tilt the mouth of the container below the edge of the beaker.

7. Empty the beaker slowly, allowing the sample to flow gently down the inside of the container with minimal entry turbulence. Continue delivery until the container is almost full. If sampling for VOCs, the container must be completely filled leaving no head space.

8. Preserve the sample in accordance with requirements specified in the project-specific field work plan, field sampling plan, or QAPP. Ensure that a Teflon® liner is present in the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

9. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

10. Decontaminate the pond sampler prior to reuse or storage using the procedures in SOP No. 002 (General Equipment Decontamination).

2.4 SURFACE WATER SAMPLING WITH PERISTALTIC PUMP

To extend reach in sampling efforts, a small peristaltic pump can be used (see Figure 2). A peristaltic pump draws the sample through heavy-wall Teflon® tubing and pumps it directly into the sample container. Use of a peristaltic pump allows the operator to reach out into a liquid body, to sample from a depth or to sweep the width of a narrow stream. A battery-powered pump is preferable because it eliminates the need for a direct current generator or an alternating current inverter.

If medical-grade silicone tubing is used in the peristaltic pump, it is suitable for sampling almost any parameter, including most organics. However, some VOC stripping may occur and some sample material may adhere to the tubing. Teflon® tubing may be used in place of silicone tubing on the intake side of the pump to minimize the amount of sample adherence to the tubing. If tubing is to be reused, it should be cleaned before and after each use following the procedures specified in SOP No. 002 (General Equipment Decontamination). Depending on project requirements, it may be necessary to replace the Teflon® intake tubing and the pump silicone tubing between sampling locations to prevent cross contamination.

Procedures for sampling surface water with a peristaltic pump are as follows:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements specified in the project-specific field work plan, field sampling plan, or QAPP.

2. If required by the project, collect field parameter measurements using procedures in relevant specific Tetra Tech SOPs. Record this information on the field sheet or in the logbook.

3. Install clean, medical-grade silicone tubing in the pump head according to the manufacturer's instructions. Allow enough tubing on the discharge side to facilitate

delivery of liquid into the sample container. Allow only enough tubing on the suction end for attachment to the intake line. This will minimize sample contact with the tubing.

4. Select the length of intake tubing needed to reach the required sample location. Attach it to the intake side of the pump tubing. Heavy-wall Teflon® tubing of a diameter equal to that of the required pump tubing suits most applications. A heavier tubing wall will allow slightly greater lateral reach.

5. If possible, allow several liters of surface water to pass through the pump before collecting the sample. Collect this purge volume. Return it to the source after the samples have been withdrawn.

6. Fill the sample container by allowing the pump discharge to flow gently down the inside of the bottle with minimal entry turbulence. Continue delivery of the sample until the container is almost full.

7. If sampling for VOCs, the VOC sample must be collected using one of the “soda straw” variations. Ideally, the tubing intake will be placed at the depth from which the sample is to be collected and the pump will be run for several minutes to fill the tubing with water representative of that interval. After several minutes, the pump is turned off and the tubing string is retrieved. The pump speed is then reduced to a slow pumping rate and the pump direction is reversed. After the pump is turned back on, the sample stream is collected into the VOC vials as it is pushed from the tubing by the pump. Care must be taken to prevent any water that was in contact with the peristaltic pump head tubing from being incorporated into the sample.

8. Preserve the sample in accordance with requirements specified in the project-specific field work plan, field sampling plan, or QAPP. Ensure that a Teflon® liner is present in the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

9. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

10. Allow the pump to drain, and then disassemble it. Decontaminate the tubing before reuse using the procedures in SOP No. 002 (General Equipment Decontamination), or dispose of it.

2.5 SURFACE WATER SAMPLING WITH KEMMERER SAMPLER

The Kemmerer sampler (see Figure 3) is used to collect surface water samples when the required sample depth is greater than that which can be sampled with a pump. A Kemmerer sampler may be constructed of various materials to be compatible with the required analytical technique. The sampler should be cleaned before and after each use.

Procedures for sampling surface water with a Kemmerer sampler are as follows:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in specified in the project-specific field work plan, field sampling plan, or QAPP.

2. If required by the project, collect field parameter measurements using procedures in relevant specific Tetra Tech SOPs. Record this information on the field sheet or in the logbook.

3. Inspect the body of the Kemmerer sampler to ensure that the drain line valve is closed, as appropriate. Measure and mark the sample line (cable) at the desired sampling depth.

4. Open the sampler by lifting the upper stopper-trip head assembly.

5. Gradually lower the sampler into the surface water until the sample liquid reaches the sample line.

6. Place a messenger on the sample line and release it, closing the sampler.

7. Retrieve the sampler. Prevent accidental opening of the lower stopper by holding the center rod of the sampler.

8. Rinse or wipe off the exterior of the sampler. Recover the sample by grasping the lower stopper and sampler body with one hand. Transfer the sample by lifting the upper stopper with the other hand and carefully pouring the contents into the sample container. If a drain line valve is present, hold the valve over the sample container, and open the valve slowly to release the sample.

9. Transfer the sample slowly, allowing it to flow gently down the inside of the container with minimal entry turbulence. Continue delivery until the container is almost full. If sampling for VOCs, the container must be completely filled leaving no head space.

10. Preserve the sample in accordance with requirements specified in the project-specific field work plan, field sampling plan, or QAPP. Ensure that a Teflon® liner is present in the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

11. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

12. Decontaminate the Kemmerer sampler prior to reuse or storage using the procedures in SOP No. 002 (General Equipment Decontamination).

2.6 SURFACE WATER SAMPLING WITH BUCKET

A plastic bucket is used to collect surface water samples for measurement of water quality parameters (such as pH, temperature, and conductivity) or classical water quality parameters (ammonia, nitrate-nitrite, phosphorus, and total organic carbon). This method is not recommended for collecting samples for chemical analysis. A bucket is commonly used to collect a sample when the water depth is too great for wading, it is not possible to deploy a boat, or access is restricted (excessive vegetation or steep embankments) and the water column is well mixed. The water body is usually accessed from a bridge. The bucket is lowered by rope over the side of the bridge and, upon retrieval, the water is poured into the appropriate sample containers.

FIGURE 1

POND SAMPLER

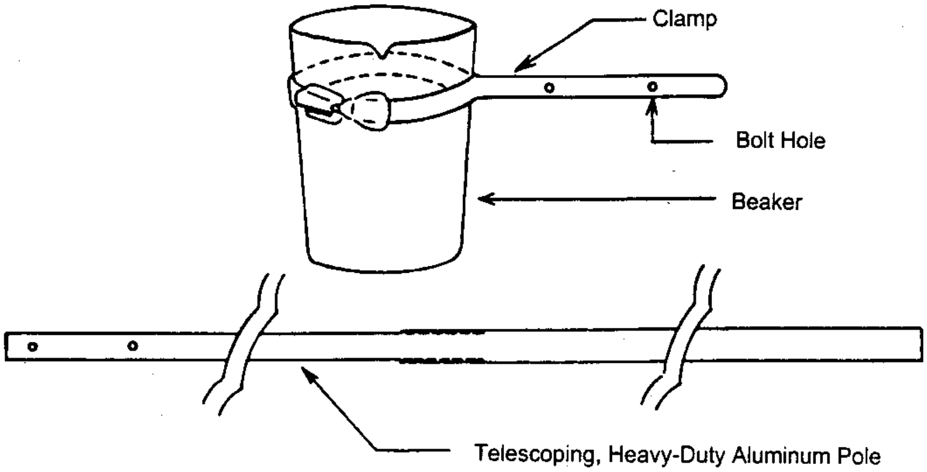


FIGURE 2

PERISTALTIC PUMP FOR LIQUID SAMPLING

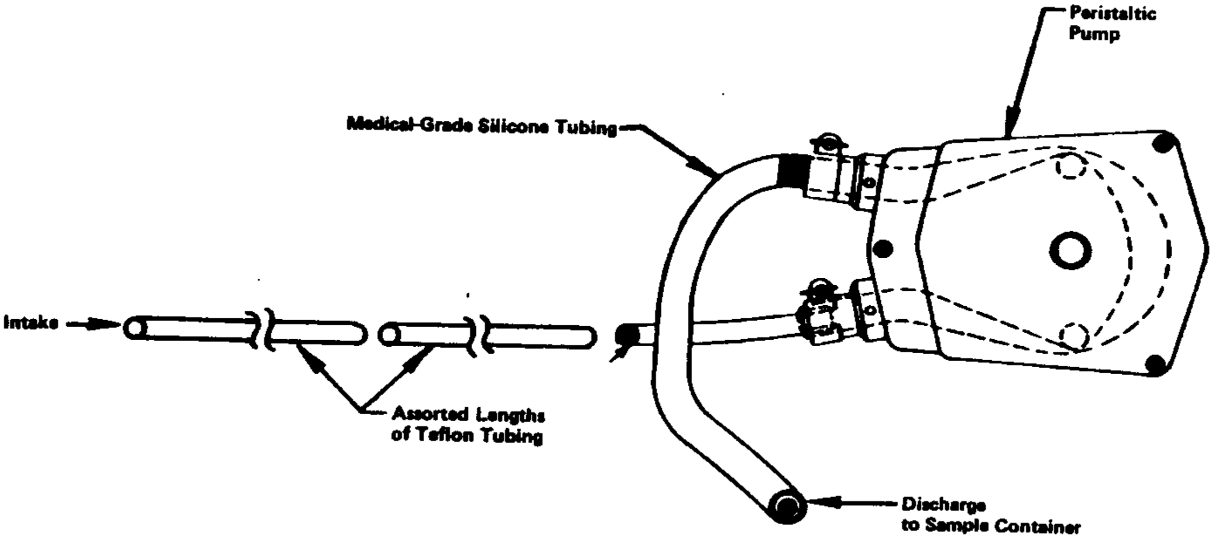
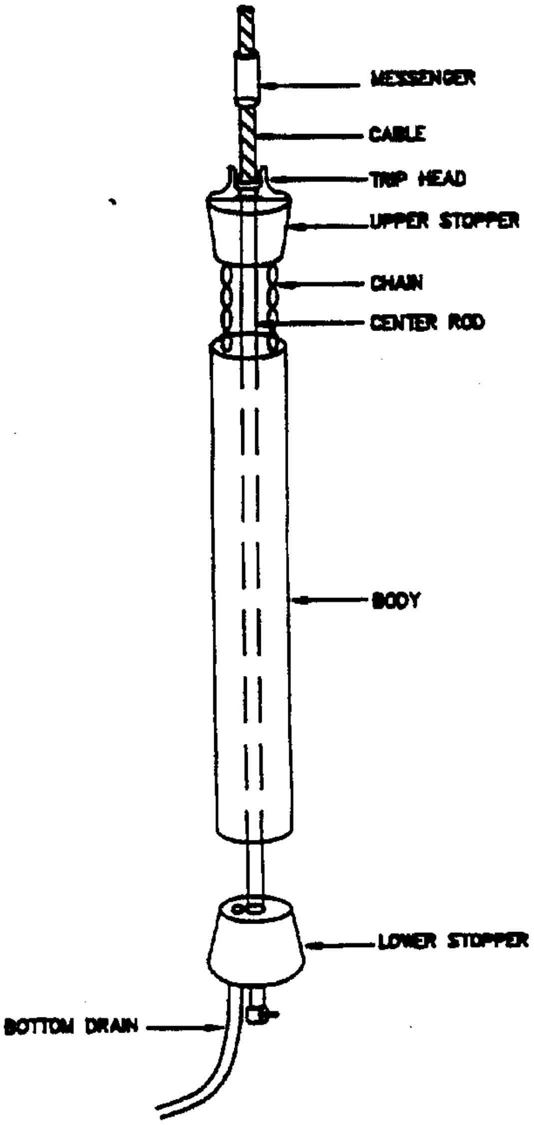


FIGURE 3

KEMMERER SAMPLER



SOP APPROVAL FORM

SulTRAC

PROJECT-SPECIFIC

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

GROUNDWATER SAMPLING

SOP NO. 010

REVISION NO. 4

Last Reviewed: August 2013

19 August 2013

Quality Assurance Approved

Date

1.0 BACKGROUND

Groundwater sampling may be required for a variety of reasons, such as to examine potable or industrial water supplies, check for and track contaminant plume movement in the vicinity of a land disposal or spill site, conduct Resource Conservation and Recovery Act (RCRA) compliance monitoring, or examine a site where historical information is minimal or nonexistent, but where groundwater may be contaminated.

Groundwater is usually sampled through an in-place well, either temporarily or permanently installed. SOP No. 020 (Monitoring Well Installation) provides guidance for installing new monitoring wells. However, groundwater can also be sampled anywhere it is present, such as in a pit or a dug or drilled hole.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for determining the quality of groundwater entering, leaving, or affected by site activities through groundwater sampling. The samples are obtained by retrieving water from a well screened in the aquifer or aquifers underlying a site.

1.2 SCOPE

This SOP provides general guidance for groundwater sampling activities conducted in the field. SOP No. 015 (Groundwater Sample Collection Using Micropurge Technology) provides additional specific guidance for using low-flow methods to collect groundwater samples.

1.3 DEFINITIONS

Bailer: A cylindrical sampling device with valves on either end, used to extract water from a well. Bailers are usually constructed of an inert material such as stainless steel or polytetrafluoroethylene (Teflon). The bailer is lowered and raised by means of a cable that may be cleaned and reused, or by disposable rope.

Electrical Water Level Indicator: An electrical device that has a light or sound alarm connected to an open circuit, used to determine the depth to liquid. The circuit is closed when the probe intersects a conducting liquid. The wire used to raise and lower the probe is usually graduated.

Immiscible Phase: A liquid phase that cannot be uniformly mixed or blended with water. Heavy immiscible phases sink, and light immiscible phases float on water.

Interface Probe: An electrical probe that determines the distance from the surface to air-water, air-immiscible, or immiscible-water interfaces.

Purge Volume: The volume of water that needs to be removed from the well prior to sampling to ensure that the sample collected is representative of the formation groundwater.

Riser Pipe: The length of well casing above the ground surface.

Total Well Depth: The distance from the reference measuring point (top of well casing or ground surface) to the bottom of the well.

Water Level: The level of water in a well, measured as depth to water or as elevation of water, relative to a reference mark or datum.

1.4 REFERENCES

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1.5 REQUIREMENTS AND RESOURCES

There are various options available to obtain groundwater samples. The procedures are outlined in the following section. The equipment needed to accomplish these procedures includes the following:

- Organic vapor detector with a flame ionization detector (FID) or a photoionization detector (PID)
- Pipe wrench and/or other tools needed to open monitoring wells (socket wrench, mallet, etc.)
- Electrical water level indicator or interface probe
- Purging device (type needed depends on well depth, casing diameter, and type of sample desired; see sampling devices below)
- Graduated cylinder or bucket
- Sampling device (type needed depends upon depth to water and type of sample desired)
    - Teflon bailer
    - Stainless steel bailer
    - Teflon bladder pump
    - Stainless steel submersible (non-oil-bearing) pump
    - Existing dedicated equipment
    - Peristaltic pump
- Sample containers
- Wastewater containers
- Field logbook
- Stopwatch

Additional equipment is required to complete measurement of field parameters (for example, pH, specific conductance, and temperature) of the groundwater in the well. Refer to SulTRAC project-specific SOP 061 (Field Measurement of Dissolved Oxygen, Oxidation-Reduction Potential, pH, Specific Conductance, Temperature, and Turbidity Using a Multi-Parameter Water Quality Meter) or individual field parameter SOPs as appropriate.

2.0 PROCEDURE

Prior to sampling, a site-specific sampling plan should be developed. The plan should take into consideration the site characteristics and should include:

- Specific, repeatable well measurement techniques and reference points for determining the depth to water and the depth to the bottom of the well
- Specific method of purging and selection of purging equipment
- Specific methods and equipment for measurements of field parameters
- Specific method of sample collection and the sampling equipment that will be used
- Specific parameters for which samples will be analyzed
- Order in which sample bottles will be filled, based on the analytical parameters

The following sections discuss procedures for approaching the well, establishing a sample preparation area, making preliminary well measurements, purging the well, and collecting samples.

2.1 APPROACHING THE WELL

In general, all wells should be assumed to pose a health and safety risk until field measurements indicate otherwise. Approach the well from the upwind side. Record the well appearance and the general condition of the protective casing, surface seal, and surrounding area in the logbook.

Once at the well, the lead person should systematically use the organic vapor detector to survey the immediate area around the well (from the breathing zone to the top of the casing to the ground). If elevated FID and PID meter readings are encountered, retreat to a safe area and instruct the sampling team to either let vapors dissipate and approach the well again or put on the appropriate level of personal protective equipment (PPE), as specified in the site-specific health and safety plan. See SOP No. 003 (Organic Vapor Air Monitoring) for additional air monitoring guidance.

Some monitoring wells have the potential to contain pressurized headspace—for example, through the generation of gases from contaminated groundwater, due to biological processes, degradation of contaminants, or simply based on location such as near a landfill or in areas that intersect lithological abnormalities; or through intentional artificial means such as those associated with air sparging systems. Injection or extraction wells may be artificially pressurized and may remain so for several days after the system has been turned off. This presents a hazard to people opening these wells. SulTRAC personnel shall employ the following practices to minimize these hazards:

- Wear safety glasses to protect the eyes. If site-specific observations and conditions indicate that the wells may be pressurized, wear a full-face shield over the safety eye protection.
- Do not place the face or any other part of the body over the well when opening because this may place you in a strike zone.
- Open the well cover at arm's length, then step away and allow the well to off gas and stabilize.

Upon opening the well casing, the lead person should systematically survey inside the well casing, above the well casing in the breathing zone, and in the immediate area around the well. If elevated FID or PID meter readings in the breathing zone are encountered (see health and safety plan for action levels), retreat and put on appropriate PPE. It is important to remember that action levels are based on readings in the breathing zone, not within the well casing. Representative organic vapor detector readings should be recorded in the logbook. Also look out for insects or other animals that may nest in well openings, and refer to the health and safety plan for specific hazards.

2.2 ESTABLISHING A SAMPLE PREPARATION AREA

The sample preparation area is generally located upwind or to either side of the well. If elevated readings are encountered using an organic vapor detector, this area should be taped off and the sample preparation area should be located upwind of the well, where ambient conduction are measured.

2.3 MAKING PRELIMINARY WELL MEASUREMENTS

Several preliminary well measurements should be made prior to initiating sampling of the well. These include determining water level and total well depth measurements, determining the presence of immiscible phases, and calculating purge volumes. All preliminary measurements will be recorded in the logbook or the Groundwater Sampling Data Sheet (included as an exhibit to this SOP) as they are determined. SOP No. 014 (Static Water Level, Total Well Depth, and Immiscible Layer Measurement) provides additional information concerning these preliminary measurements.

2.3.1 Water Level and Total Well Depth Measurements

SulTRAC typically uses an electric water level indicator for water level and total well depth measurements. This device sounds an alarm or triggers a light when the measuring probe touches the water surface, thus closing an electrical circuit. The electric cable supporting the probe is usually graduated to 0.01 foot and can be read at the well site directly. The distance between the static water level and the marked or notched location at the top of the riser pipe is measured. The height of the riser pipe above ground surface, as obtained from well location survey data, is then subtracted from the total reading to give the depth to static water. To improve accuracy, three separate readings should be made, and the values averaged. This helps to eliminate any errors due to kinks or bends in the cables, which may change in length when the water level indicator is raised and lowered.

The total well depth can be measured by lowering the probe into the well until resistance is met, indicating that the probe has reached the bottom of the well. The total well depth is then read to the 0.01-foot fraction. The distance between the bottom of the well and the marked or notched location on the riser pipe is measured. The height of the riser pipe above the ground surface, as obtained from well survey data, is then subtracted from the total reading to give the depth to the bottom of the well. To improve accuracy, three separate readings should be made, and the readings averaged.

2.3.2 Determining if Immiscible Nonaqueous-Phases Liquids are Present

If immiscible, nonaqueous-phase liquid (NAPL) are present, the following measurement activities should be undertaken. Organic liquids are measured by lowering an interface probe slowly to the surface of the liquid in the well. When the audible alarm sounds, record the depth. If the alarm is continuous, a floating immiscible layer has been detected. To determine the thickness of this layer, continue lowering the probe slowly until the alarm changes to an oscillating signal. The oscillating signal indicates that the probe has detected an aqueous layer. Record this depth as the depth to water and determine the thickness and the volume of the immiscible layer.

Continue lowering the probe into the well to determine if dense immiscible phases (sinkers) are present. If the alarm signal changes from oscillating to a continuous sound, a heavier immiscible layer has been detected; record this depth.

Continue lowering the probe to the bottom of the well and record the total depth. Calculate and record the sinker phase volume and total water volume in the well (see equation in Section 2.3.3). If immiscible phases are present, immediately refer to Section 2.5.3 or 2.5.4 of this SOP for sample collection procedures.

2.3.3 Determination of Purging Volume

If the presence of immiscible phases does not need to be determined, determine the depth to water and the total depth of the well as described in Section 2.3.1. Once these measurements have been made and recorded, use Table 1 to calculate the total volume of water in the well. Multiply this volume by the purging factor to determine purging volume. The minimum purging factor is typically three casing volumes but may be superseded by site-specific program requirements, individual well yield characteristics, or stabilization of field parameters measured during purging. Field parameters (for example, pH, specific conductance, and temperature) should be measured prior to purging and after each well volume. All field parameter data should be recorded in the field logbook, Groundwater Sampling Data Sheet, or personal digital assistant (PDA). Refer to specific SulTRAC SOP 061 as appropriate for more detailed procedures for determining these field parameters.

The volume of water in the well is based on the following formula:

$$V = \pi r^2 h \times 7.48$$

where

V = static volume of water in the well (gallons)
r = inside radius of the well (feet)
h = length of water in the well (total well depth minus depth to water) (feet)
7.48 = conversion factor (cubic feet to gallons)

Common well sizes and corresponding volumes are as follows:

- 1-inch well = h x 0.041 gal/ft
- 2-inch well = h x 0.163 gal/ft
- 3-inch well = h x 0.367 gal/ft
- 4-inch well = h x 0.652 gal/ft

2.4 PURGING THE WELL

Currently, SulTRAC standards allow for six options for purging wells:

1. Teflon bailers

2. Stainless steel bailers
3. Teflon bladder pumps
4. Stainless steel submersible (non-oil-bearing) pumps
5. Existing dedicated equipment
6. Peristaltic pumps (these devices are for shallow wells only)

As previously stated, the minimum purging volume is typically three casing volumes. Exceptions to this standard may be made in the case of low-yield wells. When purging low-yield wells, purge the well until the water level is equal to the top of screen elevation (if possible). Samples should be collected no sooner than 2 hours after purging and when sufficient groundwater volume is available.

The well should be purged until measured field parameters have stabilized. If any field parameter has not stabilized, additional purging should be performed. To be considered stable, field parameters should change by no more than the stabilization criteria listed on Table 1 between each well volume purged. If the above conditions have not been met after a specified period of time, purging will be considered complete and sampling can begin. Refer to the field sampling plan or quality assurance project plan for specified time period. Record the final well stabilization parameters on the Groundwater Sampling Data Sheet, and indicate if the well purging was considered complete due to stabilized parameters or exceeding the specified period of time.

At no time should the purging rate be high enough to cause the groundwater to cascade back into the well, as this could result in excessive aeration and potential stripping of volatile constituents.

The actual volume of purged water can be measured using several acceptable methods:

- When bailers are used, the actual volume of each bailer’s contents can be measured using a calibrated bucket.
- If a pump is used for purging, the pump rate can be determined by using a bucket of known volume, stopwatch, and the duration of pumping time necessary to purge the known volume.

2.5 SAMPLE COLLECTION

This section first describes general groundwater sample collection procedures. This section also describes procedures for collecting groundwater samples for volatile organic analysis (VOA) and for collecting samples when light or heavy immiscible layers are present in a monitoring well. Samples of light and heavy immiscible layers should be collected before the well is purged. Site-specific sampling plans may indicate that, based on the presence of NAPL, no groundwater sample is to be collected.

2.5.1 General Groundwater Sampling Procedures

The technique used to withdraw a groundwater sample from a well should be selected based on the parameters for which the sample will be analyzed. To ensure that the groundwater samples are representative, it is important to avoid physically altering or chemically contaminating the sample during collection, withdrawal, or containerization. If the samples are to be analyzed for volatile organic compounds, it is critical that air does not become entrained in the water column.

Acceptable sampling devices for all parameters are double check valve stainless steel or Teflon bailers, bladder pumps, low-flow positive displacement pumps, or for shallow wells, peristaltic pumps. Additional measurements of field parameters should be performed at the time of sampling.

In some cases, it may become necessary to use dedicated equipment already in the well to collect samples. This is particularly true of high-volume, deep wells (>150 feet) where bladder pumps are ineffective and bailing is impractical. If existing equipment must be used, however, determine the make and model of the pump and obtain information on component construction materials from the manufacturer or facility representatives. If an existing pump is to be used for sampling, make sure the flow volume can be reduced so that a reliable VOA sample can be taken. Record the specific port, tap, or valve from which the sample is collected. If nondedicated sampling equipment is used, the least contaminated wells should be purged and sampled first and most contaminated wells should be purged and sampled last (if past sampling data are available to make this determination).

General sampling procedures are as follows:

- Clean sampling equipment should not be placed directly on the ground. Use a plastic drop cloth or feed line from clean reels. Never place contaminated lines back on reels.
- Check the operation of the bailer check valve assemblies to confirm free operation.
- If the bailer cable is to be decontaminated and reused, it must be made of Teflon-coated stainless steel.
- Lower sampling equipment slowly into the well to avoid degassing the water and damaging the equipment.

- Pump flow rates should be adjusted to eliminate intermittent or pulsed flow. The settings should be determined during the purging operations.
- A separate sample volume should be collected to measure necessary field parameters. Samples should be collected and containerized following procedures outlined in the project-specific field sampling plan (FSP), quality assurance project plan (QAPP), and in the order of the parameters' volatilization sensitivity. Table 2 lists the preferred collection order for common groundwater parameters.

Intermediate containers should never be used to prepare VOA samples and should be avoided for all parameters in general. All VOA containers should be filled at a single sampling point or from a single bailer volume. Also refer to site-specific sampling plan for other sample handling requirements that may be unique to a site or to specific chemical constituents.

2.5.2 Collection of Volatile Organics Samples

This section discusses in detail the collection of samples for VOA using either a bailer or bladder pump. Other pumps (such as positive displacement or peristaltic) can be used. The following factors are critical to the collection of representative samples for VOA: ensuring that no air has become entrained in the water column, achieving low pump flow rates (less than 100 milliliter [mL] per minute, if possible), avoiding flow surges, and adjusting sample preservatives if they are found to cause reactions with the sample.

2.5.2.1 Collection with Bailers

Samples for VOA should be collected from the first bailer removed from the well after purging is complete. The most effective means requires two people. One person should retrieve the bailer from the well and pour its contents into the appropriate number of 40-mL VOA vials held by the second person. Each vial should be capped and inverted to check if any air bubbles are present. If a bubble exists, unscrew the cap and add more water, or discard and repeat if vials are not pre-preserved. If bubbling persists in a vial containing acid preservative, the sample may need to be collected without the preservative and the laboratory notified to add preservative upon receipt. The sample should be transferred from the bailer to the sample container in a manner that will limit the amount of agitation in order to reduce the loss of volatile organics from the sample.

Always fill VOA vials from a single bailer volume. If the bailer is refilled, samples cannot be considered duplicates or splits.

2.5.2.2 Collection with a Bladder Pump (Well Wizard)

To successfully perform VOA sampling with a Well Wizard bladder pump, the following steps must be completed:

1. Following manufacturer's directions, activate the pump. Full water flow from the discharge tubing will begin after 5 to 15 pumping cycles. These initial pumping cycles are required to purge air from the pump and discharge tubing. The discharge and recharge settings must be manually set and adjusted to pump at optimum flow rates. To activate the bladder, it is best to set the initial cycle at long discharge and recharge rates.

2. Reduce water flow rate for VOA sample collection. To reduce the water flow rate, turn the throttle control valve (located on the left side of the Well Wizard pump control panel) counterclockwise.

3. Collect VOA sample from discharge tubing. VOA vials must be placed beneath the discharge tubing while avoiding direct contact between the vials and the tubing. Never place tubing past the mouth of the VOA vial. The pump throttle control must be turned as necessary to maintain a trickle of water in order to obtain a meniscus in the vial.

4. Continue with non-VOA sampling. Increase pump flow rate by turning the throttle control knob clockwise.

2.5.3 Sampling of Light Immiscible Floaters

The approach used when collecting a sample of a floating layer depends on the depth to the floating layer and the thickness of that layer. If the thickness of the floater is 2 feet or greater, a bottom-filling valve bailer should be used. Slowly lower the bailer until contact is made with the floater surface, and lower the bailer to a depth less than that of the floater-water interface depth as determined by preliminary measurements with the interface probe.

When the thickness of the floating layer is less than 2 feet and the depth to the surface of the floating layer is less than 15 feet, a peristaltic pump can be used to extract a sample.

When the thickness of the floating layer is less than 2 feet and the depth to the surface of the floating layer is beyond the effective "lift" of a peristaltic pump (greater than 25 feet), a bailer can be modified to allow filling from the top only (an acceptable alternative is to use a top- loading Teflon or stainless-steel bailer). Disassemble the bailer's bottom check valve and insert a piece of 2-inch-diameter Teflon sheet between the ball and ball seat. This will seal off the bottom valve. Remove the ball from the top check valve, thus allowing the sample to enter from the top. To overcome buoyancy when the bailer is lowered into the floater, place a length of 1-inch stainless steel pipe on the retrieval line above the bailer (this pipe may have to be notched to allow sample entry if the pipe remains within the top of the bailer). As an alternative, use a top-loading stainless-steel bailer. Lower the device, carefully measuring the depth to the surface of the floating layer, until the top of the bailer is level with the top of the floating layer. Lower the bailer an additional one-half thickness of the floating layer and collect the sample. This technique is the most effective method of collection if the floating layer is only a few inches thick.

2.5.4 Sampling of Heavy Immiscible Sinkers

The best method for collecting a sample of a sinker is the use of a double check valve bailer. The key to sample collection is controlled, slow lowering and raising of the bailer to and from the bottom of the well. Sample collection methods are equivalent to those described in Section 2.5.3 above.

TABLE 1

STABILIZATION CRITERIA FOR WATER QUALITY PARAMETERS

Parameter	**Stabilization Criterion**
pH	± 0.1 units
Specific Conductance	± 3 percent
Oxidation-Reduction Potential	± 10 millivolts
Turbidity	± 10 percent (when greater than 10 nephelometric turbidity units [NTU])
Dissolved Oxygen	± 0.3 milligrams per liter or ± 10 percent

TABLE 2

ORDER OF PREFERRED SAMPLE COLLECTION

1. VOA
2. Purgeable organic halogens (POX)
3. Total organic halogens (TOX)
4. Cyanide
5. Extractable organics
6. Purgeable organic carbon (POC)
7. Total metals
8. Dissolved metals
9. Total organic carbon (TOC)
10. Phenols
11. Sulfate and chloride
12. Nitrate and ammonia
13. Radionuclides

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EXHIBIT: GROUNDWATER SAMPLING DATA SHEET

Page ________ of ________

Date ________

Well Name ____________________________

Project ____________________________

Screen Interval ____________________________

Station Elevation ________ GND ____ TOC ______

Immiscible Phases Present Yes No

Project No. ____________________________

Well Location ____________________________

Sample Date ____________________________

Sampling Personnel ____________________________

____

____

Sample ID ____________________________

Duplicate ID ____________________________

Static Water Level (from TOC) ____________________

Well Stick Up ____________________________

Static Elevation ____________________________

Well Depth ____________ MEAS ____ RPTD ______

Feet of Water ____________________________

Gallons/Foot ____________________________

Casing Volume ____________________________

Type ____________________________

Measured with ____________________________

PID Readings (background) ________________

PID Reading (TOC) ____________________

Wells Installed by ____________________

Installation Date ____________________

Development Date(s) ________________

FIELD CHEMISTRY CALIBRATIONS

Date/Time ____________________________

Spec. Conductance: Standard µmhos/c Reading µmhos/cm at °C

pH: pH 4.00 - _______ at _________ °C

pH 7.00 - _______ at _________ °C

pH 10.00 - ______ at _______ °C

Slope ______________

Dissolved Oxygen: D.O. Meter _______ mg/L at _______°C

PID: Calibration Gas ___________ PPM _______ Span _______ Reading ___

PURGING

Time	Discharge Rate (mL/min)	Dissolved Oxygen (mg/L)	pH	Eh/ORP (mV)	Temp. (°C)	Specific Conduct. (µmhos/cm at °C)	Turbidity (NTU)	Cumulative Volume of Water Removed (Purged)		PID/OVA Reading		Depth to Water (ft)	Comments
								Gallons	Casing Vol.	Location	Value		

SAMPLE PARAMETERS

Condition of well: ____

Remarks: ____

SOP APPROVAL FORM

ST SulTRAC

PROJECT-SPECIFIC

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

STATIC WATER LEVEL, TOTAL WELL DEPTH, AND IMMISCIBLE LAYER MEASUREMENT

SOP NO. 014

REVISION NO. 1

Last Reviewed: August 2013

John

Quality Assurance Approved

19 August 2013

Date

1.0 BACKGROUND

Measurement of static water level, total well depth, and any immiscible layers is necessary before a well can be sampled and groundwater flow direction can be determined. If an immiscible layer is present, its depth and thickness must be determined. In addition, the static water level and total depth of a monitoring well are often needed to determine a purging volume prior to sampling.

1.1 PURPOSE

This standard operating procedure (SOP) provides guidelines for field personnel measuring: (a) static water levels, (b) total water depths, and (c) immiscible layers in monitoring wells or piezometers.

1.2 SCOPE

This SOP describes the methodologies for measuring static water level, total well depth, and immiscible layer depth and thickness.

1.3 DEFINITIONS

Electrical Water Level Meter: An electrical probe used to determine the depth to fluid. The probe has a light or sound alarm connected to an open circuit. The circuit is closed and the alarm is activated when the probe contacts a conducting fluid such as water.

Immiscible Layer: A liquid phase that cannot be uniformly mixed or blended with water. Heavy immiscible phases or dense nonaqueous-phase liquids (DNAPL) sink in water; light immiscible phases or light nonaqueous-phase liquids (LNAPL) float on water.

Interface Probe: An electrical probe used to determine the presence and thicknesses of LNAPL or DNAPL in the water column of a monitoring well.

Ionization Detector: A photoionization detector (PID) or a flame ionization detector (FID) is used to measure the level of volatile organic compounds (VOC) in the gaseous phase. Ionization detectors are generally not compound-specific and thus measure only total volatile organic compounds. A PID generally cannot detect as complete a range of compounds as the FID. This difference is the result of the relative ionization energies of the two detectors. Most PIDs cannot detect methane, but FIDs can. The

Photovac and Minirae are examples of PIDs; the Foxboro organic vapor analyzer (OVA) is an example of an FID.

Static Water Level: The level of water in a monitoring well or piezometer left open and allowed to stabilize by equilibrating to existing atmospheric pressure prior to measuring. The static water level can be measured as the depth to water or as the elevation of water relative to a reference mark or datum.

Total Well Depth: The distance from the ground surface to the bottom of a monitoring well or piezometer

1.4 REFERENCES

Tetra Tech EM Inc. 2009. SOP No. 002, General Equipment Decontamination

U.S. Environmental Protection Agency (USEPA). 2013. "Groundwater Level and Well Depth Measurement." Region 4, Field Branches Quality System and Technical Procedures. January 29. http://www.epa.gov/region4/sesd/fbqstp/Groundwater-Level-Measurement.pdf

U.S. EPA. 2000. "Manual Water Level Measurement." Environmental Response Team. SOP #2043 (Rev. #0.0, 02/11/00). http://www.dem.ri.gov/pubs/sops/wmsr2043.pdf

1.5 REQUIREMENTS AND RESOURCES

The equipment required for measuring static water levels, total well depths, and immiscible layers is as follows:

- Electrical water level meter
- Interface probe
- PID or FID
- Decontamination supplies (wash and rinse solutions, buckets, paper towels, etc.)
- Keys or wrenches for well locks and well caps
- Light source such as a flashlight or mirror to inspect inside well vaults

- Appropriate level of personal protective equipment (PPE) as specified in the site-specific health and safety plan
- Appropriate signage and traffic control to protect personnel during monitoring activities

2.0 PROCEDURES

This section provides general guidance followed by specific procedures for measurement of static water level, total well depth, and immiscible layer.

Procedures for measuring the depth to water and the depth to the bottom of a monitoring well should be identified in the planning stage of field work. Also at this stage, measuring devices should be chosen, and an individual should be assigned to take and record measurements.

All measurement instruments should be decontaminated before and after use and between measurement locations. Refer to SOP No. 002, General Equipment Decontamination. In addition, as a general rule wells with the lowest known contaminant concentrations should be measured first followed by progressively more contaminated wells. This technique helps avoid cross-contamination from a more contaminated water source to a less contaminated water source resulting in inaccurate or false sample analytic results.

Before initiating any measuring activities, the ambient air at a monitoring well head should be monitored for possible emissions of VOCs using a PID or an FID. The site-specific health and safety plan for on-site activities should provide action levels and the rationale for selection of the appropriate ionization detector.

The sampling team should wear appropriate respiratory protection equipment when necessary. The sampling team should approach wells from the upwind side and systematically survey the inside of the well casing, the area from the casing to the ground, the area from above the well casing to the breathing zone, and the area around the well for VOCs. VOC monitoring for comparison to action levels should occur in the breathing zone(s) rather than from within the well casing. If PID or FID readings of VOCs are above action levels, the sampling team should retreat to a safe area and determine if engineering controls, increased PPE, or both are necessary to reduce exposure to VOCs above action levels listed in the site-specific health and safety plan.

Sampling team should be careful when opening a well to check for insects or reptiles within the protective casing and use appropriate work gloves when reaching inside. The site-specific health and safety plan should be consulted for information on insects or reptiles that may be present in the project area.

2.1 STATIC WATER LEVEL MEASUREMENT

An electric water level meter is typically used to measure static water levels. The electrical probe of the meter is lowered into the monitoring well until the light or sound alarm is activated indicating the probe has touched the water surface. To ensure accuracy, the well or piezometer should be opened to allow groundwater in the well to equilibrate with atmospheric pressures before measuring depth to water. Ideally, static groundwater measurements will be collected prior to purging or sampling activities. The static water level is read directly from the graduated tape on the meter to the nearest 0.01-foot. Two to three measurements should be taken over several minutes to ensure water levels are not fluctuating. If water levels continue to fluctuate, the groundwater has not equilibrated or become static. Measurements should continue until static water levels readings are obtained before recording a final measurement. If the monitoring well top is not flush with the ground surface, the distance between the static water level and the top of the riser pipe should be measured; the height of the riser pipe above ground surface should then be subtracted from the first measurement to determine the depth to static water below ground surface. Surveyed elevations should be used if available to establish the water level elevation. The well number, measurement date and time, and individual readings should be recorded in a field logbook.

2.2 TOTAL WELL DEPTH MEASUREMENT

Total well depth can also be measured using an electric water level meter. The electrical probe of the indicator is lowered into the monitoring well until resistance is met, indicating that the probe has reached the bottom of the well. During measurement, the probe should be positioned in the well so that resistance is met, but there is no slack in the measuring tape. The total well depth is read directly from the graduated tape on the indicator to the nearest 0.01-foot. Measurements become less accurate with increased depth as the length of measuring tape deployed increases and the distance under the water column increases making it more difficult to determine if the bottom of the well has reached. In this circumstance, a weighted water level meter or measuring tape may be necessary to allow the user to “feel” the bottom of the well. Care should be taken to ensure the use of only inert and chemical-free weighted devices such as stainless steel are used. If the monitoring well top is not flush with the ground surface, the distance between the bottom of the well and the top of the riser pipe should be measured; the height of the riser pipe above ground surface should then be subtracted from the first measurement to determine the depth from ground surface to the bottom of the well. The well number, measurement date and time, and individual readings should be recorded in a field logbook.

2.3 IMMISCIBLE LAYER DETECTION AND MEASUREMENT

A LNAPL immiscible layer in a monitoring well can be detected by slowly lowering an interface probe to the surface of the water in the well. When the audible alarm sounds, the depth of the probe should be recorded. If the alarm is continuous, a light immiscible layer has been detected. To measure the thickness of this layer, the probe should then be slowly lowered until the alarm changes to an oscillating signal. The oscillating alarm indicates that the probe has reached a water layer. The probe depth at the time the alarm begins oscillating should be recorded as the depth to water. The thickness of the light immiscible layer should then be determined by subtracting the depth at which a continuous alarm occurred from the depth at which the alarm began to oscillate. The well number, measurement date and time, individual readings for depth and thickness, and average values for depth and thickness should be recorded in a field logbook.

To determine whether a DNAPL immiscible layer is present, the interface probe is lowered into the monitoring well and allowed to slowly drop below the water causing an oscillating alarm. If the alarm changes from an oscillating to a continuous signal, a heavier immiscible layer has been detected, and the probe depth should be recorded at that point. Total well depth obtained in Section 2.2 should be used for calculating the thickness of the DNAPL layer within the well casing. The DNAPL layer is then calculated by subtracting the depth at which the alarm became continuous from the total well depth. This procedure provides an estimate of the thickness of the DNAPL layer in the monitoring well. The well number, measurement date and time, and individual readings for depth and thickness should be recorded in a field logbook.

SOP APPROVAL FORM

SulTRAC

PROJECT-SPECIFIC

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

RECORDING NOTES IN FIELD LOGBOOKS

SOP NO. 024

REVISION NO. 1

Last Reviewed: December 2013

John Diego

Quality Assurance Approved

04 December 2013

Date

1.0 BACKGROUND

The field logbook should contain detailed records of all the field activities, interviews of people, and observations of conditions at a site. Entries should be described in as much detail as possible so that personnel can accurately reconstruct, after the fact, activities and events during their performance of field assignments. Field logbooks are considered accountable documents in enforcement proceedings and may be subject to review. Therefore, the entries in the logbook must be accurate and detailed; and they must reflect the importance of the field events.

1.1 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide guidance to ensure that logbook documentation for any field activity is correct, complete, and adequate. Logbooks are used for identifying, locating, labeling, and tracking samples. A logbook should document any deviations from the project approach, work plans, quality assurance project plans, health and safety plans, sampling plans, and any changes in project personnel. They also serve as documentation of any photographs taken during the course of the project. In addition, the data recorded in the logbook may assist in the interpretation of analytical results. A complete and accurate logbook also aids in maintaining good quality control. Quality control is enhanced by proper documentation of all observations, activities, and decisions.

1.2 SCOPE

This SOP establishes the general requirements and procedures for recording notes in the field logbook.

1.3 DEFINITIONS

None

1.4 REFERENCES

Compton, R.R. 1985. *Geology in the Field*. John Wiley and Sons. New York, N.Y.

1.5 REQUIREMENTS AND RESOURCES

The following items are required for field notation:

- Field logbooks
- Ballpoint pens with permanent ink
- 6-inch ruler (optional).

Field logbooks should be bound (sewn) with water-resistant and acid-proof covers; they should have preprinted lines and wide columns. They should be approximately 7 ½ by 4 ½ inches or 8 ½ by 11 inches in size. Loose-leaf sheets are not acceptable for field notes. If notes are written on loose paper, they must be transcribed as soon as possible into a regular field logbook by the same person who recorded the notes.

Logbooks can be obtained from an individual’s office supply room or directly from outside suppliers. Logbooks must meet the requirements specified in this SOP and should include preprinted pages that are consecutively numbered. If the numbers must be written by hand, the numbers should be circled so that they are not confused with data.

2.0 PROCEDURES

The following subsections provide general guidelines and formatting requirements for field logbooks, and detailed procedures for completing field logbooks.

2.1 GENERAL GUIDELINES

- A separate field logbook must be maintained for each project. If a site consists of multiple subsites, designate a separate logbook for each subsite. For special tasks, such as periodic well water-level measurements, data from multiple subsites may be entered into one logbook that contains only one type of information.

- All logbooks must be bound and contain consecutively numbered pages.

- No pages can be removed from the logbook for any purpose.

- All field activities, meetings, photographs, and names of personnel must be recorded in the site logbook.

- Each logbook pertaining to a site or subsite should be assigned a serial number based on the date the logbook is issued to the project manager. The first issued logbook should be assigned number 1, the next issued logbook assigned number 2, and so on. The project manager is to maintain a record of all logbooks issued under the project.

- All information must be entered with a ballpoint pen with waterproof ink. Do not use pens with “wet ink,” because the ink may wash out if the paper gets wet. Pencils are not permissible for field notes because information can be erased. The entries should be written dark enough so that the logbook can be easily photocopied.

- Do not enter information in the logbook that is not related to the project. The language used in the logbook should be factual and objective.

- Begin a new page for each day’s notes.

- Write notes on every line of the logbook. If a subject changes and an additional blank space is necessary to make the new subject title stand out, skip one line before beginning the new subject. Do not skip any pages or parts of pages unless a day’s activity ends in the middle of a page.

- Draw a diagonal line on any blank spaces of four lines or more to prevent unauthorized entries.

2.2 LOGBOOK FORMAT

The layout and organization of each field logbook should be consistent with other field logbooks. Guidelines for the cover, spine, and internal pagination are discussed below.

2.2.1 FORMAT OF FIELD LOGBOOK COVER AND SPINE

Write the following information in clear capital letters on the front cover of each logbook using a Sharpie® or similar type permanent ink marker:

- Logbook identification number

- The serial number of the logbook (assigned by the project manager)

- Name of the site, city, and state

- Name of subsite if applicable

- Type of activity
- Beginning and ending dates of activities entered into the logbook
- SulTRAC, City and State
- “REWARD IF FOUND”

Some of the information listed above, such as the list of activities and ending dates, should be entered after the entire logbook has been filled or after decision that the remaining blank pages in the logbook will not be filled.

The spine of the logbook should contain an abbreviated version of the information on the cover: for example, “1, Col. Ave., Hastings, 5/12 - 8/12.”

2.2.2 First Page of the Field Logbook

Spaces are usually provided on the inside front cover (or the opening page in some logbooks), for the company name (SulTRAC), address, contact name, and telephone number. If preprinted spaces for this information are not provided in the logbook, write the information on the first available page.

2.3 ENTERING INFORMATION IN THE LOGBOOK

Enter the following information at the beginning of each day or whenever warranted during the course of a day:

- Date
- Starting time
- Specific location
- General weather conditions and approximate temperature
- Names of personnel present at the site, noting the affiliation(s) and designation(s) of all personnel

- Equipment calibration and equipment models used
- Changes in instructions or activities at the site
- Levels of personal protective clothing and equipment
- A general title of the first task undertaken (for example, well installation at MW-11, decontamination at borehole BH-11, groundwater sampling at MW-11)
- Approximate scale for all diagrams. If this can’t be done, write “not to scale” on the diagram. Indicate the north direction on all maps and cross-sections. Label features on each diagram.
- Corrections, if necessary, including a single line through the entry being corrected. Initial and date any corrections made in the logbook.
- After last entry on each page, initials of the person recording notes. No information is to be entered in the area following these initials.
- At the end of the day, signature of the person recording notes and date at the bottom of the last page. Indicate the end of the work day by writing “Left site at (time).” A diagonal line must be drawn across any remaining blank space at the bottom of this last page.

The following information should be recorded in the logbook after taking a photograph:

- Time, date, location, direction, and, if appropriate, weather conditions
- Description of the subject photographed and the reason for taking the picture
- Sequential number of the photograph
- Name of the photographer.

The following information should be entered into the logbook when collecting samples:

- Location description
- Name(s) of sampler(s)
- Collection time
- Designation of sample as a grab or composite sample
- Type of sample (water, sediment, soil gas, etc.)

- On-site measurement data (pH, temperature, specific conductivity)
- Field observations (odors, colors, weather, etc.)
- Preliminary sample description
- Type of preservative used
- Instrument readings

If pre-printed field data forms are available (forms such as the micropurge field data collection form), data should be entered on these pre-printed forms rather than into field logbooks. Note in the logbook that the field data are recorded on separate forms.

2.4 PRECAUTIONS

Custody of field logbooks must be maintained at all times. Field personnel must keep the logbooks in a secure place (locked car, trailer, or field office) when the logbook is not in personal possession. Logbooks are official project documents and must be treated as such.

SOP APPROVAL FORM

ST SulTRAC

PROJECT-SPECIFIC

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

FIELD MEASUREMENT OF GROUNDWATER INDICATOR PARAMETERS

SOP NO. 061

REVISION NO. 2

Last Reviewed: August 2013

John

Quality Assurance Approved

19 August 2013

Date

1.0 BACKGROUND

Various water quality monitoring systems can be used for determining groundwater indicator parameters in the field. Commonly measured field indicator parameters include pH, specific conductance, temperature, oxidation-reduction potential (ORP), dissolved oxygen (DO) and turbidity. Groundwater field measurements are typically collected in conjunction with groundwater sampling or monitoring well development (see SOPs 010, 015, and 021).

Various types of water quality systems exist including down-hole systems and flow through cells. SulTRAC used several common water quality meters including various types of In-Situ, YSI, Hydac, and Horiba meters (see Figure 1 at the end of this SOP). The sampling team should select the type of meter or monitoring system based on site-specific conditions including data collection needs, the types of wells being sampled, and the sampling procedures used. Multiple parameter systems should be used when multiple field parameters are to be measured.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the general requirements and procedures for using various water quality monitoring systems for determining groundwater pH, specific conductance, temperature, ORP, DO and turbidity in the field.

1.2 SCOPE

This SOP applies to general procedures for calibrating and operating water quality monitoring systems in the field. The project work plan or field sampling plan should identify the types of systems to be used and the actual project-specific field parameters to be measured. For each type of water quality system, the manufacturer’s manual should be consulted for specific operating instructions.

1.3 DEFINITIONS

Single Parameter System: A meter or monitoring system consisting of a single probe designed to measure a single indicator parameter.

Multiple Parameter System: A meter or monitoring system consisting of multiple probes capable of measuring multiple indicator parameters.

Open Container Measurements: Field measurements performed in an open container such as a cup, a jar, or a bucket where an air/water interface exists.

Flow-Through Chamber or Cell: A plastic cell or chamber connected to the sample pump discharge tubing so that a continuous flow of water passes across the probes. Additional tubing is used to route water from the flow-through cell to a waste container or final discharge point.

Down-Hole Monitoring System: A meter or monitoring system where probes are submerged by inserting them into the well. The probes are attached to the meter (located at the well head or ground surface) by one or more cables.

pH: A measure of the acidity or alkalinity of a solution. The pH scale ranges from 0 to 14 with strongly acidic solutions at the low end, strongly basic solutions at the high end, and “pure” or neutral water at 7. Field measurements of pH are recorded in standard units.

Specific Conductance: The ability of a solution to conduct electricity; a measure of the solution’s ionic activity and content. The higher the concentration of ionic (dissolved) constituents, the higher the conductivity. Conductivity of the same water changes substantially with temperature. Specific conductivity is generally found to be a good measure of the concentration of total dissolved solids (TDS) and salinity. Conductivity is measured by placing two electrodes (with opposite electrical charge) in the water. For a known electrical current, the voltage drop across the electrodes reveals the solution’s resistance. Since the resistance of aqueous solution changes with temperature (resistance drops with increasing temperature), the resistance is corrected to the resistance of the solution at 25 ºC. Field measurements are recorded in units of microsiemens per centimeters (µS/cm).

Temperature: The degree of hotness or coldness of the solution being measured. Field measurements are typically recorded in degrees Celsius (°C).

ORP: ORP, or redox potential, is the tendency of a chemical species to acquire electrons and be reduced. In aqueous solutions, the reduction potential is the tendency of the solution to either gain or lose electrons when new chemical species are introduced. A solution with a higher (more positive) reduction potential than the new species will have a tendency to gain electrons from the new species (to be reduced by oxidizing the new species) and a solution with a lower (more negative) reduction potential will have a tendency to lose electrons to the new species (to be oxidized by reducing the new species). Just as the transfer of hydrogen ions between chemical species determines the pH of an aqueous solution, the transfer of electrons between chemical species determines the reduction potential of an aqueous solution. Like pH, the reduction potential represents an intensity factor. It does not characterize the capacity of the system for oxidation or reduction, in much the same way that pH does not characterize the buffering capacity. Field measurements are typically recorded in millivolts (mV).

DO: Dissolved oxygen (or oxygen saturation) is a relative measure of the amount of oxygen dissolved or carried in a given medium. In aquatic environments, dissolved oxygen is a relative measure of the amount of oxygen (O_2) dissolved in the water. Field measurements are typically recorded in milligrams per liter (mg/L).

Turbidity: Turbidity is the cloudiness or haziness of a fluid caused by individual particles (suspended solids). Fluids can contain suspended solid matter consisting of particles of many different sizes. While some suspended material will be large enough and heavy enough to settle rapidly to the bottom of the container if a liquid sample is left to stand, very small particles will settle only very slowly or not at all if the sample is regularly agitated or the particles are colloidal. These small solid particles cause the liquid to appear turbid. Field measurements are typically recorded in Nephelometric Turbidity Units (NTU).

1.4 REFERENCES

Essential Handbook of Ground-Water Sampling by Gillian Nielsen, 2007.

SulTRAC. August 2013. Project-Specific SOPs 010, 015, and 021.

1.5 REQUIREMENTS AND RESOURCES

The following items are typically required to measure groundwater pH, specific conductance, temperature, ORP, DO, and turbidity using this SOP:

- Single or multiple parameter water quality measuring system
- Specific conductance calibration solutions
- Buffer solutions of pH 4, 7, and 10 for pH calibration
- Distilled or deionized water
- Rinse bottle
- 50-milliliter (mL) sample cups or beakers
- Sample tubing and connectors (specific to each type of system)
- Waste container to collect purge water
- Logbook or field data sheets

2.0 PROCEDURES

The procedures outlined in this SOP are general and may apply to various types of water quality monitoring systems to measure groundwater pH, specific conductance, temperature, ORP, DO and turbidity in the field. General procedures for testing and calibrating the monitoring systems are presented first, followed by procedures for using the instruments and making field measurements. Each particular monitoring system should be identified in the project work plan or field sampling plan and should be operated in accordance with the manufacturer's instruction manual.

2.1 TESTING AND CALIBRATION PROCEDURES

Each field meter or monitoring system should be calibrated according to manufacturer's specifications. In general, equipment should be thoroughly cleaned then calibrated and tested before the start-up of sampling at each site. Equipment should be calibrated and tested using manufacturer provided solutions and standards. Care should be taken to rinse the probes between testing and calibration to prevent cross contaminating solutions. Solutions should be poured from the manufacturer's container into another container to prevent compromising the entire solution provided by the manufacturer. Calibration and testing of field equipment should be documented each time it is performed in field logbooks (or field data sheets, if applicable). If testing and calibration measurements are out of tolerance, the instrument must be serviced or repaired.

2.2 FIELD MEASUREMENT PROCEDURES

Each field meter or monitoring system should be operated according to manufacturer's specifications. The actual field procedures will vary depending on the type of monitoring system being used (open container systems, flow-through cell systems, or down-hole systems) and the types of field parameters being measured. In addition, most systems include a data logging option. A description of open container, flow-through cell, and down-hole measurement processes are discussed below, followed by a general procedural summary and a summary of common errors associated with field measurements of indicator parameters.

2.2.1 Open Container Measurements

Open container measurements consist of collecting groundwater and placing it in a cup or container for field measurements using a hand held system. This method of field measurements is commonly used when bailing wells, but can also be used when pumping wells. Prior to field measurements, the equipment must be cleaned and calibrated following manufacturer's specifications. Field measurements should then be made at the frequency and for the indicator parameters specified in the project work plan or field sampling plan. To make open container field measurements, samplers collect groundwater from the well and place in a cup or container large enough to adequately submerge the probe or probes, as specified in the manufacturer's operations manual. For open containers, measurements should be taken in the following order: temperature, specific conductance, pH, and turbidity. Open container systems are not recommended for low-flow sampling as flow-through systems are more appropriate. The probes and cup or container should be thoroughly rinsed after each field measurement and between sampling locations.

2.2.2 Flow-Through Cell Measurements

Flow-through cell systems consist of measuring groundwater parameters as a continuous flow of water passes across the probes through a cell or chamber, and is primarily used when pumping wells and using low-flow sampling procedures. Prior to field measurements, the equipment must be cleaned and calibrated following manufacturer's specifications. Field measurements should then be made at the frequency and for the indicator parameters specified in the project work plan or field sampling plan.

The flow-through cell or chamber is placed "in line" between the discharge tubing of the pump and the container used to collect purged water. The outlet from the pump must be connected to the sample chamber input. The sample chamber outlet must then be connected or routed to a waste container (or to another designated discharge point). Tubing, fittings, and adaptors are generally required and may be provided by the manufacturer. Pump discharge tubing and chamber inlets and outlets are typically 1/2 or 3/8 inch diameter.

After the cell or chamber is connected to the pump discharge tubing and waste collection container, the sensors should be inserted into the sensor mounting plate in their respective ports. Any unused sensor ports must have plugs installed to close off the sample chamber. The probe cables are then connected to the meter following manufacturer's specifications.

With the system connected, the sampler should turn on the pump according to the manufacturer's instructions and then turn on the water quality monitor. Before recording any values, the sample chamber should be full, all air should be voided, and all of the displayed values should be stable. The probes and sample chamber should be thoroughly rinsed between sampling locations.

2.2.3 Down-Hole Measurements

Down-hole measurement systems consist of inserting the probes (or a multi-parameter sensor housing) inside a well to obtain field measurements, and is primarily used when pumping wells. Prior to field measurements, the equipment must be cleaned and calibrated following manufacturer's specifications. Field measurements should then be made at the frequency and for the indicator parameters specified in the project work plan or field sampling plan.

The probes or sensor are attached to a hand held meter or control unit by a cable and lowered inside the well to be sampled. Limiting factors when using down-hole systems include probe or sensor diameters and available cable lengths. The probes should be thoroughly decontaminated between sampling locations.

2.2.4 General Procedures for Field Measurements of Indicator Parameters

The following section discusses general procedures that typically apply to making field measurements of indicator parameters using various types of field instruments. Each particular type of meter or monitoring system should be identified in the project work plan or field sampling plan and should be operated in accordance with the manufacturer’s instruction manual.

1. Inspect the instrument and batteries prior to the field effort.
2. Check the integrity of the buffer solutions used for field calibration since frequent replacement is necessary as a result of degradation upon exposure to the atmosphere.
3. If applicable, make sure all electrolyte solutions within the electrode(s) are at proper levels and no air bubbles are present within the electrode(s).
4. Calibrate the meter and electrode(s) on a daily use basis (or as recommended) following manufacturer's instructions and record data in field logbook or on field data sheets.
5. Immerse the electrode(s) in the sample. Stabilization may take several seconds to several minutes. If the parameter values continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be occurring in the sample, or the meter or electrode may be malfunctioning. The failure of the measurements to stabilize should be clearly noted in the logbook or field data sheet. For DO, provide for sufficient flow past the membrane by gently stirring the sample. Probes without stirrers placed in wells (down-hole measurements) may be gently moved up and down to achieve the required mixing.
6. Read and record the value of each parameter being measured making sure units of measure are clearly recorded.
7. Rinse the electrode(s) with deionized water.
8. Store the electrode(s) in accordance with manufacturer’s instructions

2.2.5 Common Errors or Problems Associated With Field Measurements

The project work plan or field sampling plan should clearly identify the types of parameters to be measured, the measurement frequency, and “stabilization” requirements. It is essential to ensure that the type of monitoring system selected is compatible with the monitoring well sampling or development methods to be utilized. Some common errors to avoid are identified below:

- No, or incorrect equipment calibration
- Incorrect or expired calibration standards
- Poor equipment maintenance
- Inadequate training or unfamiliarity with equipment
- No record of units of measure and "+" or "-" values for ORP
- Too much time taken to measure temperature sensitive parameters
- DO and ORP measured in closed systems (flow-through cell or down-hole) instead of closed cell systems

FIGURE 1

THE HORIBA U-10 WATER QUALITY MONITORING SYSTEM
THE IN-SITU TROLL 9500 LOW-FLOW SYSTEM
THE YSI HAND HELD 556 METER

Horiba U-10

In-Situ Troll

YSI 556

ATTACHMENT C

TESTAMERICA, INC. STANDARD OPERATING PROCEDURES

(227 Sheets)

Controlled Copy
Copy No. ____________

TESTAMERICA KNOXVILLE
SOP CHANGE FORM

SOP NUMBER: KNOX-OP-0021

SOP TITLE: Extraction of Polychlorinated Biphenyl (PCB) Isomers for Analysis by Isotope Dilution HRGC/HRMS (EPA Methods 1668A, 1668B, and 1668C)

SOP SECTIONS AFFECTED BY CHANGE: Section 8.0

REASON FOR ADDITION OR CHANGE: Response to Wisconsin Audit

CHANGE EFFECTIVE FROM: 6/7/13

CHANGE OR ADDITION (SPECIFY SECTION; USE ADDITIONAL SHEETS IF NECESSARY)

Add Section 8.4. If residual chlorine is present, add 80 mg sodium thiosulfate per liter of water.

SUBMITTED BY/DATE: KRM 6/4/13

APPROVED BY:

Technical Reviewer Signature	6/4/13 **Date**
Environmental Health & Safety Signature	6-4-13 **Date**
QA Signature	6/4/13 **Date**
Management Signature	06/04/13 **Date**

Controlled Copy
Copy No. unc

SOP No.: KNOX-OP-0021
Revision No.: 3
Revision Date: 03/26/13
Implementation Date: 03/29/13
Page 1 of 45

TESTAMERICA KNOXVILLE

STANDARD OPERATING PROCEDURE

TITLE: Extraction of Polychlorinated Biphenyl (PCB) Isomers for Analysis by Isotope Dilution HRGC/HRMS (EPA Methods 1668A, 1668B and 1668C)

(SUPERSEDES: KNOX-OP-0021, Revision 2)

Prepared By: Michael A. Patty

Reviewed By: 3/29/13
Technical Specialist

Approved By: Kevin McGee 3/29/13
Quality Assurance Manager

Approved By: 3·29·13
Environmental, Health and Safety Coordinator

Approved By: 03/29/13
Laboratory Director

Copyright Information:

1. Scope and Application

1.1 This procedure is used by TestAmerica Knoxville for the extraction of PCB isomers from a variety of environmental matrices. This procedure is based on EPA methods 1668A, 1668B and 1668C, and is designed to meet analytical program requirements where isotope dilution HRGC/HRMS analysis of polychlorinated biphenyl (PCB) isomers is specified.

1.2 This procedure is based on "performance-based" methods. These reference methods allow modifications to overcome interferences or lower the cost of measurements if all performance criteria in the methods are met and method equivalency is established. Deviations from the referenced methods have been incorporated into this procedure and are listed in section 17.1.

1.3 Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCBs. It is the responsibility of all laboratory personnel to ensure that safe handling procedures are employed. Section 5 of this procedure discusses safety procedures.

2. Summary of Method

2.1 Screening and protocol assignment

2.1.1 All solid, sediment, waste and tissue samples are screened by GC/ECD prior to extraction. Aqueous samples may be screened if the potential for congener levels is suspected to be above 40 ng/L. Variations to sample size, spiking levels and final volume are established based on the screening result. Refer to Table 5.

2.2 Extraction

2.2.1 Aqueous samples (samples containing less than one percent solids): Stable isotopically labeled analogs of the toxic PCBs plus additional labeled PCBs (i.e., $^{13}C_{12}$ labeled internal standards) are typically spiked into a 1-L sample, and the sample is extracted with methylene chloride using separatory funnel techniques.

2.2.2 Solid and sediment samples: A 1.0 g sub-sample is screened to determine the greatest concentration for individual congeners. Based on the screen results, the sample is prepared by one of four protocols (refer to Table 5). Each protocol defines the sample amount to be extracted, the fraction of the extract to be used, and the final extract volume. Protocol 1 (or M1) is for samples which can be processed without adjustments for high levels of PCB congeners. In this protocol, the internal standards are spiked into a sample containing 10 g of solids. The sample is extracted for 18-24 hours with 1:1 hexane/acetone using a Soxhlet extractor; alternatively, solid samples may be extracted with 1:1 hexane/acetone by microwave with client approval. The extract is concentrated for cleanup.

NOTE: Sample sizes may be adjusted for dry weight or processed as received. The laboratory default for soil samples is to process as received. The laboratory default for sediment samples is to adjust the sample amount extracted to a minimum dry weight.

2.2.3 Multi-phase samples: Samples containing multiple phases are separated and the phases are extracted following the procedures for the appropriate matrix. The extracts may be combined for cleanup and analysis or processed separately. Specific handling of multi-phase samples should be discussed and documented with the project manager prior to extraction of samples.

2.2.4 Tissue samples: A 1.0 g sub-sample is screened to determine the greatest concentration for individual congeners. Based on the screen results, the sample is prepared by one of four protocols (refer to Table 5). Each protocol defines the sample amount to be extracted, the fraction of the extract to be used, and the final extract volume. Protocol 1 is for samples which can be processed without adjustments for high levels of PCB congeners. In this protocol, a 10 g aliquot of homogenized tissue is blended with sodium sulfate, spiked with the internal standards, and extracted for 18-24 hours in a Soxhlet extractor using 1:1 hexane/acetone. If required, a portion of the extract is used to determine the lipid content. (Refer to SOP KNOX-OP-0020, “Gravimetric Percent Lipids Determination” current revision.) The remaining extract is concentrated for cleanup.

2.2.5 Waste samples: Non-aqueous liquids such as oils and organic solvents are diluted or solvent exchanged in hexane.

2.2.6 Refer to SOP KNOX-ID-0012, current revision, for extraction of source air and ambient air samples.

2.3 After extraction, samples may be cleaned up using back-extraction with sulfuric acid, gel-permeation chromatography, silica gel chromatography, Florisil column chromatography, and/or TBA sulfur cleanup. Cleanup standards are added to each extract prior to initiating any cleanup steps.

2.4 After cleanup, the extract is concentrated to the appropriate final volume (refer to Table 5). Recovery standards are added to each extract and extracts are delivered to the HRMS lab for analysis. Refer to SOP KNOX-ID-0013, current revision.

3. Definitions

NOTE: Terminology differences existing in some isotope dilution reference methods regarding the functionality of the labeled analogs may lead to confusion. For example, EPA's Office of Solid Waste methods (8280, 8290) use the term "Internal Standards" to describe the labeled analogs which are added to the sample prior to extraction and used to quantitate the native targets. EPA's Office of Water methods (1613B, 1668, Revision A) use the term "Labeled Analogs" to describe these same compounds while using the term "Internal Standards" to describe the labeled analogs which are added to the extract just prior to analysis and used to quantitate the recovery of the labeled analogs added before extraction. EPA's Office of Solid Waste methods (8280, 8290) uses the term "Recovery Standards" to describe these later labeled analogs.

The terminology conventions established by the EPA's Office of Solid Waste methods (8280, 8290) are used in the laboratory for all Standard Operating Procedures and internal communications as defined in this section.

3.1 Cleanup Standards: Isotopically labeled compounds that are added to samples, blanks, quality control samples, and calibration solutions. They are added to the samples after extraction but prior to extract cleanup, and are used to judge the efficiency of the cleanup procedures.

3.2 Internal Standards: Isotopically labeled analogs of the target analytes that are added to every sample, blank, quality control spike sample, and calibration solution. They are added to the sample before extraction and are used to calculate the concentration of the target analytes or detection limits.

3.3 Recovery Standards: Isotopically labeled compounds which are added to every sample, blank, and quality control spike sample extract prior to analysis. They are used to measure the recovery of the internal standards and the cleanup standards.

3.4 Additional definitions can be found in the Knoxville Quality Assurance Manual.

4. Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. Specific selection of reagents and solvents may be required. Where possible, reagents are cleaned by extraction or solvent rinse. The non-coplanar PCB congeners 105, 114, 118, 123, 156, 157, 167, and 180 have been shown to be very difficult to completely eliminate from the laboratory at the minimum levels in this method, and baking of glassware in a kiln or furnace at 450 - 500°C may be necessary to remove these and other contaminants.

4.2 Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples but may also remove the analytes of interest by adsorption on the glass surface. For specific glassware cleaning procedures, see SOP KNOX-QA-0002, “Glassware Cleaning”, current revision.

4.3 All materials used in the analysis shall be demonstrated to be free from interferences by running laboratory method blanks (section 9.5) initially and with each sample batch (samples started through the extraction process on a given 12-hour shift, to a maximum of 20 samples).

4.4 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCBs. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxy-diphenyl ethers, benzylphenyl ethers, polynuclear aromatics and pesticides. Because very low levels of PCBs are measured by this method, the elimination of interferences is essential. The cleanup steps given in section 11.10 can be used to reduce or eliminate these interferences and thereby permit reliable determination of the PCBs at the levels required by the method.

4.5 Cleanup of tissue - The natural lipid content of tissue can interfere in the analysis of tissue samples for PCBs. The lipid contents of different species and portions of tissue can vary widely. Lipids are soluble to varying degrees in organic solvents and may be present in sufficient quantity to overwhelm the column chromatographic cleanup procedures used for sample extracts. Lipids must be removed by the sulfuric acid cleanup procedure (section 11.10.5) followed by GPC cleanup (section 11.10.6).

5. Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Corporate Safety Manual), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3 Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. For the operations described herein, Nitrile gloves are to be worn. For operations using solvents that may splash, SilverShield® gloves are recommended. SilverShield® gloves protect against breakthrough for most of the solvents used in this procedure.

5.4 When using a scalpel, wear cut-resistant gloves and cut away from your hand.

5.5 Primary Materials Used: The following is a list of the materials used in this method which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. 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 the method can be found in the reagents and materials section. 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.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid (1)	Corrosive, Oxidizer, Dehydradator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Methylene Chloride	Carcinogen, Irritant	25 ppm-TWA, 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable, Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable, Poison, Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Toluene	Flammable, Poison, Irritant	200 ppm-TWA, 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Nonane	Flammable	None established	Harmful if inhaled/swallowed. Vapor/mist is irritating to eyes, mucous membranes and upper respiratory tract. Causes skin irritation.
2-Propanol	Flammable	400 ppm -TWA	Flammable liquid and vapor. Harmful if swallowed or inhaled. Causes irritation to eyes and respiratory tract. Affects central nervous system. May be harmful if absorbed through the skin. May cause irritation to the skin.
Ethyl Ether	Flammable, Irritant, Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. **May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of within six months of opening the container. All unopened containers should be disposed of by the manufacturer's expiration date. See section 5.8.1.**

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Benzene	Flammable, Toxic, Carcinogen	PEL: 1 ppm TWA ; 5 ppm, 15 min. STEL	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.6 Chemicals that have been classified as **carcinogens**, **potential carcinogens**, or **mutagens include** benzene, methylene chloride, polychlorinated biphenyls, and toluene. The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.

5.7 Chemicals known to be **flammable** include acetone, benzene, hexane, nonane, 2-propanol, ethyl ether, and toluene.

5.8 The following material is known to be **corrosive**: Sulfuric acid.

5.9 Peroxides are highly reactive and/or explosive compounds. Diethyl ether may form peroxides when stored or evaporated to dryness. Once peroxides are formed, they can be detonated by simply moving the container. Employees who handle these materials must be aware of this problem. They must know when to submit the materials for disposal, how to check for peroxides, and what to do if peroxides have formed. Rules for handling peroxide forming compounds are:

5.9.1 Write the date the container is received and first opened on the label. The materials in the container must be submitted for disposal within 6 months of when the container is opened or by the manufacturer's expiration date on the container, whichever comes first.

5.9.2 Whenever possible, purchase these materials in the smallest available containers and only open those containers that will be exhausted within 6 months.

5.9.3 Cap all containers tightly and store in a dark area away from heat sources. If possible, purge the container with nitrogen before putting the cap on (suggestion: before replacing the cap).

5.9.4 Keep all materials that may form peroxides out of regular refrigerators. If they must be refrigerated, place them in explosion proof or flammable refrigerators or freezers.

5.9.5 Perform monthly checks with peroxide test strips on all open containers.

5.9.6 Collect and preserve all waste that might form peroxides with alcohol or other anti-oxidizers.

5.9.7 Avoid evaporating these types of materials to dryness. If you have to evaporate the materials to near dryness, use a stream of nitrogen. If possible, contain the apparatus behind an explosion shield.

5.9.8 If the shelf life of a container of one of these materials is exceeded, a container has been open for more than 6 months, or there is evidence of peroxide formation, contact the Environmental Health and Safety Coordinator (EHSC) or Environmental Health and Safety Director (EHSD). <u>DO NOT ATTEMPT TO OPEN OR MOVE THE CONTAINER</u>. This could cause the peroxide to detonate. Visible evidence of potential peroxide formation includes formation of crystals around the cap, formation of a viscous layer at the bottom of the container, or rust around the surface of the can.

5.10 Exposure to chemicals will be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.11 All procedures that involve solvents such as acetone, toluene, methylene chloride and hexane (e.g., glassware cleaning and the preparation of standards and reagents) must be conducted in a fume hood with the sash closed as far as the operations permit.

5.12 Safety glasses or a face shield **must** be used when employees are using solvents to rinse or clean glassware

5.13 Personal Hygiene: Thorough washing of hands and forearms is recommended after each manipulation and before breaks (coffee, lunch, and shifts).

5.14 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

5.15 All work must be stopped in the event of a known or potential compromise to the health or safety of laboratory personnel. The situation must be reported immediately to a laboratory supervisor.

6. Equipment and Supplies

NOTE: All glassware used in extraction and cleanup procedures is pre-cleaned as described in SOP KNOX-QA-0002, “Glassware Cleaning”, current revision. When extracting PCB samples, all of the reusable glassware used in the extraction and concentration process is identified with a "P" (for aqueous sample glassware), “L” or “LL” (for solid sample glassware) etched mark. All glassware used must be low level glassware.

6.1 Miscellaneous Laboratory Equipment

6.1.1 Laboratory fume hood of sufficient size to contain the equipment used for sample preparation

6.1.2 Oven, capable of maintaining a temperature of 125 to 135°C

6.1.3 Balance, >100 g capacity, accurate to ± 0.01 g

6.1.4 Balance, >2000 g capacity, accurate to ± 0.1 g

6.1.5 Syringes, various sizes

6.1.6 Borosilicate 5.75 inch and 9.0 inch disposable pipettes with rubber bulbs

6.1.7 Glass pipets, 1 mL, Class A

6.1.8 Graduated cylinders, 100 mL and 1000 mL capacity

6.1.9 PTFE squirt bottles, 500 mL

6.1.10 250 mL and 500 mL flasks

6.1.11 Ultra-Pure PTFE boiling stones

6.1.12 Glass wool, solvent rinsed

6.2 Tissue Homogenization Equipment:

6.2.1 Laboratory blender with glass body and stainless steel blades

6.2.2 Industrial meat grinder, Intedge Industries, Model C2H, or equivalent

6.2.3 Laboratory homogenizer, OMNI GLH-01, Model LR060902, or equivalent

6.2.4 Scalpels or knives

6.2.5 Cut-resistant gloves

6.3 Aqueous Sample Extraction Equipment

6.3.1 Separatory funnels, 250, 500, and 2000 mL, with PTFE stopcocks

6.3.2 Separatory funnel rotator

6.4 Solid/Tissue Sample Extraction Equipment

6.4.1 Soxhlet extractors, 50-mm ID, 200-mL capacity with 500 mL flasks

6.4.2 Glass condensers, capable of fitting top of Soxhlet apparatus

6.4.3 Whatman high purity glass fiber extraction thimbles

6.4.4 Heating mantles with temperature control

6.4.5 Flasks, Erlenmeyer, 500 mL

6.4.6 Beakers, 500 mL

6.4.7 Spatulas, stainless steel

6.5 Microwave Extraction Equipment

6.5.1 Microwave, Milestone, Ethos EX Microwave Labstation or equivalent

6.5.2 PRO-24 High-Throughput Sample Rotor with 24-position rotor body, Teflon vessels, 75 mL PEEK vessels and reference vessel for temperature monitoring

6.5.3 PRO-24 disposable glass inserts, 55 mL

6.5.4 Fiber optic temperature probe

6.5.5 Ceramic fiber optic probe sleeve

6.5.6 Q.P. Sensor for solvents

6.5.7 Teflon disk for reference vessel, Milestone part number APC4021

6.5.8 Magnetic stir bars, Fisher part number 14-513-58, or equivalent

6.6 Filtration Equipment

6.6.1 Glass or stainless steel funnels

6.6.2 Phase separation paper, Whatman 41 or equivalent

6.6.3 Buchner funnel, 15 cm

6.6.4 Glass-fiber filters for Buchner funnel

6.6.5 Filtration flasks, 1.5 to 2.0 L, with side arm

6.7 Cleanup Equipment

6.7.1 Disposable pipets, 150 mm long x 5 mm ID

6.7.2 Disposable pipets, 230 mm long x 5 mm ID

6.7.3 20 mm x 240 mm glass columns with support ring and tapered tip, for silica gel and Florisil cleanup

6.7.4 Vortex mixer

6.7.5 Gel Permeation chromatography system

6.8 Concentration Equipment

6.8.1 Nitrogen blowdown apparatus, N-EVAP (Organomation Associates, Inc., South Berlin, MA), installed in a fume hood

6.8.2 Kuderna-Danish (KD) Apparatus - 500 mL

6.8.3 Concentrator Tube, 10 mL, attached to KD with clips

6.8.4 Snyder Column, three-ball macro

6.8.5 Water Bath, heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.

6.8.6 Heating mantles with temperature control

6.9 Sample Vials

6.9.1 Borosilicate glass, 12 mL and 40 mL disposable with Teflon caps

6.9.2 Mini-vials, 1.1 mL capacity with a tapered bottom, with Teflon-faced, rubber septa and screw caps

6.9.3 Amber glass vials with Teflon-lined screw caps

6.10 Screening Equipment

6.10.1 Shaker table

6.10.2 An analytical system complete with a gas chromatograph and a ^{63}Ni electron capture detector and a data system capable of measuring peak height.

7. Reagents and Standards

CAUTION: Refer to Material Safety Data Sheets (MSDS) for specific safety information on chemicals and reagents prior to use or as needed

CAUTION: During preparation of reagents, associates shall wear a lab coat, gloves, safety glasses with side shields, and laboratory approved shoes as a minimum. Reagents shall be prepared in a fume hood.

7.1 Pre-cleaned glass wool - Pre-clean the glass wool with methylene chloride in a Soxhlet extractor for a minimum of 4 hours.

7.2 Sulfuric acid, concentrated, reagent grade (specific gravity 1.84)

7.3 Sodium chloride, reagent grade - Prepare a 5% (w/v) solution in reagent water.

7.4 Sodium sulfate, reagent grade, anhydrous, J.T Baker 3375, or equivalent

7.4.1 Sodium sulfate may be cleaned by putting approximately 600 g of sodium sulfate in large amber-colored glass jars and completely covering with methylene chloride, stirring the mixture with a stirring rod and letting the sodium sulfate soak for 5 minutes. The methylene chloride is drained and this step is repeated. After the methylene chloride is drained, the sodium sulfate is transferred to a Buchner funnel fitted onto a vacuum flask and rinsed 2 times with methylene chloride while a vacuum is being applied. The sodium sulfate is then placed into shallow borosilicate glass dishes where it is allowed to dry. It is placed in an oven at 125-130°C for 1 hour to complete the drying process. After drying, the sodium sulfate is transferred into pre-cleaned glass jars with Teflon® lined screw caps. These are placed in a desiccator until needed.

7.5 Hydromatrix, Agilent Technologies part number 198004

7.5.1 Hydromatrix is cleaned using the same procedure listed above for sodium sulfate, except toluene is used instead of methylene chloride.

7.6 Purified nitrogen

7.7 Solvents - Acetone, toluene, n-hexane, 2-propanol, methanol, methylene chloride, ethyl ether, benzene and nonane; pesticide quality.

7.8 Prepare 6% diethyl ether in hexane (v/v) by combining 940 mL of hexane and 60 mL of diethyl ether in a 4000 mL amber glass bottle. Shake well to mix. Label the container and store in the solvent cabinet.

7.9 Reagent water must be produced by a Millipore DI system, or equivalent, that is capable of producing water with ≥18 megohm-cm (MΏ-cm) resistivity. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.10 Florisil, pesticide residue (PR) grade (60/100) mesh; purchased activated at 1250°C (677°F), stored in a clear glass container with a ground glass top. Place in an oven at 125-135 °C for a minimum of 4 hours. Remove from oven and allow to cool before use. Store in the oven while not in use.

7.11 Silica gel, F679-212, Fisher Chromatographic Silica Gel, 100-200 mesh or equivalent - Prepare by Soxhlet extraction with methylene chloride for at least 6 hours. Transfer to a shallow, borosilicate glass dish and air dry. After drying, cover with aluminum foil and activate in an oven at 125-130°C for a minimum of 4 hours. Store in labeled glass jars in a desiccator until use.

7.12 3.3% Deactivated silica gel – To prepare, add 6.6 mL of reagent water to 200 g of pre-cleaned silica gel (section 7.11) in a 500 mL amber glass jar with a PFTE lined screw cap. Mix thoroughly by shaking until no lumps are visible, and the silica gel is free flowing and no longer sticks to the side of the jar. The Tornado II portable paint shaker may be used to aid in mixing these reagents. Store in a desiccator. Shake well again before each use.

7.13 Acidic silica gel – To prepare, add 57 mL of concentrated sulfuric acid to 180 g of pre-cleaned silica gel (section 7.11) in a 500 mL amber glass jar with a Teflon® lined screw cap. Mix thoroughly by shaking until no lumps are visible and the silica gel is free flowing and no longer sticks to the side of the jar. The Tornado II portable paint shaker may be used to aid in mixing these reagents. Store in a desiccator. Shake well again before each use.

7.14 Tetrabutylammonium hydrogen sulfate, Sigma Aldrich Cat. No 155837-100g, or equivalent, 97% purity

7.14.1 Tetrabutylammonium (TBA) sulfite reagent – Prepare the reagent by dissolving 3.39 g of tetrabutylammonium hydrogen sulfate in 100 mL of reagent water in a wide mouth clear jar with a Teflon® lined screw cap. To remove impurities, extract this solution three times with 20 mL portions of hexane, taking the hexane portion off the top and discarding. After discarding the last hexane wash, pour the TBA into a 250 mL amber glass jar and slowly add 25 g of sodium sulfite (in increments) to the solution. Shake the bottle until the sodium sulfite dissolves. Record the date prepared, initials of the preparer and expiration date on the bottle label. This solution can be stored for 1 month at room temperature in an amber bottle with a Teflon® lined lid.

7.15 Sodium sulfite, Sigma Aldrich Cat. No. 54872-1KG, or equivalent, 98+% purity

7.16 8082 Surrogate Mix: A surrogate spiking solution which contains 0.2 mg/L tetrachloro-m-xylene and decachlorobiphenyl. This mix is spiked into samples that are being prepared for PCB screening.

7.17 $^{13}C_{12}$ Labeled PCB Congener Standards: Obtained as individual Certified Reference Standards from Cambridge Isotope Laboratories (CIL, Andover Massachusetts) and Wellington Laboratories (Guelph, Ontario, Canada). Refer to Tables 2 and 3 for a list of individual standards. These individual standards are purchased at concentrations of 40 µg/mL or 50 µg/mL in nonane. If the chemical purity is 98% or greater, the weight may be used without correction to compute the concentration of the standard. Once a standard ampoule has been vortexed and opened, the solution is transferred to an amber glass vial with a Teflon®-lined screw cap. When not being used, standards are stored in the dark in a refrigerator. These purchased individual standards are used to prepare the following mixed stock solutions and spiking solutions:

7.17.1 Internal Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled internal standards listed in Table 2, to a concentration of 1000 ng/mL in nonane. The concentration is verified by GC/MS before use. Alternatively, a 1000 ng/mL mixture may be obtained from CIL. Refer to Table 2a.

7.17.2 Internal Standard Spiking Solutions: Different concentration spiking solutions are prepared for spiking aqueous samples and for spiking solid samples. They are prepared by diluting the 1000 ng/mL internal standard stock solution to a concentration of 2.0 ng/mL (aqueous spiking solution) and 10 ng/mL (solid spiking solution) in acetone. 1.0 to 4.0 mL of the 10 ng/mL solution is added to each solid/tissue sample prior to extraction. (Refer to Table 5, "Assignment of Sample Preparation Protocols" to determine the exact volume to add.) 1.0 mL of the 2.0 ng/mL solution is added to each aqueous sample prior to extraction.

7.17.3 Recovery Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled recovery standards listed in Table 3 to a concentration of 1000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.17.4 Recovery Standard Spiking Solution: Prepared by diluting the 1000 ng/mL recovery standard stock solution to a concentration of 100 ng/mL in nonane. 100 µL of this spiking solution is added to each solid/tissue sample extract prior to analysis, whereas, 20 µL is added to each aqueous sample extract.

7.17.5 Cleanup Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled cleanup standards listed in Table 3, to a concentration of 5000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.17.6 Cleanup Standard Spiking Solution: Prepared by diluting the 5000 ng/mL cleanup standard stock solution to a concentration of 10 ng/mL in hexane. 1.0 mL of this solution is added to each solid/tissue sample extract prior to cleanup (refer to Table 5), whereas, 0.20 mL is added to each aqueous sample extract.

7.18 Native PCB Congener Standard Mix - Obtained as a Certified Reference Standard from Accustandard (New Haven, CT). This standard contains all 209 PCB congeners at 4000 ng/mL in nonane. If the chemical purity is 98% or greater, the weight may be used without correction to compute the concentration of the standard. Once a standard ampoule has been vortexed and opened, the solution is transferred to an amber glass vial with a Teflon®-lined screw cap. When not being used, the standard is stored in the dark in a refrigerator at 4 ± 2°C. This purchased standard is used to prepare the LCS spiking solution:

7.18.1 LCS Spiking Solutions: Different concentration spiking solutions are prepared for spiking aqueous LCSs and for spiking solid LCSs. They are prepared by diluting the 4000 ng/mL native PCB congener standard to a concentration of 1.0 ng/mL (aqueous LCS spiking solution) and 5.0 ng/mL (solid LCS spiking solution) in acetone. The concentration is verified by GC/MS before use. 1.0 mL of the 5.0 ng/mL solution is added to each solid/tissue LCS prior to extraction, whereas, 1.0 mL of the 1.0 ng/mL solution is added to each aqueous LCS.

7.19 QC Check Sample: A QC Check Sample should be obtained from a source independent of the calibration standards. This check sample is a certified standard reference material (SRM) containing the PCBs in known concentrations in a sample matrix similar to the matrix under test. The National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland has SRM 1944 – New York/New Jersey Waterway Sediment, that the NYSDEC recommends for use.

8. Sample Collection, Preservation and Storage

8.1 Sampling is not performed for this method by TestAmerica Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, “Sample Receipt and Login”, current revision.

8.2 Sample Storage

8.2.1 Store aqueous samples in the dark at ≤6°C.

8.2.2 Store solid and tissue samples in the dark at ≤-10°C.

8.3 Holding Times

8.3.1 If stored according to the conditions specified in section 8.2, samples may be stored for up to one year.

8.3.2 Store sample extracts in the dark at room temperature until analyzed. If stored in the dark at room temperature, sample extracts may be stored for up to one year.

9. Quality Control

9.1 An initial demonstration of capability (IDOC) is performed to demonstrate the ability to generate acceptable precision and accuracy.

9.1.1 For aqueous samples, extract, clean, concentrate, and analyze four 1-L aliquots of reagent water spiked with internal standards, cleanup standard, recovery standard and the LCS spiking solution, according to the procedures in section 11. For solid/tissue samples, extract, clean, concentrate, and analyze four aliquots of sodium sulfate/corn oil spiked with internal standards, cleanup standard, recovery standard and the LCS spiking solution, according to the procedures in section 11. All sample processing steps that are to be used for processing samples, including preparation, extraction and cleanup, shall be included in this test.

9.1.2 Using the results of the set of four analyses, compute the average percent recovery (%R) of the extracts and the relative standard deviation (RSD) of the concentration in ng/mL for each compound.

9.1.3 For each PCB and labeled compound, compare the RSD and %R with the corresponding limits for initial precision and recovery in Table 4. If the RSD and %R for all compounds meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, any individual RSD exceeds the precision limit or any individual %R falls outside the range for accuracy, system performance is unacceptable for that compound. Correct the problem and repeat the test.

9.2 Internal Standards: Every sample, blank, and QC sample is spiked with $^{13}C_{12}$ labeled internal standards prior to extraction. Internal standards in samples, blanks, and QC samples are used to calculate the concentration of the target analytes or detection limits.

9.3 Cleanup Standards: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled cleanup standards after extraction but prior to extract cleanup. They are used to assess the efficiency of the cleanup procedures.

9.4 Recovery Standards: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled recovery standards prior to analysis. They are used to measure the recovery of the internal standards and the cleanup standards.

9.5 Method Blank: A laboratory method blank must be run along with each batch of 20 or fewer samples. The method blank is processed in the same manner and at the same time as the associated samples. The method blank is used to identify any background interference or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data. Refer to the table below for a cross-reference between the sample matrix and method blank matrix.

Sample Matrix	**Method Blank Matrix**
Aqueous	Reagent water
Solid (Soxhlet)	Sodium sulfate
Solid (Microwave)	Hydromatrix
Tissue	Sodium sulfate
Waste	Hexane

9.6 Laboratory Control Sample: A laboratory control sample (LCS) is prepared and analyzed with every batch of 20 or fewer samples. The LCS extract must be subject to the same cleanup procedures as the associated sample extracts. LCS spike components and concentrations are listed in Table 1. Refer to the table below for a cross-reference between the sample matrix and LCS matrix.

Sample Matrix	**LCS Matrix**
Aqueous	Reagent water
Solid (Soxhlet)	Sodium sulfate
Solid (Microwave)	Hydromatrix
Tissue	Sodium sulfate
Waste	Hexane

9.7 QC Check Sample: Analyze the QC Check Sample (section7.19) periodically to assure the accuracy of calibration standards and the overall reliability of the analytical process. It is suggested that the QC Check Sample be analyzed at least annually.

9.8 Quality Control Batch: The batch is a set of up to 20 client samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank and a laboratory control sample.

10. Calibration and Standardization

10.1 Not applicable.

11. Procedure

11.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variations in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variations in the procedure, except those specified by project specific instructions, shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist, Project Manager, and QA Manager. If contractually required, the client shall be notified.

11.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3 Samples are extracted by the following procedures depending upon sample matrix. Water samples are prepared by separatory funnel liquid/liquid extraction. Solid samples including soils, sediments, tissues and solid waste materials are prepared by Soxhlet extraction. Non-aqueous liquid wastes and organic solvents are prepared by waste dilution techniques.

NOTE: Samples should be removed from the refrigerator several hours before extraction and allowed to come to room temperature before measuring the volume or performing the extraction.

11.4 Aqueous Samples (samples containing 1% solids or less)

11.4.1 Aqueous samples that are suspected to have congener levels above 40 ng/L may be screened before extraction to prevent instrument saturation or contamination.

11.4.1.1 Carefully shake the sample bottle well. Measure out 10 mL of water and place in a 40 mL vial.

11.4.1.2 Add 100 µL of the 0.2 mg/L 8082 surrogate mix to the 10 mL of water. (Refer to section 7.16.)

11.4.1.3 Add 10 mL of methylene chloride and shake the 40 mL vial for 2 minutes. Vent the vial several times during the 2 minutes to relieve any pressure that might have built up.

11.4.1.4 Allow the water layer and the methylene chloride layer to separate, swirling the vial gently to aid in separation of the layers.

11.4.1.5 Using a disposable pipet, pull the methylene chloride (bottom layer) out and filter it through a sodium sulfate funnel and into a clean 40 mL vial.

11.4.1.6 Concentrate the extract to approximately 100 µL. Bring the extract back up to 1 mL with hexane and deliver to the GC group for screening. Refer to section 11.5 for details re. screening.

11.4.2 Refer to Knoxville SOP KNOX-QA-0002, current revision, for information on glassware cleaning procedures for extraction glassware.

11.4.3 For water samples, only use the glassware designated for those water samples.

11.4.4 Place separatory funnels, one for each sample, in the separatory rotator set up in the hood.

11.4.5 Place a 500 mL flask directly beneath the separatory funnel.

11.4.6 Plug a glass funnel with glass wool and pour in some sodium sulfate (about 1 to 2 inches from the top). Rinse the sodium sulfate with methylene chloride. After the methylene chloride stops dripping, place the funnel on top of the flask that is fitted with a paper clip to aid in filtering.

11.4.7 Inspect the sample for solids or biphasic sample characteristics. If either condition exists, consult the project manager for further instructions.

11.4.8 Weigh the sample container on a balance (± 1 g); tare the sample and container. Transfer the sample to the extractor. Rinse the sample bottle with approximately 60 mL methylene chloride and transfer to the extractor. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter.

11.4.8.1 If solids are present in the water sample, the sample volume is measured volumetrically.

11.4.8.2 For brackish water or salt water, weigh 1.0 mL of the sample. If the weight is <0.98 or >1.02, adjust the sample volume based on the sample density.

11.4.9 For the method blank and the LCS (and LCSD, if required), use a 1000 mL graduated cylinder to measure 1000 mL of reagent water.

11.4.10 Verify the final volume of the extract. If the standard 20 µL is specified, add 1.0 mL of the 2.0 ng/mL internal standard spiking solution to each sample, method blank and LCS. Additionally, add 1.0 mL of the 1.0 ng/mL LCS spiking solution to the LCS (and LCSD, MS/MSD, if required). Record the amount of spike used and the spike standard number on the extraction benchsheet.

NOTE: Each time the samples are spiked, the spiking process should be witnessed by another analyst. Refer to Appendix II for the steps that must be taken.

11.4.11 Add 60 mL of methylene chloride to the sample bottle and shake. Then add the methylene chloride to the separatory funnel. Add 60 mL of methylene chloride to the method blank, LCS (and LCSD, if required).

11.4.12 Secure the separatory funnel with the rotator retaining straps and rotate for 2 minutes.

CAUTION: Care should be used while performing this operation. Vent the separatory funnel frequently. Goggles may be worn when performing this procedure.

11.4.13 Allow the water and the methylene chloride to separate for 10 minutes. If it is not separated after 10 minutes, try to break up the emulsion by gently swirling the sample or tilting the separatory funnel on its side.

11.4.14 Drain the methylene chloride from the separatory funnel into the glass funnel that is filled with sodium sulfate, allowing the extract to drip into the flask. Be careful not to allow water to escape the separatory funnel or the sodium sulfate will harden and block the flow of the extract. If at least 10 minutes has elapsed and other ways of breaking up or reducing the size of the emulsion have failed, the following steps may be tried to reduce the impact of the emulsion on the sodium sulfate.

11.4.14.1 Place a large piece of pre-cleaned glass wool in the funnel containing the sodium sulfate.

11.4.14.2 Spread the glass wool out, covering the entire surface of the sodium sulfate to a depth of ~5 to 10 mm. If the emulsion is hard to break up and persistent, a small, additional layer of sodium sulfate may be added on top of the glass wool.

11.4.14.3 Drain the solvent and emulsion layer into the funnel, being careful to drain no more than 60 mL of volume if a clear phase layer cannot be determined.

11.4.14.4 If this procedure is used, the funnel should be rinsed with an extra 30 mL of methylene chloride to ensure all analytes are rinsed into flask.

11.4.15 Repeat the extraction process two more times with fresh 60 mL portions of methylene chloride.

11.4.16 After the third methylene chloride portion has filtered through the sodium sulfate, rinse the funnel with approximately 40 mL of methylene chloride.

11.4.17 Remove the separatory funnel from the hood and pour the extracted water into the extracted waters waste container.

11.4.18 Remove the glass funnel from the top of the flask.

11.4.19 Add a few boiling stones to the 500 mL boiling flask. Place a 3-ball Snyder column on the boiling flask. Pre-wet the Snyder column with 1 mL of the appropriate solvent (hexane or methylene chloride). For extracts that will go through GPC cleanup, concentrate to approximately 20 mL methylene chloride. If GPC cleanup is not going to be performed, solvent exchange the extracts to hexane as follows: Concentrate the extract to approximately 20mL, then add approximately 60 mL of hexane and concentrate to ≥ 20 mL.

NOTE: Over-concentration of heated extracts is known to cause the more volatile PCB congeners to vaporize and be lost. Therefore, do not concentrate to less than 20 mL.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst **must** add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.4.20 Transfer the extract into a solvent-rinsed 40 mL vial, rinsing the 500 mL flask three times with approximately 3 mL of the appropriate solvent (hexane or methylene chloride). Add the rinses to the 40 mL vial.

11.4.21 For extracts that will go through GPC cleanup, reduce the volume to approximately 6 mL methylene chloride on the N-EVAP concentration device. Proceed to section 11.10.

11.4.22 For extracts that will not go through GPC, reduce the volume to approximately 0.5 mL hexane on the N-EVAP concentration device. Do not allow the sample to go to dryness at any time. If an additional solvent exchange is needed, add 5 mL of hexane and swirl the vial. Reduce the volume of hexane to approximately 0.5 mL again to complete the solvent exchange. Adjust the final volume of the extract with hexane to 15 mL for acid cleanup or to approximately 6 to 8 mL for column cleanup. If the sample exhibits poor solubility in hexane, add approximately 1 mL of benzene to aid in dissolving the sample. Proceed to section 11.10.

11.5 Solid/Tissue Sample Screening and Assignment of Sample Preparation Protocols

11.5.1 Because of the sensitivity of method 1668, it is necessary to screen all solid/tissue samples and non-aqueous liquids before extraction to prevent instrument saturation and/or contamination.

11.5.2 Take a 1 ± 0.1 g subsample in accordance with KNOX-QA-0006, “Subsampling”, current revision. Mix with sodium sulfate in an aluminum pan until the mixture is free flowing.

11.5.3 Transfer to a 40 mL vial and add 1.0 mL of the 0.2 mg/L 8082 surrogate mix. (Refer to section 7.16.)

11.5.4 Add 9 mL of hexane.

11.5.5 Cap tightly and secure on a platform shaker table.

11.5.6 Shake for 1 hour, minimum.

11.5.7 Transfer ~ 4 mL of extract into a fresh 12 mL vial, add ~2 mL of sulfuric acid; cap tightly, and shake the vial for 30 to 60 seconds. If necessary, centrifuge the extract.

11.5.8 Deliver the extract to the GC group for screening.

11.5.9 Perform a single point initial calibration for each sequence on the screening instrument.

11.5.10 If no pattern and no prominent peaks are present, process the sample using protocol 1.

11.5.11 If an Aroclor-like pattern is observed, calculate the estimated concentration of the largest peak expected in the determinative analysis, and assign an extraction protocol using the "PCB Protocol Selection" spreadsheet on the local area network in the MSOffice\template\STL Knx SOG directory.

11.5.11.1 Determine the concentration of the most prominent technical mixture by comparison to Aroclor standards. Enter the Aroclor sample concentration (ppb) into the spreadsheet. Circle the amount entered on the GC hardcopy result.

11.5.11.2 For sediment samples, import the sample % moisture results.

11.5.11.3 Enter the average or selected surrogate recovery in the spreadsheet. The software will correct the PCB concentration for surrogate recovery.

11.5.11.4 The software will multiply the PCB concentration by 0.1 (the spreadsheet default) to correct for the weight percent of the largest single congener in an Aroclor mix. This value may be changed to represent special cases such as altered patterns or unusual technical mixtures, but documentation of the rationale must accompany the spreadsheet.

11.5.11.5 The software will then multiply the PCB concentration by 4 to account for single-peak coelutions.

11.5.11.6 The resulting concentration is used as the estimate of the largest peak expected in the determinative analysis and to assign an extraction protocol for each sample.

11.5.11.7 An independent analyst should review the data entry and protocol assignments. Initial and date the spreadsheet. Attach the spreadsheet and supporting GC data to the extraction benchsheet.

11.5.11.8 During sample preparation, adjust the sample extraction amount, spiking volumes, split ratio, final volume and bench dilution to reflect the selected protocol. Refer to Table 5.

11.6 Sample Pretreatment for Tissue Samples

11.6.1 If the sample matrix is tissue and has not been homogenized prior to sample receipt, the entire sample is homogenized prior to extraction using an industrial meat grinder, a laboratory blender, or a laboratory homogenizer. Select the equipment that is most appropriate for the size and type of tissue received.

11.7 Solid/Tissue Sample Soxhlet Extraction (samples containing more than 1% solids)

11.7.1 This section uses protocol 1 as an example. If another protocol has been assigned, the sample amount extracted, spike volumes and percent of extract used is modified based on Table 5.

11.7.2 Prepare and label the required number of Soxhlet systems. Refer to SOP KNOX-QA-0002, current revision, for information on cleaning procedures for extraction glassware. Visually inspect all glassware prior to use for scratches or cracks. Retire, repair or replace any glassware found to be damaged.

11.7.3 For soil samples, transfer a well-mixed 10 g aliquot (± 0.5 g) of the sample into a beaker or extraction thimble. Mix thoroughly with 20 g of sodium sulfate. Record the sample weight on the extraction benchsheet.

11.7.4 For sediment samples, adjust the amount weighed to achieve 10 to 10.5 g dry weight. Determine the amount of sediment sample to extract using the “Sediment Extraction Amounts” spreadsheet on the local area network in the MSOffice\template\Knx OrgPrep directory. Transfer a well-mixed aliquot of the sample into a beaker or extraction thimble. Mix thoroughly with 20 g of sodium sulfate. Record the sample weight on the extraction benchsheet.

11.7.5 For tissue samples, weigh out 10 g (± 0.5 g) of homogenized tissue into a beaker or extraction thimble. Mix thoroughly with 20 g of sodium sulfate. Record the sample weight on the extraction benchsheet.

NOTE: If gravimetric lipids are to be determined using the tissue extracts, split the extract prior to the initiation of any cleanup steps and use 1 mL of the 10 mL extract for lipids determination. Refer to SOP KNOX-OP-0020, “Gravimetric Percent Lipids Determination”, current revision.

11.7.6 Refer to the Section 9.5 for a cross-reference between the sample matrix and method blank matrix and Section 9.6 for the LCS matrix.

11.7.7 Spike each sample with 1.0 mL of the 10 ng/mL internal standard spiking solution (see section 7.17.2).

11.7.8 Spike the LCS (and LCSD, MS/MSD, if required) with 1.0 mL of the 5.0 ng/mL LCS spiking solution (see section 7.18.1).

NOTE: Each time the samples are spiked, the spiking process should be witnessed by another analyst. Refer to Appendix II for the steps that must be taken.

11.7.9 If needed, add a small amount of glass wool to the top of the extraction thimble.

11.7.10 Pour approximately 350 mL of the 1:1 hexane/acetone into a 500 mL flask. Place the flask in the heating mantle. Add several Teflon® boiling stones.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst **must** add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.7.11 Place the extraction thimble in the glass Soxhlet extractor.

11.7.12 Assemble the Soxhlet system and secure to the lab supports.

11.7.13 Adjust the temperature of the heating mantle to bring the solvent in the flask to a rolling boil. There should be a steady drip from the condensers so that the solvent should completely cycle at least 5 times an hour.

11.7.14 Soxhlet extract the sample in the above manner for 18-24 hours.

11.7.15 Turn off the heating mantle and allow the Soxhlet apparatus to cool.

11.7.16 Remove the condensers and allow the Soxhlet extractor chamber to empty; then remove the Soxhlet extractor from the 500 mL flask.

NOTE: If the samples appear to have a water layer or moisture, dry the extract by filtering it through a sodium sulfate filled funnel.

11.7.17 Add several Teflon® boiling stones and place a solvent-rinsed 3-ball Snyder column on the 500 mL flask. Pre-wet the Snyder column with 1 mL of hexane and concentrate the extract to $\geq$ 20 mL.

NOTE: Over-concentration of heated extracts is known to cause the more volatile PCB congeners to vaporize and be lost. Therefore, do not concentrate to less than 20 mL.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst **must** add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.7.18 Transfer the extract into a solvent-rinsed 40 mL vial, rinsing the 500 mL flask three times with approximately 3 mL of hexane. Add the rinses to the 40 mL vial.

11.7.19 Place the 40 mL vial into the N-EVAP concentration device and reduce the volume to approximately 10 mL. Proceed to section 11.10 to perform acid cleanup.

11.8 Solid/Tissue Sample Microwave Extraction (samples containing more than 1% solids)

11.8.1 This section uses protocol M1 as an example. If another protocol has been assigned, the sample amount extracted, spike volumes and percent of extract used is modified based on Table 5.

11.8.2 Prepare and label the required number of glass inserts.

11.8.3 Place a magnetic stir bar into each glass insert.

11.8.4 For soil samples, transfer a well-mixed 10 g (+/- 0.5 g) aliquot of the sample into a glass insert. Record the sample weight on the extraction benchsheet.

11.8.5 For sediment samples, adjust the amount weighed to achieve a 5 g dry weight. Determine the amount of sediment sample to extract using the "Sediment Extraction Amounts" spreadsheet on the local area network in the MSOffice\template\Knx OrgPrep directory. Record the sample weight on the extraction benchsheet.

11.8.6 Add approximately 0.5 g of toluene-precleaned Hydromatrix to each sample and QC sample. Mix the contents thoroughly with a solvent-rinsed metal spatula.

11.8.7 Spike each sample and QC samples with 1.0 mL of the 10 ng/mL internal standard spiking solution (see section 7.17.2). Record the standard solution ID and volume spiked on the extraction benchsheet.

NOTE: Each time the samples are spiked, the spiking process should be witnessed by another analyst. Refer to Appendix II for the steps that must be taken.

11.8.8 Spike the LCS (and LCSD, MS/MSD, if required) with 1.0 mL of the 5 ng/mL native LCS spiking solution (see section 7.18.1). Record the standard solution ID and volume spiked on the extraction benchsheet.

11.8.9 Add 25 mL of a 1:1 hexane/acetone solution and 1 mL of reagent water to all samples and QC samples.

11.8.10 Prepare the appropriate number of PEEK/Teflon vessels by doing an initial cleaning run. This is performed by adding 30 mL of 1:1 toluene/water and following steps 11.8.10 and 11.8.13 through 11.8.17. Solvent rinse the Teflon vessels with acetone, toluene, methylene chloride, and hexane.

11.8.11 Use the flange tool on the opening of the Teflon vessels. This is to ensure the cap fits securely onto the vessel.

11.8.12 Remove the sample label from each glass insert and place it on the outside of the associated Teflon vessel.

11.8.13 Place the glass insert inside the Teflon vessel and secure the cap.

11.8.14 Place each Teflon vessel into its PEEK vessel. Screw the top onto the housing and place it into the rotor for the microwave. Secure the cover onto the rotor.

11.8.15 For each run performed by the microwave, the reference vessel must be used. For optimum efficiency, the reference vessel must be used on the wettest sample. After placing the top onto the Teflon vessel, a ceramic fiber optic sleeve is placed through the vessel opening. Secure the microwave rotor inside the track of the microwave until it is secured properly. Take the fiber optic probe and feed it through the opening of the PEEK vessel top and a Teflon wafer. The Teflon wafer should be flush with the inside of the PEEK vessel top. Otherwise the wafer could be damaged upon screwing the top. Screw the top onto the PEEK vessel.

11.8.16 Turn the microwave on and let it warm up for a minimum of 15 minutes prior to using.

11.8.17 Turn on the fan using the touchpad.

11.8.18 In order to begin the run, choose the appropriate method saved and start the method by using the touchpad.

11.8.19 After the run is complete, allow the vessels to cool.

11.8.20 Prepare solvent-rinsed 250 mL boiling flasks and glass funnels. Plug the glass funnels with precleaned glass wool and add precleaned sodium sulfate.

11.8.21 Transfer the solid sample into the funnel using a solvent-rinsed metal spatula. Thoroughly rinse the glass insert, Teflon vessel, and metal spatula into the funnel with hexane. After the filtering is complete, rinse the sodium sulfate with hexane.

11.8.22 Add several fresh boiling chips to the flask. Insert a three-ball macro Snyder column into the top of the 250 mL boiling flask.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst must add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.8.22 Apply heat to the 250 mL flask in the heating mantle and reduce the extract volume to approximately 10-15 mL.

11.8.23 Transfer the extract into a solvent-rinsed 40 mL vial, rinsing the 250 mL flask three times with approximately 3 mL of hexane. Add the rinses to the 40 mL vial.

11.8.24 Place the 40 mL vial into the N-EVAP concentration device and reduce the volume to approximately 10 mL. Proceed to section 11.10 to perform acid cleanup.

11.9 Waste Sample Extraction

11.9.1 Organic wastes, oil, solids and non-aqueous sludge samples that will dissolve in solvent may be prepared by the waste dilution technique.

11.9.2 Add an appropriate amount of sample (based on screen results) to a solvent-rinsed 40 mL vial.

11.9.3 10 mL of hexane is used for the blank and LCS.

11.9.4 Spike each sample with 1.0 mL of the 10 ng/mL internal standard spiking solution (see section 7.17.2).

11.9.5 Spike the LCS (and LCSD, MS/MSD, if required) with 1.0 mL of the 5.0 ng/mL LCS spiking solution (see section 7.18.1).

NOTE: Each time the samples are spiked, the spiking process should be witnessed by another analyst. Refer to Appendix II for the steps that must be taken.

11.9.6 Add hexane to bring the volume to 10 mL. If the sample exhibits poor solubility in hexane, add approximately 1 mL of benzene to the vial to aid in dissolving the sample. Proceed to section 11.10 to perform acid cleanup.

11.9.7 Record the weights and volumes used on the extraction benchsheets.

11.10 Extract Cleanup

11.10.1 Cleanup may not be necessary for relatively clean samples (e.g., treated effluents, groundwater or drinking water). If particular circumstances require the use of a cleanup procedure, the laboratory may use any or all of the procedures below or any other appropriate procedure. Before using a cleanup procedure, the laboratory must demonstrate that the requirements of Section 13.2 can be met using the cleanup procedure.

11.10.2 Soil, sediment and tissue sample extracts are typically taken through the following cleanup procedures: Acid cleanup, GPC and silica gel cleanup.

Aqueous sample extracts are typically cleaned using GPC and silica gel cleanup silica gel, but may be cleaned using silica gel and Florisil column cleanup procedures.

11.10.3 If the sample requires a split based on the protocols listed in Table 5, the split must be performed prior to the initiation of the first cleanup procedure and prior to the addition of cleanup standards. For protocol 1 samples, use 5 mL of the 10 mL extract. Archive the remaining 5 mL aliquot of extract.

11.10.4 Spike each sample extract with an appropriate amount (i.e., 1.0 mL for solid, tissue and waste sample extracts; 0.20 mL for aqueous sample extracts) of the 10 ng/mL cleanup standard spiking solution (see section 7.17.6) prior to initiating any cleanup procedures..

11.10.5 Acid Cleanup

11.10.5.1 For protocol 1 samples, slowly add 4-5 mL of concentrated sulfuric acid to the 5 mL hexane extract in the 40 mL vial and shake for 30-60 seconds. For the other protocols listed in Table 5, adjust the acid proportionally to the extract volume. Vent the vial frequently while shaking. Let the vial stand or centrifuge to obtain well-defined separation of acid and solvent. Remove the acid layer with a glass pipet. (Optionally, the solvent layer can be removed.) Repeat the acid washing if the solvent layer is colored. Perform a maximum of four acid washings.

11.10.5.2 Add approximately 8 mL 5% (w/v) aqueous sodium chloride to the vial and gently shake for 30 seconds. Vent the vial frequently while shaking. Let the vial stand until a well-defined separation has occurred and remove the aqueous layer with a glass pipet. Dry the hexane extract by adding 1 to 2 grams of sodium sulfate and swirling the vial.

11.10.5.3 Place the 40 mL vial into the N-EVAP concentration device and reduce the volume to approximately 1 mL. Solvent exchange to methylene chloride and bring to approximately 6 to 8 mL. Proceed to section 11.10.6, GPC Cleanup.

11.10.6 GPC Cleanup

11.10.6.1 Gel permeation chromatography (GPC) removes high molecular weight interferences that cause GC column performance to degrade. It should be used for all soil, sediment, tissue and waste sample extracts. It may also be used for aqueous sample extracts.

11.10.6.2 Refer to SOP KNOX-OP-0022, current revision for detailed instructions for GPC cleanup.

11.10.6.3 After GPC cleanup, concentrate the extract and solvent exchange to ~6 to 8 mL hexane using low-level KD glassware. Proceed to section 11.10.7, Silica Gel Column Cleanup.

NOTE: If concentrating aqueous sample extracts, use glassware designated for water extraction and cleanup.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst **must** add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.10.7 Silica Gel Column Cleanup

11.10.7.1 Prepare 20 mm columns for each extract by placing a small amount of glass wool in the bottom of each column and then solvent rinsing with hexane. Shake out the excess hexane.

11.10.7.2 Mark the level to which the column packings will be added, starting at the top of the glass wool plug and proceeding from bottom to the top.

- Layer 1 – 12 mm (2 g) of 3.3% deactivated silica gel
- Layer 2 – 16 mm (4 g) of acidic silica gel
- Layer 3 – 12 mm (2 g) of 3.3% deactivated silica gel
- Layer 4 – 10 mm of sodium sulfate

11.10.7.3 Add the column packings to the 20 mm column in the order listed above, tapping the column to settle the contents to prevent channeling.

11.10.7.4 Add 30 mL of hexane to pre-elute the column. Do not collect the pre-eluted hexane.

11.10.7.5 When the hexane reaches the top of the sodium sulfate layer, place a solvent-rinsed 250 or 500 mL flask under the column and transfer the sample extract into the column. Quickly rinse the extract vial with a small amount of hexane and add the rinse into the column. Repeat the rinse 2 more times.

11.10.7.6 As the solvent level from the last rinse reaches the top of the sodium sulfate layer, add 70 mL of hexane into the silica gel column to elute the PCB’s into the flask.

11.10.7.7 When the column stops dripping, add several Teflon® boiling stones to the flask and attach a solvent-rinsed Snyder column. Concentrate the extract to $\geq$ 20 mL on a heating mantle.

NOTE: Over-concentration of heated extracts is known to cause the more volatile PCB congeners to vaporize and be lost. Therefore, do not concentrate to less than 20 mL.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst **must** add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.10.7.8 Transfer the extract to a 40 mL vial, rinsing the flask with a small amount of hexane and add the rinse to the 40 mL vial. Repeat the rinse 2 more times.

11.10.7.9 Place the 40 mL vial into the N-EVAP concentration device and reduce the volume to approximately 10 mL. For aqueous samples that did not go through GPC cleanup, proceed to section 11.10.8, Florisil Column Cleanup. For aqueous, soil, sediment, tissue and waste samples that were put through GPC cleanup, proceed to section 11.10.10, Final Concentration.

11.10.8 Florisil Column Cleanup

11.10.8.1 Place a small ball of glass wool in the bottom of a 20 mm column. Rinse twice with hexane and shake out the excess hexane.

11.10.8.2 Attach the column to the lab support in the hood.

11.10.8.3 Pack the Florisil column with the following layers. Add the column packing while tapping the column to settle the contents to prevent channeling. The order of the layers is from bottom to top.

- Layer 1 - 11 cm (15 g) of Florisil
- Layer 2 ~ 2 cm (~2 g) of sodium sulfate

11.10.8.4 Pour 80 mL of 6% diethyl ether in hexane (v/v) into a graduate cylinder (one graduate cylinder for each column) and set aside for later use in the procedure.

11.10.8.5 Pre-elute the column with 50 mL of 6% diethyl ether in hexane (v/v). This may be pushed through with a pipet bulb. Then pre-elute with 50 mL of hexane. Discard these pre-elutions.

11.10.8.6 Just before the level of hexane reaches the top of the sodium sulfate layer, place a solvent-rinsed 250 or 500 mL flask under the column and transfer the sample extract into the top of the column. Quickly rinse the vial 3 times with a small amount of hexane and add these rinses to the column.

11.10.8.7 Just before the solvent level reaches the top of the sodium sulfate, pour the 80 mL of 6% diethyl ether in hexane (v/v) into the top of the column and allow this to drip through the column and into the flask.

11.10.8.8 When the column stops dripping, add several Teflon® boiling stones to the flask and attach a solvent-rinsed Snyder column. Concentrate the extract to ≥ 20 mL on a heating mantle.

NOTE: Over-concentration of heated extracts is known to cause the more volatile PCB congeners to vaporize and be lost. Therefore, do not concentrate to less than 20 mL.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst **must** add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.10.8.9 Transfer the extract to a 40 mL vial, rinsing the flask with a small amount of hexane and add the rinse to the 40 mL vial. Repeat the rinse 2 more times.

11.10.8.10 If TBA cleanup is needed, place the 40 mL vial into the N-EVAP concentration device and reduce the volume to approximately 10 mL and proceed to section 11.10.9. Otherwise, proceed to section 11.10.10, Final Concentration.

11.10.9 Sulfur clean-up by Tetrabutylammonium (TBA)

11.10.9.1 Prepare sodium sulfate funnels by rinsing small funnels with acetone, methylene chloride and hexane, in that order. Add a small plug of glass wool and a small amount of sodium sulfate to each funnel. Rinse with hexane.

11.10.9.2 Add 1.0 mL of TBA sulfite reagent and 2.0 mL of 2-propanol to the vial containing the extract. Cap securely and shake or vortex for approximately 1 minute.

11.10.9.3 If the extract is colorless (or the initial color is unchanged) and if clear crystals (precipitated sodium sulfite) are observed, sufficient sodium sulfite is present. However, if the precipitated sodium sulfite disappears, add more sodium sulfite in approximately 0.10 g portions and vortex until a solid residue remains.

11.10.9.4 Add 5 mL of reagent water and shake or vortex the vial for approximately 1 minute. Allow the layers to separate (at least 5 minutes). Pipet off the hexane layer (the top layer) and filter through the sodium sulfate funnel (prepared in 11.9.9.1 above) into a solvent-rinsed 40 mL vial.

11.10.9.5 Add 2 mL of fresh hexane to the sample extract vial. Vortex for 30 seconds and allow the layers to separate again. Pipet off the hexane portion and filter through the sodium sulfate funnel into the clean 40 mL vial. Repeat once.

11.10.9.6 Rinse the sodium sulfate funnel with approximately 2 mL of hexane. When the hexane stops dripping, remove the funnel and cap the vial.

11.10.9.7 Proceed to section 11.10.10 for final concentration.

11.10.10 Final Concentration

11.10.10.1 Place the 40 mL vial containing the extract in the N-EVAP concentration apparatus and reduce the solvent volume to approximately 0.5 to 1.0 mL.

11.10.10.2 Label a 1.1 mL tapered mini-vial with the sample ID after checking that the vial and cap fit together properly.

11.10.10.3 For aqueous samples, add 20 µL of the 100 ng/mL recovery standard spiking solution (see section 7.17.4) to the mini-vial and mark the level on the vial. Transfer the concentrated extract into the mini-vial, rinsing 2 times with small amounts of hexane. Then reduce the extract volume back down to 20 µL. Deliver the mini-vial to the HRMS lab for analysis.

11.10.10.4 For protocol 1 solid, sediment, tissue and waste samples, add 50 µL of the 100 ng/mL recovery standard spiking solution (see section 7.17.4) to the mini-vial and mark the level on the vial. Transfer the concentrated extract into the mini-vial, rinsing 2 times with small amounts of hexane. For protocol 1 samples, reduce the extract volume to 50 µL. Deliver the mini-vial to the HRMS lab for analysis.

11.10.10.5 For samples prepared according to protocols 2, 3 and 4, add 100 µL of the 100 ng/mL recovery standard spiking solution and dilute the extract with nonane to achieve the dilution factor shown in Table 5.Deliver the mini-vial to the HRMS lab for analysis.

12. Data Analysis and Calculations

12.1 Not applicable.

13. Method Performance

13.1 Method Detection Limit (MDL) - An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. The procedure for determination of the method detection limit is given in the SOP CA-Q-S006, current revision, based on 40 CFR Part 136 Appendix B. The result of the MDL determination must support the reporting limit.

13.2 Initial Demonstration of Capability – Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in TestAmerica Knoxville SOP KNOX-QA-0009, current revision.

13.3 Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009, current revision for further requirements for performing and documenting initial and on-going demonstrations of capability.

14. Pollution Prevention

14.1 All attempts will be made to minimize the use of solvents and standard materials.

15. Waste Management

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment, employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 The following waste streams are produced when this method is carried out.

- Waste methylene chloride, acetone, hexane and toluene shall be placed in the flammable waste stream, contained in a steel satellite accumulation container or flammable solvent container.

- Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials shall be placed in the incinerable laboratory waste stream, contained in a steel or HDPE satellite accumulation container.
- Extracted solid/tissue samples, paper funnel filters, glass wool, etc., contaminated with solvents shall be placed in the incinerable laboratory waste stream, contained in a steel or HDPE satellite accumulation container.
- Contaminated sulfuric acid used during extract cleanup shall be placed in the acidic waste stream, contained in a HDPE satellite accumulation container.
- Extracted aqueous samples, contaminated with methylene chloride shall be placed in the organic water waste stream, contained in a HDPE satellite accumulation container.

16. References

16.1 Method 1668, Revision A, Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-821-R-00-002, December 1999.

16.2 Method 1668, Revision B, Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-821-R-08-020, November 2008.

16.3 Method 1668, Revision C, Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-820-R-10-005, April 2010.

16.4 TestAmerica Knoxville SOP KNOX-ID-0013, “Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HRMS”, current revision.

16.5 TestAmerica Knoxville SOP KNOX-OP-0020, “Gravimetric Percent Lipids Determination” current revision.

16.6 TestAmerica Knoxville SOP KNOX-OP-0022, “GPC Cleanup”, current revision.

16.7 TestAmerica Knoxville SOP KNOX-QA-0002, “Glassware Cleaning”, current revision.

16.8 TestAmerica Knoxville SOP KNOX-QA-0009, “Personnel Orientation and Training”, current revision.

16.9 TestAmerica Knoxville Laboratory Quality Assurance Manual (QAM), current revision.

16.10 SW-846 Method 3546, Microwave Extraction, Revision 0, February 2007.

17. Miscellaneous

17.1 Modifications and enhancements from EPA Method 1668, Revisions A and B: Method 1668 is a “performance-based” method and allows for method modifications (e.g., alternate extraction, concentration and cleanup procedures, etc.) as long as the method performance criteria described in Section 13.2 are met.

17.1.1 The cleanup procedures described in this SOP have been optimized and may vary significantly from the referenced methods.

17.1.2 Additional recovery standards are used in this procedure. The additional standards are listed in Table 3.

17.1.3 A solvent mixture of 1:1 hexane/acetone is used for Soxhlet extraction of solid and tissue samples that have been mixed with sodium sulfate prior to extraction.

17.1.4 Method 1668 specifies that the sample bottle is rinsed twice with 5 mL of reagent water after the sample is transferred to the separatory funnel. This procedure specifies that the sample bottle is rinsed three times with methylene chloride after the sample is transferred to the separatory funnel. This modification improves the removal of target compounds from the sample bottle.

17.1.5 The acid cleanup procedure is carried out in a 40 mL vial instead of a separatory funnel. Disposable glassware is used to decrease the risk of cross contamination. The volumes of the washes used have been adjusted for use in the 40 mL vial.

17.1.6 Extracts are stored at room temperature in the dark rather than at <10 °C. Method 1668 allows for the storage of standards at room temperature. Recovery studies performed by Cambridge Isotopes Laboratories (CIL) indicate freezing or refrigeration of standards causes problems with precipitation and irreversible adsorption to the inside surface of the vial. CIL recommends the storage of standards and extracts at room temperature as long as they are protected from exposure to UV and evaporative losses.

17.1.7 Aqueous samples with < 1% solids are extracted as received by separatory funnel. Samples with high solids content, > 1% solids, are either shaken well to thoroughly mix the sample followed by extraction by separatory funnel, or separated into two different samples for extraction and analysis, dependent upon client request.

17.1.8 Solid samples are extracted as received, with %-moisture determination performed simultaneously. Dry weight correction to results is applied at the time of data reporting. Particle size determination and reduction as specified in method 1668 is not performed on a routine basis. These procedures are considered to be outside the scope of the laboratories routine extraction procedures and are only performed on a client specific or project specific basis. These procedures, if required, must be specified and documented in the appropriate QAPPs.

17.1.9 Tissue samples are homogenized as described in Section 11.6.1.

17.1.10 Samples are extracted immediately after spiking.

17.2 List of Tables and Appendices:

17.2.1 Table 1 – Concentration of Native PCB Congener Stock and Spiking Solutions

17.2.2 Table 2 – Concentration of $^{13}C_{12}$ Labeled Internal Standard Stock and Spiking Solutions

17.2.3 Table 2a - Concentration of $^{13}C_{12}$ Labeled Internal Standard Stock and Spiking Solutions (Cambridge Mix)

17.2.4 Table 3 - Concentration of $^{13}C_{12}$ Labeled Recovery Standard and Cleanup Standard Stock and Spiking Solutions

17.2.5 Table 4 – Acceptance Criteria for Performance Tests

17.2.6 Table 5 – Assignment of Sample Preparation Protocols for Soxhlet Extraction

17.2.7 Table 6 – Assignment of Sample Preparation Protocols for Microwave Extraction

17.2.8 Table 7 – Typical Microwave Conditions

17.2.9 Appendix I – Example Extraction Benchsheet

17.2.10 Appendix II – Guidelines for the Spike Witnessing Process

Table 1 - Concentration of Native PCB Congener Stock and Spiking Solutions

Native PCB Congener	BZ/ IUPAC	Standard Source	Catalog Number	Vendor Conc (ng/mL)	Aqueous Sample LCS Spiking Solution Conc. (ng/mL)	Solid Sample LCS Spiking Solution Conc (ng/mL)
2-MoCB	1	AccuStd	S-99994-4x	4000	1.0	5.0
4-MoCB	3	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’-DiCB	4	AccuStd	S-99994-4x	4000	1.0	5.0
4,4’-DiCB	15	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,6-TrCB	19	AccuStd	S-99994-4x	4000	1.0	5.0
3,4,4’-TrCB	37	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,6,6’-TeCB	54	AccuStd	S-99994-4x	4000	1.0	5.0
3,3’,4,4’-TeCB	77	AccuStd	S-99994-4x	4000	1.0	5.0
3,4,4’,5-TeCB	81	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,4,6,6’-PeCB	104	AccuStd	S-99994-4x	4000	1.0	5.0
2,3,3’,4,4’-PeCB	105	AccuStd	S-99994-4x	4000	1.0	5.0
2,3,4,4’,5-PeCB	114	AccuStd	S-99994-4x	4000	1.0	5.0
2,3’,4,4’,5-PeCB	118	AccuStd	S-99994-4x	4000	1.0	5.0
2’,3,4,4’,5-PeCB	123	AccuStd	S-99994-4x	4000	1.0	5.0
3,3’,4,4’,5-PeCB	126	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,4,4’,6,6’-HxCB	155	AccuStd	S-99994-4x	4000	1.0	5.0
2,3,3’,4,4’,5-HxCB	156	AccuStd	S-99994-4x	4000	1.0	5.0
2,3,3’,4,4’,5’-HxCB	157	AccuStd	S-99994-4x	4000	1.0	5.0
2,3’,4,4’,5,5’-HxCB	167	AccuStd	S-99994-4x	4000	1.0	5.0
3,3’,4,4’,5,5’-HxCB	169	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,3,3’,4,4’,5-HpCB	170	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,3,4,4’,5,5’-HpCB	180	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,3,4’,5,6,6’-HpCB	188	AccuStd	S-99994-4x	4000	1.0	5.0
2,3,3’,4,4’,5,5’-HpCB	189	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,3,3’,5,5’,6,6’-OcCB	202	AccuStd	S-99994-4x	4000	1.0	5.0
2,3,3’,4,4’,5,5’,6-OcCB	205	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,3,3’,4,4’,5,5’,6-NoCB	206	AccuStd	S-99994-4x	4000	1.0	5.0
2,2’,3,3’,4’,5,5’,6,6’-NoCB	208	AccuStd	S-99994-4x	4000	1.0	5.0
DeCB	209	AccuStd	S-99994-4x	4000	1.0	5.0
All other CB congeners	NA	AccuStd	S-99994-4x	4000	1.0	5.0

Table 2 - Concentration of $^{13}C_{12}$ Labeled Internal Standard Stock and Spiking Solutions

Labeled PCB Congener	BZ/ IUPAC	Standard Source	Catalog Number	Vendor Conc (ng/mL)	Stock Conc (ng/mL)	Aqueous Sample Spiking Solution Conc (ng/mL)	Solid Sample Spiking Solution Conc (ng/mL)
Internal Standards							
$^{13}C_{12}$-2-chlorobiphenyl	1L	Cambridge	EC-4908	40,000	1000	2.0	10
$^{13}C_{12}$-4-chlorobiphenyl	3L	Cambridge	EC-4990	40,000	1000	2.0	10
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	Cambridge	EC-4911	40,000	1000	2.0	10
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	Cambridge	EC-1402	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	Cambridge	EC-4909	40,000	1000	2.0	10
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	Cambridge	EC-4901	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	Cambridge	EC-4912	40,000	1000	2.0	10
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	Cambridge	EC-1404	40,000	1000	2.0	10
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	Cambridge	EC-1412	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	Cambridge	EC-4910	40,000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	Cambridge	EC-1420	40,000	1000	2.0	10
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	Cambridge	EC-4902	40,000	1000	2.0	10
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	Cambridge	EC-1435	40,000	1000	2.0	10
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	Cambridge	EC-4904	40,000	1000	2.0	10
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	Cambridge	EC-1425	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	Cambridge	EC-4167	40,000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	Cambridge	EC-1422	40,000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	Cambridge	EC-4051	40,000	1000	2.0	10
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	Cambridge	EC-4050	40,000	1000	2.0	10
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	Cambridge	EC-1416	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	Cambridge	EC-4905	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	Cambridge	EC-4913	40,000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	Cambridge	EC-1409	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	Cambridge	EC-1408	40,000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	Cambridge	EC-4199	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	Cambridge	EC-4900	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	Cambridge	EC-1419	40,000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	Cambridge	EC-1410	40,000	1000	2.0	10

Table 2a - Concentration of $^{13}C_{12}$ Labeled Internal Standard Stock and Spiking Solutions (Cambridge Mix)

Labeled PCB Congener	BZ/ IUPAC	Standard Source	Catalog Number	Vendor Conc (ng/mL)	Stock Conc (ng/mL)	Aqueous Sample Spiking Solution Conc (ng/mL)	Solid Sample Spiking Solution Conc (ng/mL)
Internal Standards							
$^{13}C_{12}$-2-chlorobiphenyl	1L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-4-chlorobiphenyl	3L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl *	**170L**	**Cambridge**	**EC-4905**	**40,000**	**1000**	**2.0**	**10**
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	Cambridge	EC-4977	1000	1000	2.0	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	Cambridge	EC-4977	1000	1000	2.0	10

*PCB 170L is not included in the mix from Cambridge. It is purchased as an individual analyte and added to the mix. It is received at a concentration of 40,000 ng/mL. Bold denotes that this analyte must be added to the mix.

Table 3 - Concentration of $^{13}C_{12}$ Labeled Recovery Standard and Cleanup Standard Stock and Spiking Solutions

Labeled PCB Congener	BZ/ IUPAC	Standard Source	Catalog Number	Vendor Conc (ng/mL)	Stock Conc (ng/mL)	Spiking Solution Conc (ng/mL)
Recovery Standards						
$^{13}C_{12}$-2,5-dichlorobiphenyl	9L	Cambridge	EC-4165	40,000	1000	100
$^{13}C_{12}$-2,4',5-trichlorobiphenyl	31L	Wellington	MBP-31	50,000	1000	100
$^{13}C_{12}$-2,4',6-trichlorobiphenyl	32L	Cambridge	EC-4163	40,000	1000	100
$^{13}C_{12}$-2,2',5,5'-tetrachlorobiphenyl	52L	Cambridge	EC-1424	40,000	1000	100
$^{13}C_{12}$-2,2',4,5,5'-pentachlorobiphenyl	101L	Cambridge	EC-1405	40,000	1000	100
$^{13}C_{12}$-3,3',4,5,5'-pentachlorobiphenyl	127L	Cambridge	EC-1421	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5'-hexachlorobiphenyl	138L	Cambridge	EC-1436	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-heptachlorobiphenyl	180L	Cambridge	EC-1407	40,000	1000	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-octachlorobiphenyl	194L	Cambridge	EC-1418	40,000	1000	100
Cleanup Standards						
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	Cambridge	EC-1413	40,000	5000	10
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	111L	Cambridge	EC-1415	40,000	5000	10
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	178L	Cambridge	EC-1417	40,000	5000	10

Table 4 - Acceptance Criteria for Performance Tests

Toxic & LOC Congeners	IUPAC	Test Conc (ng/mL)[1]	IDOC %RSD	IDOC %R	LCS %R
2-chlorobiphenyl	1	50	40	60-140	50-150
4-chlorobiphenyl	3	50	40	60-140	50-150
2,2’-dichlorobiphenyl	4	50	40	60-140	50-150
4,4’-dichlorobiphenyl	15	50	40	60-140	50-150
2,2’,6-trichlorobiphenyl	19	50	40	60-140	50-150
3,4,4’-trichlorobiphenyl	37	50	40	60-140	50-150
2,2’,6,6’-tetrachlorobiphenyl	54	50	40	60-140	50-150
3,3’,4,4’-tetrachlorobiphenyl	77	50	40	60-140	50-150
3,4,4’,5-tetrachlorobiphenyl	81	50	40	60-140	50-150
2,2’4,6,6’-pentachlorobiphenyl	104	50	40	60-140	50-150
2,3,3’,4,4’-pentachlorobiphenyl	105	50	40	60-140	50-150
2,3,4,4’,5-pentachlorobiphenyl	114	50	40	60-140	50-150
2,3’,4,4’,5-pentachlorobiphenyl	118	50	40	60-140	50-150
2’,3’,4,4’,5-pentachlorobiphenyl	123	50	40	60-140	50-150
3,3’,4,4’,5-pentachlorobiphenyl	126	50	40	60-140	50-150
2,2’,4,4’,6,6’-hexachlorobiphenyl	155	50	40	60-140	50-150
2,3,3’,4,4’,5-hexachlorobiphenyl	156	50	40	60-140	50-150
2,3’,4,4’,5,5’-hexachlorobiphenyl	157	50	40	60-140	50-150
2,3,3’,4,4’,5’-hexachlorobiphenyl	167	50	40	60-140	50-150
3,3’,4,4’,5,5’-hexachlorobiphenyl	169	50	40	60-140	50-150
2,2’,3,4’5,6,6’-heptachlorobiphenyl	188	50	40	60-140	50-150
2,3,3’,4,4’,5,5’-heptachlorobiphenyl	189	50	40	60-140	50-150
2,2’,3,3',5,5',6,6’-octachlorobiphenyl	202	50	40	60-140	50-150
2,3,3',4,4',5,5',6-octachlorobiphenyl	205	50	40	60-140	50-150
2,2’,3,3’,4,4’,5,5’,6-nonachlorobiphenyl	206	50	40	60-140	50-150
2,2’,3,3’,4,5,5’,6,6’-nonachlorobiphenyl	208	50	40	60-140	50-150
2,2’,3,3’,4,4’,5,5’,6,6’-decachlorobiphenyl	209	50	40	60-140	50-150
Internal Standards					
$^{13}C_{12}$-2-chlorobiphenyl	1L	100	50	35-135	30-140
$^{13}C_{12}$-4-chlorobiphenyl	3L	100	50	35-135	30-140
$^{13}C_{12}$-2,2’-dichlorobiphenyl	4L	100	50	35-135	30-140
$^{13}C_{12}$-4,4’-dichlorobiphenyl	15L	100	50	35-135	30-140
$^{13}C_{12}$-2,2’,6-trichlorobiphenyl	19L	100	50	35-135	30-140
$^{13}C_{12}$-3,4,4’-trichlorobiphenyl	37L	100	50	35-135	30-140
$^{13}C_{12}$-2,2’,6,6’-tetrachlorobiphenyl	54L	100	50	35-135	30-140
$^{13}C_{12}$-3,3’,4,4’-tetrachlorobiphenyl	77L	100	50	35-135	30-140
$^{13}C_{12}$-3,4,4’,5-tetrachlorobiphenyl	81L	100	50	35-135	30-140
$^{13}C_{12}$-2,2’,4,6,6’-pentachlorobiphenyl	104L	100	50	35-135	30-140
$^{13}C_{12}$-2,3,3’,4,4’-pentachlorobiphenyl	105L	100	50	35-135	30-140
$^{13}C_{12}$2,3,4,4’,5-pentachlorobiphenyl -	114L	100	50	35-135	30-140
$^{13}C_{12}$-2,3’,4,4’,5-pentachlorobiphenyl	118L	100	50	35-135	30-140
$^{13}C_{12}$-2’,3,4,4’,5-pentachlorobiphenyl	123L	100	50	35-135	30-140
$^{13}C_{12}$-3,3’,4,4’,5-pentachlorobiphenyl	126L	100	50	35-135	30-140
$^{13}C_{12}$-2,2’,4,4’,6,6’-hexachlorobiphenyl	155L	100	50	35-135	30-140
$^{13}C_{12}$-2,3,3’,4,4’,5-hexachlorobiphenyl	156L	100	50	35-135	30-140
$^{13}C_{12}$-2,3,3’,4,4’,5’-hexachlorobiphenyl	157L	100	50	35-135	30-140
$^{13}C_{12}$-2,3’,4,4’,5,5’-hexachlorobiphenyl	167L	100	50	35-135	30-140
$^{13}C_{12}$-3,3’,4,4’,5,5’-hexachlorobiphenyl	169L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	100	50	35-135	30-140
$^{13}C_{12}$-2,2’,3,4’,5,6,6’-heptachlorobiphenyl	188L	100	50	35-135	30-140
$^{13}C_{12}$-2,3,3’,4,4’,5,5’-heptachlorobiphenyl	189L	100	50	35-135	30-140
$^{13}C_{12}$-2,2’,3,3’,5,5’,6,6’-octachlorobiphenyl	202L	100	50	35-135	30-140

Table 4 - Acceptance Criteria for Performance Tests (continued)

Internal Standards	**IUPAC**	**Test Conc (ng/mL)** [1]	**IDOC %RSD**	**IDOC %R**	**LCS %R**
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	100	50	35-135	30-140
Cleanup Standards					
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	50	45	45-120	40-125
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	111L	50	45	45-120	40-125
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	178L	50	45	45-120	40-125

[1] Test concentrations are based on ng/mL in the sample extract or standard solution.

Table 5 - Assignment of Sample Preparation Protocols for Soxhlet Extraction

1668 Protocol	Protocol Name	Sample Amount Extracted (g)	Fraction of Extract Cleaned	IS Added (mL)	Cleanup Std Added (mL)	Recovery Std Added (µL)	Extract Delivery Volume (µL)	Prep Split Factor	QuantIMS Final Volume (µL)
P1	Clean	10	1/2	1	0.5 (added after split)	50	50	2	100
P2	Low	2	1	1	1	100	100	1	100
P3	Medium	1.25	1/2	2	1 (added after split)	100	250	2	200
P4	High	1	1/4	4	1 (added after split)	100	500	4	400

QuantIMS Final Volume (µL) = Extract Delivery Volume (µL) x Prep Split Factor x (nominal extract volume) / Extract Delivery Volume (µL) (1)
(1) Nominal extract volume is 50 µL for protocol 1 and 100 µL for protocols 2, 3 and 4.

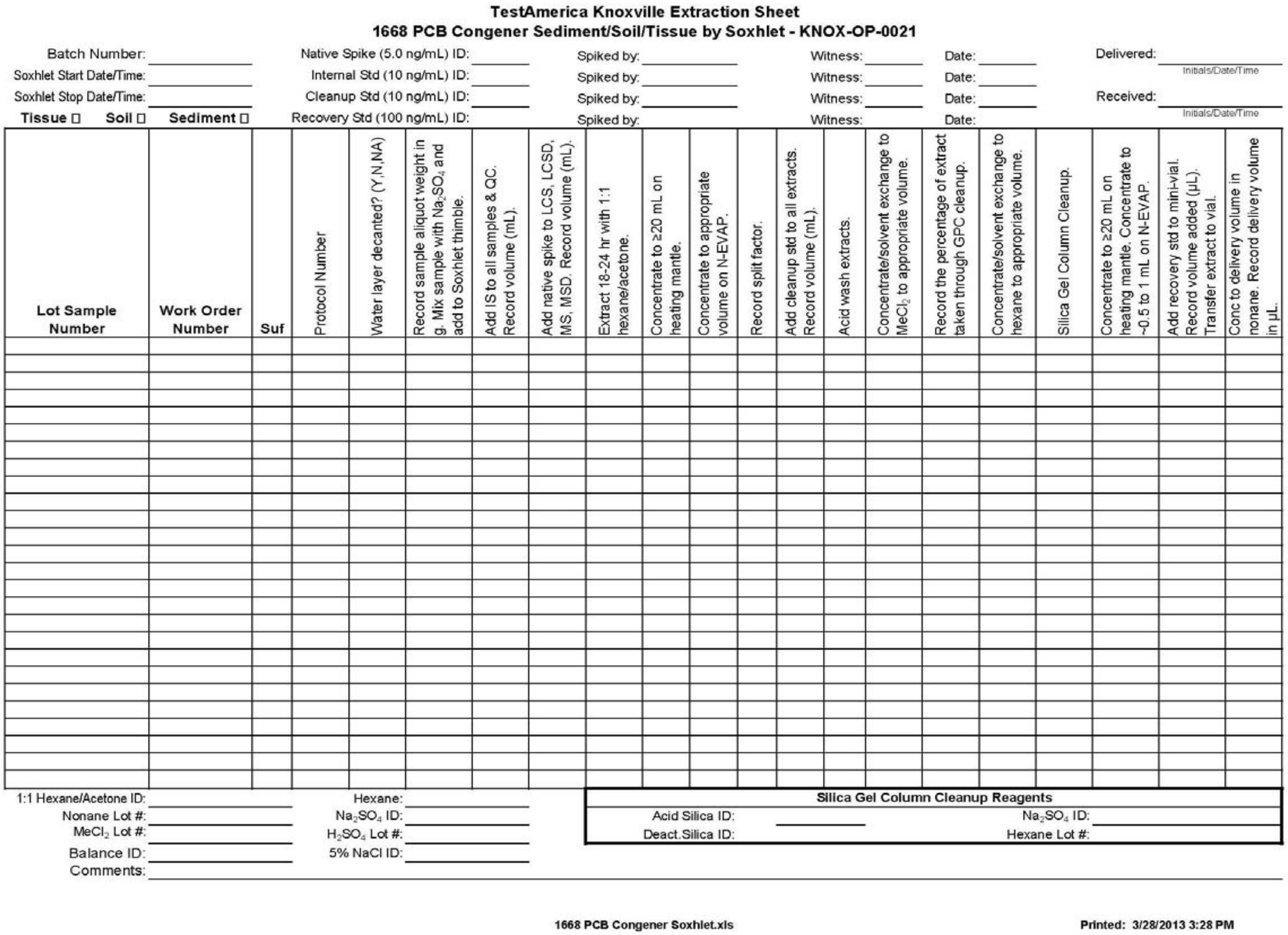
Table 6 - Assignment of Sample Preparation Protocols for Microwave Extraction

1668 Protocol	Matrix	Protocol Name	Sample Amount Extracted (g)	Fraction of Extract Cleaned	IS Added (mL)	Cleanup Std Added (mL)	Recovery Std Added (µL)	Extract Delivery Volume (µL)	Prep Split Factor	QuantIMS Final Volume (µL)
M1	Soil	Clean	10	1/2	1	0.5 (added after split)	50	50	2	100
M1	Sediment	Clean	10 g wet wt (max) (5 g dry wt) (5 g for blank/LCS)	1	1	1	50	50	1	50
M2	Soil or Sediment	Low	2	1	1	1	100	100	1	100
M3	Soil or Sediment	Medium	1.25	1/2	2	1 (added after split)	100	250	2	200
M4	Soil or Sediment	High	1	1/4	4	1 (added after split)	100	500	4	400

Table 7– Typical Microwave Conditions

Parameter	Typical Conditions
Temperature program	20 min. ramp to 130 degrees Celsius; 30 min. hold at 130 degrees Celsius

Appendix I – Example Extraction Benchsheet



TestAmerica Knoxville Extraction Sheet
1668 PCB Congener Sediment/Soil/Tissue by Soxhlet - KNOX-OP-0021

Batch Number: ____________
Soxhlet Start Date/Time: ____________
Soxhlet Stop Date/Time: ____________
Tissue ☐ Soil ☐ Sediment ☐

Native Spike (5.0 ng/mL) ID: ________ Spiked by: ________ Witness: ________ Date: ________
Internal Std (10 ng/mL) ID: ________ Spiked by: ________ Witness: ________ Date: ________
Cleanup Std (10 ng/mL) ID: ________ Spiked by: ________ Witness: ________ Date: ________
Recovery Std (100 ng/mL) ID: Spiked by: Witness: Date:

Delivered: ____________ Initials/Date/Time
Received: ____________ Initials/Date/Time

Lot Sample Number	Work Order Number	Suf	Protocol Number	Water layer decanted? (Y,N,NA)	Record sample aliquot weight in g. Mix sample with Na_2SO_4 and add to Soxhlet thimble.	Add IS to all samples & QC. Record volume (mL).	Add native spike to LCS, LCSD, MS, MSD. Record volume (mL).	Extract 18-24 hr with 1:1 hexane/acetone.	Concentrate to ≥20 mL on heating mantle.	Concentrate to appropriate volume on N-EVAP.	Record split factor.	Add cleanup std to all extracts. Record volume (mL).	Acid wash extracts.	Concentrate/solvent exchange to $MeCl_2$ to appropriate volume.	Record the percentage of extract taken through GPC cleanup.	Concentrate/solvent exchange to hexane to appropriate volume.	Silica Gel Column Cleanup.	Concentrate to ≥20 mL on heating mantle. Concentrate to ~0.5 to 1 mL on N-EVAP.	Add recovery std to mini-vial. Record volume added (µL). Transfer extract to vial.	Conc to delivery volume in nonane. Record delivery volume in µL.

1:1 Hexane/Acetone ID: ____________
Nonane Lot #: ____________
$MeCl_2$ Lot #: ____________
Balance ID: ____________
Comments: ____________

Hexane: ____________
Na_2SO_4 ID: ____________
H_2SO_4 Lot #: ____________
5% NaCl ID: ____________

Silica Gel Column Cleanup Reagents

Acid Silica ID: ____________ Na_2SO_4 ID: ____________
Deact.Silica ID: Hexane Lot #:

1668 PCB Congener Soxhlet.xls **Printed: 3/28/2013 3:28 PM**

Appendix II - Guidelines for the Spike Witnessing Process

- Make sure there are no distractions, for example, phone calls, checking samples on water bath, people coming in to ask questions.
- The person spiking must tell the person who is witnessing what is being spiked and how much. Make sure the paperwork shows the spike amounts and spike IDs.
- The person witnessing should make sure they know and understand what is to be spiked and how much. Check the paperwork to verify.
- Check the syringe for air bubbles and also check the spike volume.
- It is a good idea to also check for cracks in the glassware.
- If client service requires spiking to occur when another analyst is not available, a witness is not required. In this case, the analyst will serve as his/her own witness, and must carefully double check the spike solutions and spike amounts added to the client samples and associated quality control samples. The analyst enters his/her initials as both the analyst and witness.

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TESTAMERICA KNOXVILLE

STANDARD OPERATING PROCEDURE

TITLE: Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HRMS

(SUPERSEDES: KNOX-ID-0013, Revision 11)

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Copyright Information:

1 Scope and Application

1.1 This procedure is designed to meet analytical program requirements where HRGC/HRMS analysis of polychlorinated biphenyl (PCB) isomers is specified. The procedure is used by TestAmerica Knoxville for the qualitative and quantitative measurement of all 209 PCB congeners in a variety of environmental matrices at part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations. This procedure is based on EPA method 1668A, 1668B and 1668C.

1.2 The compounds listed in Table 1 may be determined by this procedure. The detection limits and quantitation levels in this method are usually dependent on the level of interferences rather than instrumental limitations. The estimated minimum levels (EMLs) in Table 4 are the levels at which the PCBs can be determined with only common laboratory interferences present. The actual limits of detection and quantitation will vary depending on the complexity of the matrix.

1.3 The Low Calibration Levels (LCLs) of the method are listed in Table 3 for individual congeners. Analysis of a one-tenth aliquot of the sample permits measurements of concentrations up to 10 times the upper calibration range. Samples containing concentrations of PCBs that are greater than ten times the upper calibration are analyzed by protocols designed for such concentration levels.

1.4 The GC/MS portions of this method are for use only by analysts experienced with HRGC/HRMS or under the close supervision of such qualified persons. Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in section 9.1.

1.5 This procedure is a "performance-based" method. These reference methods allow modifications to overcome interferences or lower the cost of measurements, if all performance criteria in the methods are met and method equivalency is established. Deviations from the referenced methods have been incorporated into this procedure and are listed in section 17.1. Deviations to this procedure are only allowed as specified in section 11.1.

1.6 Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCBs. It is the responsibility of the laboratory personnel to ensure that safe handling procedures are employed. Section 5 of this procedure discusses safety procedures.

2 Summary of Method

2.1 All solid and semi-solid samples are screened by GC/ECD prior to extraction. Other sample matrices may be screened if the potential for high congener concentrations exists. Variations in sample size, spiking levels and final volume are established based on the screening result.

2.2 After sample extraction, cleanup, and concentration, recovery standards are added to each extract, and an aliquot of the extract is injected into the gas chromatograph. The analytes are separated by the GC and detected by a high-resolution (≥10,000) mass spectrometer. Two exact masses are monitored for each analyte.

2.3 An individual PCB congener is identified by comparing the GC retention time and ion-abundance ratio of two exact masses with the corresponding retention time of an authentic standard and the theoretical or acquired ion-abundance ratio of the two exact masses.

2.4 Quantitative analysis is performed using selected ion current profile (SICP) areas using the internal standard technique.

2.5 The quality of the analysis is assured through reproducible calibration and verification of the extraction, cleanup, and GC/MS systems.

3 Definitions

These definitions and purposes are specific to this method but conform to common usage as much as possible.

Note: Terminology differences existing in some isotope dilution reference methods regarding the functionality of the labeled analogs may lead to confusion. For example, EPA's Office of Solid Waste methods (8280, 8290) use the term "Internal Standards" to describe the labeled analogs which are added to the sample prior to extraction and used to quantitate the native targets. EPA's Office of Water methods (1613B, 1668) use the term "Labeled Analogs" to describe these same compounds while using the term "Internal Standards" to describe the labeled analogs which are added to the extract just prior to analysis and used to quantitate the recovery of the labeled analogs added before extraction. EPA's Office of Solid Waste methods (8280, 8290) uses the term "Recovery Standards" to describe these later labeled analogs.

The terminology conventions established by the EPA's Office of Solid Waste methods (8280, 8290) are used in the laboratory for all Standard Operating Procedures and internal communications as defined in this section.Analyte – A PCB tested for by this method. The analytes are listed in Table 1.

3.1 Calibration verification standard (VER) – The mid-point calibration standard (CS3) that is used to verify calibration. See Table 6a.

3.2 CB – Chlorinated biphenyl congener. One of the 209 individual chlorinated biphenyl congeners determined using this method. The 209 CBs are listed in Table 1.

3.3 Cleanup Standard – Isotopically labeled compounds that are added to samples, blanks, quality control samples, and calibration solutions. They are added to the samples after extraction but prior to extract cleanup, and are used to assess the efficiency of the cleanup procedures.

3.4 Congener – Any member of a particular homologous series, for example, 2,2’-DiCB.

3.5 CS0.5, CS1, CS2, CS3, CS4, CS5 – See Calibration standards and Table 6a.

3.6 Estimated Detection Limit (EDL) – The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level (noise level).

3.7 Estimated Maximum Possible Concentration (EMPC) – The calculated concentration of a signal having the same retention time as a PCB congener but which does not meet all of the other qualitative identification criteria defined in the method.

3.8 Estimated Minimum Level (EML) – The lowest concentration at which an analyte can be measured reliably with common laboratory interferences present. This is the reporting limit (RL).

3.9 Field blank – An aliquot of reagent water or other reference matrix that is placed in a sample container in the laboratory or the field, and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample.

3.10 GC – Gas chromatograph or gas chromatography.

3.11 Homologous Series – A series of compounds in which each member contains the same number of chlorine atoms. The members of the series are called homologs.

3.12 HRGC – High resolution GC.

3.13 HRMS – High resolution MS.

3.14 ICV – Initial Calibration Verification Standard. A calibration standard from a second source, traceable to a national standard if possible. The ICV is analyzed after the Initial calibration to verify the concentration of the initial calibration standards.

3.15 Internal Standards (IS) – Isotopically labeled analogs of the target analytes that are added to every sample, blank, quality control spike sample, and calibration solution. They are added to the sample before extraction and are used to calculate the concentration of the target analytes or detection limits.

3.16 IPR (also known as IDOC) – Initial precision and recovery; four aliquots of the PAR standard analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed prior to the first time this method is used and any time the method or instrumentation undergoes significant modification.

3.17 Isomer – PCB congeners that contain the same number of chlorine atoms, but differ in the structural arrangement of the chlorine atoms. For example, PCB-4 and PCB-9 are isomers.

3.18 Laboratory blank – See Method blank.

3.19 Laboratory control sample (LCS) – See ongoing precision and recovery standard (OPR).

3.20 Laboratory reagent blank – See Method blank.

3.21 Level of Chlorination (LOC) Congeners – The first and last eluting congeners in each homolog (or level of chlorination). (For the SPB-Octyl Column the LOC Congeners are 1, 3; 4, 15; 19, 37; 54, 77; 104, 126; 155, 169; 188, 189; 202, 205; 208, 206; 209)

3.22 Method blank – An aliquot of a clean test matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

3.23 Minimum Level (ML) – The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

Note: EML is the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present

3.24 MS – Mass spectrometer or mass spectrometry.

3.25 OPR (also know as ODOC) – Ongoing precision and recovery standard (OPR); a laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

3.26 PAR – Precision and recovery standard; secondary standard that is diluted and spiked to form the IPR and OPR.

3.27 PFK – Perfluorokerosene; the mixture of compounds used to calibrate the exact mass scale of the HRMS.

3.28 Primary dilution standard – A solution containing the specified analytes that is purchased or prepared from stock solutions and diluted as needed to prepare calibration solutions and other solutions.

3.29 Quality control check sample (QCS) – A sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process.

3.30 PCB – Polychlorinated biphenyl.

3.31 Reagent water – Water demonstrated to be free from the analytes of interest and potentially interfering substances at the method minimum level for the analyte.

3.32 Recovery Standard (RS) – Isotopically labeled compounds which are added to every sample, blank, and quality control spike sample extract prior to analysis. They are used to measure the recovery of the internal standards and the cleanup standards.

3.33 Relative Percent Difference (RPD) – A measure of the difference between two values normalized to one of the values. It is used to determine the accuracy of the concentration measurements of second source verification standards.

3.34 Relative standard deviation (RSD) – The standard deviation times 100 divided by the mean. Also termed "coefficient of variation."

3.35 RF or RRF (Relative Response Factor) - The ratio of the response of the mass spectrometer to a known amount of a compound relative to that of a known amount of a reference standard as measured in the initial and continuing calibrations. It is used to determine instrument performance and it is used to calculate the concentration of target analytes, internal standard recoveries, or detection limits in samples, blanks, and quality control samples. See Section 10.3.4.2.

3.36 SICP – Selected ion current profile.

3.37 SPE – Solid-phase extraction; an extraction technique in which an analyte is extracted from an aqueous sample by passage over or through a material capable of reversibly adsorbing the analyte. Also termed liquid-solid extraction.

3.38 Specificity – The ability to measure an analyte of interest in the presence of interferences and other analytes of interest encountered in a sample.

3.39 Stock solution - A solution containing an analyte that is prepared using a reference material traceable to EPA, the National Institute of Science and Technology (NIST), or a source that will attest to the purity and authenticity of the reference material.

3.40 Surrogate Standards (SS) - Isotopically labeled compounds that are added to XAD samples and calibration solution. They are added to XAD sampling tubes before sampling and are used to measure sampling and recovery efficiency.

3.41 Toxic Congeners (or Toxic Isomers) – PCBs determined by the World Health Organization and USEPA to have dioxin-like toxicity. (PCBs 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, 189)

3.42 Toxic/LOC Congeners – PCBs belonging to either the Toxic Congeners list or the LOC Congeners list.

3.43 VER – See Calibration verification standard.

3.44 Additional definitions can be found in the TestAmerica Knoxville Quality Assurance Manual (QAM), current revision.

4 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. Where possible, glassware is cleaned by extraction or solvent rinse. The non-coplanar PCB congeners 105, 114, 118, 123, 156, 157, 167, and 180 have been shown to be very difficult to completely eliminate from the laboratory at the minimum levels in this method, and baking of glassware in a kiln or furnace at 450 - 500°C may be necessary to remove these and other contaminants.

4.2 All materials used in the analysis shall be demonstrated to be free from interferences by running laboratory method blanks (section 9.5) initially and with each sample batch.

4.3 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCBs. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxy-diphenyl ethers, benzylphenyl ethers, polynuclear aromatics, and pesticides. Because very low levels of PCBs are measured by this method, the elimination of interferences is essential. Cleanup steps can be used to reduce or eliminate these interferences and thereby permit reliable determination of the PCBs at the levels shown in Table 3.

5 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Corporate Safety Manual), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately.

5.3 The effluents of sample splitters for the gas chromatograph and roughing pumps on the mass spectrometer must be vented to the laboratory hood exhaust system or must pass through an activated charcoal filter.

5.4 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them or use thermal protection when working on them while they are above room temperature.

5.5 The mass spectrometer is under high vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source. Alternatively, the source may be removed from the vacuum manifold through a vacuum interlock.

5.6 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power. If the work involved requires measurement of voltage supplies, the instrument may be left on.

5.7 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. 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 the method can be found in the reagents and materials section. 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.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen, Irritant	25 ppm-TWA, 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable, Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol	Flammable, Poison, Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Toluene	Flammable, Poison, Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Nonane	Flammable	None established	Harmful if inhaled/swallowed. Vapor/mist is irritating to eyes, mucous memebranes and upper respiratory tract. Causes skin irritiation.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.8 Chemicals that have been classified as **carcinogens**, **potential carcinogens**, or **mutagens include**: methylene chloride, polychlorinated biphenyls, and toluene. The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.)

5.9 Chemicals known to be **flammable** are: acetone, hexane, nonane and toluene.

5.10 Exposure to chemicals will be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.11 The preparation of all standards will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.12 Personal Hygiene: Thorough washing of hands and forearms is recommended after each manipulation and before breaks (coffee, lunch, and shifts).

5.13 Confinement: Work areas should be isolated and posted with signs. Glassware and tools should be segregated. Bench tops should be covered with plastic backed absorbent paper.

5.14 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.

5.15 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

5.16 All work must be stopped in the event of a known or potential compromise to the health or safety of laboratory personnel. The situation must be reported immediately to a laboratory supervisor.

6 Equipment and Supplies

6.1 Gas chromatograph - Shall have splitless or on-column injection port for capillary column, temperature program with isothermal hold, and shall meet all of the performance specifications in Section 10.

6.1.1 Column #1 – 30±5-m long x 0.25±0.02-mm ID; 0.25-µm film SPB-Octyl (Supelco 2-4218, or equivalent).

6.2 Mass spectrometer - Electron impact ionization, shall be capable of repetitively selectively monitoring 20 exact masses minimum at high resolution (≥10,000) during a period less than 1.0 second, and shall meet all of the performance specifications in Section 10.

6.3 GC/MS interface - The mass spectrometer (MS) shall be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beams.

6.4 Data system - Capable of collecting, recording, and storing MS data.

7 Reagents and Standards

CAUTION: Refer to Material Safety Data Sheets (MSDS) for specific safety information on chemicals and reagents prior to use or as needed.

CAUTION: During preparation of reagents, associates shall wear lab coat, gloves, safety glasses with side shields, and laboratory approved shoes as a minimum. Reagents shall be prepared in a fume hood.

7.1 Solvents - Acetone, toluene, n-hexane, methanol, methylene chloride, and nonane; pesticide quality.

7.2 Perfluorokerosene (PFK) high boiling mass spectroscopy grade; bp 210-260°C; d^{20}_4 1.94; n^{20}_D 1.330; Fluka (Catalog No. - 77275).

7.3 $^{13}C_{12}$ Labeled PCB Congener Standards: Obtained as individual Certified Reference Standards from Cambridge Isotope Laboratories (CIL, Andover Massachusetts) and Wellington Laboratories (Guelph, Ontario, Canada). Refer to Table 5b for a list of individual standards. These standards are purchased at 40 µg/mL or 50 µg/mL in nonane. If the chemical purity is 98% or greater, the stated manufacturer concentration may be used without correction to compute the concentration of the standard. Once a standard ampoule has been vortexed and opened, the solution is transferred to an amber glass vial with a Teflon®-lined screw cap. When not being used, standards are stored in a dark box at room temperature. These purchased standards are used to prepare the following mixed stock solutions and spiking solutions:

7.3.1 Internal Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled internal standards listed in Table 5b, to a concentration of 1000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.3.2 Internal Standard Spiking Solution: Prepared by diluting the 1000 ng/mL internal standard stock solution to a concentration of 10 ng/mL in acetone. 1.0 to 4.0 mL of this solution is added to each solid/tissue sample prior to extraction. Refer to Table 12, “Assignment of Sample Preparation Protocols” to determine the exact volume to add. Prior to extraction, aqueous samples are spiked with internal standard spiking solution at 1/5 the levels of solid/tissue samples (typically 20 uL).

7.3.3 Recovery Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled recovery standards listed in Table 5b to a concentration of 1000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.3.4 Recovery Standard Spiking Solution: Prepared by diluting the 1000 ng/mL recovery standard stock solution to a concentration of 100 ng/mL in nonane. Fifty to 100 µL of this spiking solution is added to each solid/tissue sample extract prior to analysis (refer to Table 12), whereas, 20 µL is added to each aqueous sample extract.

7.3.5 Cleanup Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled cleanup standards listed in Table 5b, to a concentration of 5000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.3.6 Cleanup Standard Spiking Solution: Prepared by diluting the 5000 ng/mL cleanup standard stock solution to a concentration of 10 ng/mL in hexane. One half to 1.0 mL of this solution is added to each solid/tissue sample extract prior to cleanup (refer to Table 12), whereas, 0.20 mL is added to each aqueous sample extract.

7.3.7 Sampling Surrogate Standard Stock Solution: Prepared by diluting the individual

$^{13}C_{12}$ labeled sampling surrogate standards listed in Table 5b to a concentration of 5000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.3.8 Sampling Surrogate Spiking Solution: Prepared by diluting the 5000 ng/mL sampling surrogate stock solution to a concentration of 50 ng/mL in nonane.

7.4 Native PCB Congener Standard Mix: Obtained as a Certified Reference Standard from Accustandard (New Haven, CT). This standard contains all 209 PCB congeners at 4000 ng/mL in nonane. If the chemical purity is 98% or greater, the weight may be used without correction to compute the concentration of the standard. Once a standard ampoule has been vortexed and opened, the solution is transferred to an amber glass vial with a Teflon®-lined screw cap. When not being used, the standard is stored in a dark box at room temperature. This purchased standard is used to prepare the following native stock solution and spiking solution:

7.4.1 Native PCB Congener Stock Solution: Prepared by diluting the 4000 ng/mL native PCB congener standard mix to a concentration of 40 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.4.2 LCS Spiking Solution: Prepared by diluting the 4000 ng/mL native PCB congener standard to a concentration of 5.0 ng/mL in acetone. One mL of this solution is added to each solid/tissue LCS prior to extraction, whereas, 0.20 mL is added to each aqueous LCS.

7.5 Calibration Standard Solutions (CS 0.5 through CS 5) are prepared by dilution of the native PCB congener standards in section 7.4 and 7.4.1 and the labeled standards in section 7.3 in nonane. Table 6a shows the calibration solution analytes and final concentrations. Table 6b provides details for preparation of these calibration solutions.

7.5.1 This series of solutions is used to establish linearity and relative response factors for all compounds in the initial calibration solutions. These RRFs are used to quantify PCB congeners in the calibration verification (VER) and all samples. The CS3 standard is used for calibration verification. The VER solution is also used to verify chromatographic performance.

7.6 PCB Congener Mix 1 through 5 standard solutions containing all 209 isomers are Certified Reference Standards (Accustandard Product No's. M-1668A-1, M-1668A-2, M-1668A-3, M-1668A-4, M-1668A-5). Stock solutions are purchased at 250-750 µg/mL in isooctane. Once the ampoule has been sonicated and opened, the solution is transferred to an amber glass vial with Teflon®-lined cap and is used as received. These five mixes are run in triplicate to determine the retention times for each of the congeners and which congeners will co-elute for each new SPB Octyl column used.

7.6.1 209 PCB ICAL Verification stock solution: Prepared by combining the five PCB Congener Mixes referred to in section 7.6 and diluting to a concentration of 5000-15000 ng/mL in nonane.

7.6.2 Initial Calibration Verification Standard. This is a single solution containing all 209 individual PCBs as well as internal standards and recovery standards at the following concentrations:

- mono, di, and tri CBs at 50 ng/mL
- tetra, penta, hexa and hepta CBs at 100 ng/mL
- octa, nona, and deca CBs at 150 ng/mL
- internal standards and recovery standards are at the same concentration as the calibration standards (CS 0.5 – CS 5).

This solution is always analyzed immediately after the initial calibration.

7.6.2.1 Combine 100 uL of 209 PCB ICAL verification stock solution (Section 7.6.1) (equivalent to 20 uL of each mix) with 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled internal standard stock solution, 100 uL of a 1/10 dilution of the 5000 ng/mL $^{13}C_{12}$ labeled cleanup standard stock solution, 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled recovery standard stock solution and 600 uL of nonane to produce the concentrations listed in section 7.6.2.

7.6.3 Retention Time Calibration Mixes: These are 5 solutions injected in triplicate to establish the retention time data referenced in section 10.2.3. Combine 20 uL of the Accustandard PCB Congener Mix 1 (Section 7.6) with 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled internal standard stock solution, 100 uL of a 1/10 dilution of the 5000 ng/mL $^{13}C_{12}$ labeled cleanup standard stock solution, 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled recovery standard stock solution and 600 uL of nonane to produce the concentrations listed in section 7.6.2. Repeat the process using the Accustandard PCB Congener Mixes 2 through 5.

7.7 QC Check Sample - A QC Check Sample should be obtained from a source independent of the calibration standards. This check sample is a certified standard reference material (SRM) containing the PCBs in known concentrations in a sample matrix similar to the matrix under test. The National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland has an SRM 1944 – New York/New Jersey Waterway Sediment that the NYSDEC recommends for use.

8 Sample Collection, Preservation and Storage

8.1 Sampling is not performed for this method by TestAmerica Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, “Sample Receipt and Login”, current revision.

8.2 Holding Times

8.2.1 Store sample extracts in a dark box at room temperature until analyzed. If stored in the dark at room temperature, sample extracts may be stored for up to one year.

9 Quality Control

9.1 Initial precision and recovery (IPR) or initial demonstration of capabilty (IDOC) samples are analyzed to demonstrate the ability to generate acceptable precision and accuracy.

9.1.1 For aqueous samples, extract, clean, concentrate, and analyze four 1-L aliquots of reagent water spiked with internal standards, cleanup standard, recovery standard and the LCS spiking solution, according to the procedures in section 11. For solid/tissue samples, extract, clean, concentrate, and analyze four aliquots of sodium sulfate/corn oil spiked with internal standards, cleanup standard, recovery standard and the LCS spiking solution, according to the procedures in section 11. All steps that are to be used for processing samples, including preparation, extraction and cleanup, shall be included in this test.

9.1.2 Using the results of the set of four analyses, compute the average percent recovery (%R) of the extracts and the relative standard deviation (%RSD) of the concentration in ng/L (aqueous) and ng/g (solid) for each compound.

9.1.3 For each PCB and labeled compound, compare the %RSD and %R with the corresponding limits for initial precision and recovery in the approprirate Table 10A or 10B. If the RSD and %R for all compounds meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, any individual %RSD exceeds the precision limit or any individual %R falls outside the range for accuracy, system performance is unacceptable for that compound. Correct the problem and repeat the test.

9.2 Internal Standards

9.2.1 Every sample, blank, and QC sample is spiked with internal standards. Internal standard recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. When properly applied, results from isotope dilution techniques are independent of recovery. The recovery of each internal standard should be within the limits in Table 10A or 10B depending on the method revision being used. If the recovery is outside these limits the following corrective action should be taken:

- Check all calculations for error.
- Ensure that instrument performance is acceptable; if applicable, a daily CCV will be analyzed to confirm instrument performance.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- If the recovery of any internal standard is less than the limits indicated in Tables 10A and 10B, calculate the S/N ratio of the internal standard. If the S/N is > 10 and the estimated detection limits (EDLs) are less than the estimated minimum levels (EMLs), report the data as is with qualifiers in the report and a discussion in the case narrative. If the S/N is < 10 or the estimated detection limits (EDLs) are greater than the estimated minimum levels (EMLs), re-extract and reanalyze the sample. If the ion chromatogram of the PFK lock mass indicates ion suppression in the region where the internal standard elutes, reanalyzing the extract at up to a 1/10 dilution may improve the internal standard recovery. If the poor internal standard recovery is judged to be a result of sample matrix, a reduced portion of the sample may be re-extracted or

additional cleanups may be employed. The decision to reanalyze or flag the data should be made in consultation with the client.

9.2.2 Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.3 Cleanup Standards: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled cleanup standards after extraction but prior to extract cleanup. They are used to assess the efficiency of the cleanup procedures.

9.4 Recovery Standards: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled recovery standards prior to analysis. They are used to measure the recovery of the internal standards and the cleanup standards.

9.5 Method Blanks

9.5.1 A laboratory method blank must be run along with each analytical batch of 20 or fewer samples. The method blank consists of reagent water for aqueous samples, sodium sulfate for solid and tissue samples, processed in the same manner and at the same time as the associated samples. The method blank is used to identify any background interference or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data. Analyze the blank immediately after analysis of the LCS to demonstrate freedom from contamination. The method blank should not contain any of the compounds of interest at a concentration above the estimated minimum level (EML) shown in Table 4.

9.5.2 Corrective action is required when compounds of interest are detected in the method blank above the EML. Corrective action may include reanalysis of the method blank. Contact the Project Manager to determine further corrective action. At a minimum, all associated results are qualified with a B flag. Re-extraction and reanalysis of all samples associated with a contaminated method blank is required if requested by the client or Project Manager. Investigation of the source of the method blank contamination will be initiated before further samples are extracted.

9.5.3 The method blank must have acceptable internal standard recoveries. If internal standard recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If internal standard recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.5.4 Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.6 Instrument Blank

9.6.1 Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed.

9.6.2 An instrument blank consists of solvent with the internal standards and recovery standards added. It is evaluated in the same way as the method blank.

9.7 Laboratory Control Sample – A laboratory control sample (LCS) is prepared and analyzed with every batch of 20 or fewer samples. All analytes must be within established control limits specified in the appropriate Table 10A or 10B. The LCS is spiked with the compounds listed in Table 5a.

9.7.1 If any analyte in the LCS is outside the control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.
- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported and the failure is documented in the project narrative.

9.8 QC Check Sample - Analyze the QC Check Sample (section 7.7) periodically to assure the accuracy of calibration standards and the overall reliability of the analytical process. It is suggested that the QC Check Sample be analyzed at least annually.

10 Calibration and Standardization

10.1 Three types of calibration procedures are required. The first type establishes retention times, relative retention times and relative retention time windows to be used during the subsequent calibrations and analyses. The second type, initial calibration, is required to establish response factors and is required before any samples are analyzed. It may be required intermittently throughout sample analyses as dictated by the results of continuing calibration procedures described below. The third type, continuing calibration, consists of analyzing the continuing calibration verification solution (VER). No samples are to be analyzed until acceptable calibration as described in sections 10.2, 10.3 and 10.4 is demonstrated and documented.

10.2 Retention Time Calibration

Retention time calibration is required if the retention time criteria cannot be met.

10.2.1 The absolute retention time of CB 209 must exceed 55 minutes. Otherwise the GC temperature program must be adjusted and the test repeated until the requirement is met.

10.2.2 **NOTE**: When adjusting chromatographic conditions, the resolution requirements of sections 10.4.5.7 to 10.4.5.8 must be maintained.

10.2.3 Tune the instrument to meet the mass resolution and mass accuracy requirements of section 10.3.2. Document the resolution and accuracy.

10.2.4 Analyze 2µL of each of the five individual PCB mixtures (section 7.6.3). Repeat the series twice more in succession to provide 3 runs of each mix. It is not necessary to interrupt this analytical sequence to perform a 12-hour resolution check. Set the switch-points for the MID descriptors. The switch-points must be set to insure that the first and last eluting isomer of each homolog group and the labeled internal standards are acquired properly. Determine the average retention time of each PCB congener using the elution order information in Table 11.

NOTE 1: PCB Mixture 5 (M-1668A-5) contains the first and last eluting isomer in each homolog group for the SPB-Octyl column (see Table 7).

NOTE 2: Laboratory data has indicated that the SPB-Octyl column can exhibit significant differences in performance from column to column. It has also been indicated that the column's performance can change significantly due to oxidation with subsequent changes in congener retention times and elution order. The individual PCB mixtures should be analyzed whenever the column's performance or specific congeners retention times are in doubt.

10.2.5 Calculate the relative retention times for all native and labeled congeners, using their retention time references from Table 2 (RT Ref). Calculate the relative retention time for each run in which the congener and its retention time reference are present (i.e., three RRTs will be calculated for each native congener. Fifteen RRTs will be calculated for each internal standard.) Use the calculated average retention times for all native and labeled congeners as the RT calibration source in the calculation software.

10.2.6 Calculate the relative retention time window using the absolute retention time windows (RT Window) from Table 2.

$$\text{RRT Limit Low} = \frac{RT_A - (RT_{WIN}/2)}{RT_{IS}}$$

$$\text{RRT Limit High} = \frac{RT_A + (RT_{WIN}/2)}{RT_{IS}}$$

Where:

RT_A = Average retention time of analyte.

RT_{IS} = Average retention time of RT reference.

RT_{WIN} = Absolute RT window in seconds from Table 2.

10.2.7 A single pair of RRT limits is used for all congeners in coeluting set. Use the RRT Limit Low that was calculated for the first eluting congener, and the RRT Limit High calculated for the last eluting congener (in the coelution set).

10.3 Initial Calibration

Initial calibration is required before any samples are analyzed for PCBs. Initial calibration is also required if any continuing calibration (section 10.4) does not meet the required criteria in section 10.4.5 after routine maintenance.

10.3.1 Prepare multi-level calibration standards containing the compounds and concentrations as specified in Table 6a. Calibration standards should be stored at room temperature and preferably in amber vials. Calibration standard solutions have an expiration date of ten (10) years from date of receipt based on stability of PCBs and history of vendor's recertification of standard lots.

10.3.2 Establish operating parameters for the GC/MS system (suggested operating conditions are displayed in Figure 1 and Figure 2). By using a PFK molecular leak, tune the instrument (see the appropriate instrument manufacturer's operating manual for tuning instructions) to meet the minimum resolving power of 10,000 (10 percent valley) at mass 342.97924 (PFK). For each MID descriptor group, monitor and record the mass resolution and exact masses of three reference peaks covering the mass range of the descriptor (see below). By using peak matching techniques, verify that the deviation between the exact mass and the theoretical mass for each mass monitored is less than 5 ppm. Iteratively adjust operating parameters and tuning values until the mass resolution and mass accuracy criteria are met for each ion. Document the mass resolution and mass accuracy for each of MID group ion sets. Because of the extensive mass range covered in each MID group, it may not be possible to maintain 10,000 resolution throughout the mass range of the MID group. Therefore, resolution must be greater than 8,000 throughout the mass range and must be greater than 10,000 in the center of the mass range for each MID group. The minimum resolution of 10,000 must be met for mass 342.97924.

MID Group 1 PFK ions - 192.9888, 230.98563, 280.98243
MID Group 2 PFK ions - 268.98243, 292.98243, 380.97605
MID Group 3 PFK ions - 342.97924, 380.97605, 430.97285
MID Group 4 PFK ions - 404.97604, 442.97285, 530.96646

10.3.3 Inject a 2 µL aliquot of the CS 0.5 calibration solution.

10.3.3.1 Ion abundance ratios, minimum levels, and signal-to-noise ratios.

10.3.3.1.1 Measure the SICP areas for each congener or congener group, and compute the ion abundance ratios at the exact masses specified in Table 8. Compare the computed ratio to the theoretical ratio given in Table 9.

10.3.3.1.2 All Toxic/LOC and labeled compounds in the CS-0.5 standard must be within the QC limits in Table 9 for their respective ion abundance ratios; otherwise, the mass spectrometer must be adjusted and this test repeated until the mass ratios fall within the limits specified. If the adjustment alters the resolution of the mass spectrometer, resolution must be verified (Section 10.3.2) prior to repeat of the test.

10.3.3.1.3 The peaks representing the CBs and labeled compounds in the CS-0.5 calibration standard must have signal-to-noise ratios (S/N) ≥ 10; otherwise, the mass spectrometer must be adjusted and this test repeated until the signal to noise criteria are met.

10.3.3.1.4 An exception to the ion abundance ratio and signal to noise ratio requirements is the secondary ion for dichlorinated biphenyls (mass 223.9974). High background from PFK fragments at 223.9974 results in noise levels which exceed 10% of the signal height at levels that are reliably quantifiable.

10.3.4 Analyze 2 µL of each of the other calibration standards.

10.3.4.1 Isomer specificity

10.3.4.1.1 Use the CS-3 calibration standard to evaluate column performance. The toxic isomers must be uniquely resolved from all other congeners. Isomers may be unresolved so long as they have the same toxicity equivalency factor (TEF) and response factor and so long as these unresolved isomers are uniquely resolved from all other congeners. For example, the SPB-Octyl column achieves unique GC resolution of all Toxics except congeners with IUPAC numbers 156 and 157. This isomeric pair is uniquely resolved from all other congeners and these congeners have the same TEF and response factor.

10.3.4.1.2 Evaluate and document the percent valley between PCBs 34 and 23. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.3.4.1.3 Evaluate and document the percent valley between PCBs 187 and 182. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.3.4.1.4 Classify each congener as resolved or as a member of a coelution set. To be documented as resolved, the valleys between any two isomers must be less than 40 percent of the height of the shorter of the two adjacent peaks. Each member of a coelution set is designated with a qualifier in the format of CXXX, where XXX = the lowest numbered congener in the set. For example, if PCB 156 and PCB 157 coelute, qualify PCB 157 with "C156".

10.3.4.2 Calculate the RRF of each compound of interest (target analytes, coelution sets, internal standards, cleanup standards, and surrogate standards) vs. the appropriate reference standard (as specified in Table 2) using the following equation:

$$RRF = \frac{As \times Cis}{Ais \times Cs}$$

Where:

As = sum of the areas of the quantitation ions of the compound of interest.
Ais = sum of the areas of the quantitation ions of the appropriate reference standard.
Cis = concentration of the appropriate reference standard.
Cs = concentration of the compound of interest.

NOTE: When calculating the RRF for a coelution set, sum the areas of all isomers in the set. Use the resulting RRF for all congeners within the set.

10.3.4.3 Calculate the mean relative response factor (mean RRF) and the percent relative standard deviation (%RSD) of the response factors for each compound of interest in the six calibration standard solutions using the following equations:

$$\overline{RRF}_{n=6} = \frac{1}{n} \times \sum_{i=1}^{n} RF_i$$

$$\%RSD_{n=6} = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RRF}\right)^2}{n-1}} \times \frac{100}{\overline{RRF}}$$

10.3.4.4 Criteria for Acceptable Calibration - The criteria listed below for acceptable calibration must be met before sample analyses are performed. If acceptable initial calibration is not achieved, identify the root cause, perform corrective action, and repeat the initial calibration. If the root cause can be traced to problems with an individual analysis within the calibration series, repeat the individual analysis and recalculate the percent relative standard deviation. If the calibration is acceptable, document the problem and proceed, otherwise repeat the initial calibration.

10.3.4.5 The percent relative standard deviation (%RSD) for the mean relative response factors for the unlabeled native analytes calculated by isotope dilution must not exceed 20 percent. The percent RSD for the mean relative response factors for the unlabeled native analytes calculated by internal standard must not exceed 35 percent. The percent RSD for the mean relative response factors for the labeled standards must not exceed 35 percent.

10.3.5 Analyze 2µL of the Initial Calibration Verification (ICV) Standard in section 7.6.2 after completion of the ICAL prior to sample analysis. Calculate the concentration of the ICV using the RRFs from the CS-3 standard analyzed in section 10.3.4. Calculate the percent difference (%D) between the expected and the calculated ICV concentration using the following formula:

$$\%D = \frac{(C_{Exp} - C_{Calc})}{C_{Exp}} \times 100$$

Where:
C_{Exp} = The expected concentration of the ICV Standard.
C_{Calc} = The calculated concentration of the ICV Standard.

10.3.5.1 The criteria for acceptance of the ICV Standard are as follows:

10.3.5.2 The %D may not exceed ±35% for more than 4 of the native and labeled compounds.

- The %D may not exceed ±50% for any native or labeled compound.

10.3.5.3 All data associated with compounds with percent differences exceeding ±35% must be reviewed before acceptance and shall be documented as a NCM. In addition, all data associated with these compounds require corrective action that may include the following:

- Reanalyze the ICV Standard.
- Replace and reanalyze the ICV Standard.
- Evaluate the instrument performance.
- Evaluate the Initial Calibration Standards.

10.4 Continuing Calibration

10.4.1 Continuing calibration is performed at the beginning of a 12-hour period after successful mass resolution check.

10.4.2 Document the mass resolution performance as specified in section 10.3.2 at both the beginning and end of the 12-hour period.

10.4.3 Analyze 2 μL of the Continuing Calibration Verification Standard (VER/CS3). Calculate the concentration (C) of the compounds of interest (target analytes, internal standards, cleanup standards, and surrogate standards) vs. the appropriate quantitation reference (as specified in Table 2) using the following equation:

$$C = \frac{As \times Cis}{Ais \times RRF}$$

Where:

As = sum of the areas of the quantitation ions of the compound of interest.
Ais = sum of the areas of the quantitation ions of the appropriate reference standard.
Cis = concentration of the appropriate reference standard.
RRF = mean relative response factor from section 10.3.4.2.

10.4.4 Calculate the concentrations as percentages of the test concentrations and compare them to the limits specified in the appropriate Table 10A or 10B using the following equation:

$$C_{ver}\% = \frac{C_{ver}}{C_{test}} \times 100$$

Where:

C_{ver} = the concentration of the VER standard calculated in section 10.4.3
C_{test} = the test concentration of the VER standard listed in Table 6a.

10.4.5 Criteria for Acceptable Calibration - The criteria listed below for acceptable calibration must be met before sample analyses are performed. If the acceptance criteria are met, the calibration is deemed to be in control and the RRFs generated from the initial calibration are used to quantify samples. If acceptable calibration is not achieved, identify the root cause, perform corrective action, and repeat the continuing calibration. If a second consecutive attempt at a continuing calibration fails, two consecutive calibrations must meet the criteria, or an initial calibration must be run before proceeding with client samples.

10.4.5.1 The ion abundance ratios of the peaks representing the Toxics/LOCs and labeled standards must be within the control limits specified in Table 9.

10.4.5.2 The S/N for the GC signals present in every SICP (including those for labeled standards) must be ≥ 10.

10.4.5.3 For Toxic and LOC congeners, as listed in the first section of Table 10A or 10B, the percent of the calculated concentration relative to the test concentration must be within 70-130% for 1668A and 1668B and 75-125% for 1668C.

10.4.5.4 For non-Toxic congeners the calculated concentrations must be within 70-130% of the test concentrations for 1668A. 1668B and 1668C.

10.4.5.5 The absolute retention times (RT) of the labeled internal standards must be within ±15 seconds of the retention times obtained during initial calibration.

10.4.5.6 The relative retention times (RRT) of the Toxics/LOC congeners must be within their respective RRT limits generated in the retention time calibration in section 10.2.

10.4.5.6.1 If the RRTs or RTs are not within the limits above, the GC may not be performing properly. However, routine column maintenance may include removing short amounts of the beginning of the column when active sites or non-volatile compounds in sample extracts cause poor chromatography and loss of specificity. Shortening of the column can cause the RRTs or RTs to fall outside the above limits.

10.4.5.6.2 When the RRT of any compound or the RT of any internal standard is not within the above limits, corrective action must be taken. If the GC is not performing properly, correct the problem and repeat the test. If the GC is performing properly but the RRTs or RTs have changed due to routine column maintenance, adjust the GC or replace the GC column, then repeat the test or repeat the retention time calibration.

10.4.5.7 Evaluate and document the percent valley between PCBs 34 and 23. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.4.5.8 Evaluate and document the percent valley between PCBs 187 and 182. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.4.6 Daily calibration must be performed every 12 hours of instrument operation. The 12-hour shift begins with the documentation of the mass resolution followed by the injection of the Continuing Calibration Standard (VER).

11 Procedure

11.1 One time procedural variations are allowed only if deemed necessary in the professional judgement of supervision to accommodate variations in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variations in the procedure, except those specified by project specific instructions, shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist, Project Manager, and QA Manager. If contractually required, the client shall be notified.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2 Sample Extraction and Cleanup

The extraction and cleanup procedures are described in SOP KNOX-OP-0021, "Extraction of Polychlorinated Biphenyl (PCB) Isomers for Analysis by Isotope Dilution HRGC/HRMS", current revision.

11.3 Sample Analysis

11.3.1 Analyze the sample extracts under the same instrument operating conditions used to perform the instrument calibrations. Inject 2 µL into the GC/MS and acquire data beginning at 11 minutes and ending after decachlorobiphenyl has eluted from the column.

11.3.2 Record analysis information in the instrument logbook. The following information is required:

- Date of analysis
- Time of analysis

- Instrument data system filename
- Analyst
- Lab sample identification

Additional information may be recorded in the logbook if necessary.

11.3.3 Generate integrated ion chromatograms for the masses listed in Table 8 that encompass the expected retention windows of the PCB homologous series.

11.3.4 Generate a reduced peak list file from the integrations shown in the ion chromatograms.

11.3.5 Load the reduced peak list file into the calculation software.

11.3.6 The RTs of the unambiguous labeled congeners (RT Markers) are used to calculate a least squares best fit regression for retention times compared to those of the retention time calibration.

11.3.7 The resulting regression is used to calculate predicted retention times for target analytes. These predicted retention times are used by the software to identify candidate peaks for targets.

11.3.8 The analyst reviews the peaks identified as targets and determines whether to accept the identification. This determination is made by evaluating the delta values (RT shift from predicted), knowledge of peak patterns and observations of localized shifting.

11.3.9 A RRT window is calculated by multiplying the RRT Limit High and the RRT Limit Low by the retention time of the designated RT reference. The software applies a qualitative flag to each peak identified as a target that has a RRT outside the RRT window.

11.4 HRGC/HRMS Troubleshooting Guide

11.4.1 Perform the instrument's leak check: Evaluate the air spectrum. Mass 28 should be less than 5 times mass 69. If the air spectrum is not acceptable, replace transfer line ferrule and/or pump out PFK reservoir.

11.4.2 Check the voltage on the 5V power supply: If voltage is not reading 5V, adjust voltage to read 5V.

11.4.3 Check daily calibration standard: Evaluate the signal to noise (S/N), examine peak shape/chromatography, and evaluate response factors. Refer to specific SOP's requirements and acceptance criteria for all natives and internal standards. If daily calibration is not acceptable , perform the following troubleshooting options as needed and reanalyze a daily CS-3 calibration standard:

- Replace the inlet seal and clean the injection port body with methanol to improve response factor.

- Perform column/injector port maintenance and/or retune the instrument to resolve chromatography/resolution, signal to noise issues.

- Perform a gain test on the electron multiplier.
- Adjust the tuning parameters of the instrumentation to achieve optimum sensitivity and peak shape.
- Evaluate carryover from sample analyses. If carryover or contamination is suspected, run solvent rinses (nonane) under MID to evaluate the contamination.
- When troubleshooting cannot resolve an instrument problem, a manufacture's service engineer may be consulted for possible solution, or called onsite for diagnosis/repair.

11.5 Refer to the TestAmerica Knoxville Quality Assurance Manual, KX-QAM, current revision for the HRGC/HRMS instrument equipment maintenance table.

11.6 Refer to TestAmerica Knoxville SOP KNOX-IT-0001, current revision for requirements for computer hardware and software.

12 Data Analysis and Calculations

12.1 Qualitative Identification Criteria for PCBs

For a gas chromatographic peak to be identified as a PCB, it must meet all of the following criteria:

12.1.1 The signals for the two exact masses in Table 8 must be present and must maximize within ±2 seconds.

12.1.2 The signal to noise ratio (S/N) for each GC peak at each exact mass must be greater than or equal to (≥) 2.5. (This requirement does not apply to the secondary ion for dichlorinated biphenyls [mass 223.9974]). High background from PFK fragments at 223.9974 results in noise levels which exceed 10% of the signal height at levels that are reliably quantifiable.

12.1.3 The ratio of the integrated areas of the two exact masses specified in Table 8 must be within the limits in Table 9 . Alternately, the ratios may be within ±15% of the ratio in the midpoint (CS-3) calibration or calibration verification (VER), whichever is most recent.

12.1.4 The relative retention time of the peak for a CB must be within the RRT QC limits calculated in section 10.2.5.

NOTE: For native CBs determined by internal standard quantitation, a given CB congener may fall within more than one RT window and be misidentified unless the RRT windows are made very narrow, as in Table 2. Therefore, consistency of the RT and RRT with other congeners and the labeled compounds may be required for rigorous congener identification. Retention time regression analysis may be employed for this purpose.

12.1.5 If identification is ambiguous, (i.e., some, but not all of the identification criteria are met for a congener) an experienced analyst must determine the presence or absence of the congener.

12.2 Quantitation for PCBs

12.2.1 Calculate the Internal Standard Recoveries (Ris) relative to the Recovery Standard according to the following equation:

$$Ris = \frac{Ais \times Qrs}{Ars \times RRFis \times Qis} \times 100\%$$

Where:

Ais = sum of the areas of the quantitation ions of the appropriate internal standard
Ars = sum of the areas of the quantitation ions of the recovery standard
Qrs = ng of recovery standard added to extract
Qis = ng of internal standard added to sample
RRFis = mean relative response factor of internal standard obtained during initial calibration

NOTE: In some situations, such as source testing, the extract is split for multiple analyses. In this case, Qrs must be correctly calculated to account for the splitting of extracts before the recovery standard was added.

$$\mathrm{Qrs} = \mathrm{Qrss} \times \mathrm{Split}$$

Where:

Qrs = ng of recovery standard added to extract
Qrss= ng of recovery standard added to the split portion of the extract
Split= split ratio of the extract

12.2.2 Calculate the concentration of individual PCBs according to the following equation:

$$Concentration = \frac{As \times Qis}{Ais \times RRF \times W \times S}$$

Where:

As = sum of the areas of the quantitation ions of the compound of interest
Ais = sum of the areas of the quantitation ions of the appropriate internal standard
Qis = ng of internal standard added to sample
RRF = mean relative response factor of compound obtained during initial calibration
W = amount of sample extracted (grams or liters)
S = decimal expression of solids (optional, if results are requested to be reported on dry weight basis)

12.2.3 If reporting results for Total Homolog Groups, calculate the total concentration of all isomers within each homolog group by summing the concentrations of the individual PCB isomers within that homolog group.

12.2.4 If no peaks are present in the region of the ion chromatogram where the compounds of interest are expected to elute, calculate the estimated detection limit (EDL) for that compound according to the following equation:

$$EDL = \frac{N \times 2.5 \times Qis}{His \times RRF \times W \times S}$$

Where:

N = sum of peak to peak noise of quantitation ion signals in the region of the ion chromatogram where the compound of interest is expected to elute
His = sum of peak heights of quantitation ions for appropriate internal standard
Qis = ng of internal standard added to sample
RRF = mean relative response factor of compound obtained during initial calibration
W = amount of sample extracted (grams or liters)
S = decimal expression of solids (optional, if results are requested to be reported on dry weight basis.

Note: do not use **S** if results are to be reported by QuantIMS since it performs all necessary moisture corrections.)

12.2.5 If peaks are present in the region of the ion chromatogram which do not meet the qualitative criteria listed in section 12.1, calculate an Estimated Maximum Possible Concentration (EMPC). Use the equation in section 12.2.2, except that “As” should represent the sum of the area under each mass peak calculated using the theoretical chlorine isotope ratio. The peak selected to calculate the theoretical area should be the one which gives the lower of the two possible results (i.e., the EMPC will always be lower than the result calculated from the uncorrected areas).

12.2.6 If the concentration in the final extract of any PCB isomer exceeds the upper method calibration limits, a dilution of the extract or a re-extraction of a smaller portion of the sample must be performed. Dilutions of up to 1/10 may be performed on the extract. If compound concentrations exceeding the calibration range cannot be brought within the calibration range by a 1/10 dilution, extraction of a smaller aliquot of sample may be performed or the sample may be analyzed by a more appropriate analytical technique such as HRGC/LRMS. Consultation with the client should occur before any re-extraction is performed. The lab may report the measured concentration and indicate that the value exceeds the calibration limit by flagging the results with “E”. Consultation with the client should occur before compounds are reported which exceed the calibration range.

12.3 The estimated minimum level (EML) is defined as the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present assuming a sample is extracted at the recommended weight or volume and is carried through all normal extraction and analysis procedures. The EMLs for different matrices and extract volumes are listed in Table 4. Deviations from the extraction amounts or final volumes listed will result in corresponding changes in the actual sample EMLs.

12.4 Flag all compound results in the sample which are below the estimated minimum level with a “J” qualifier.

12.5 Flag all compound results in the sample which were detected in the method blank with a “B” qualifier.

12.6 Flag all compound results in the sample which are above the upper calibration limit with an “E” qualifier.

12.7 Flag all compound results in the sample which are “Estimated Maximum Possible Concentrations” with a “Q” qualifier.

12.8 Flag compound results in the sample that may contain co-eluting compounds with a “C” qualifier.

12.8.1 Flag congeners known to coelute with a higher numbered congener with a “C” qualifier.

12.8.2 Flag congeners that coelute with a lower numbered congener with a “Cx” qualifier where x is the CAS PCB number of the lowest numbered congener in the coeluting group.

12.9 If DOD QSM requirements are used, refer to SOP KNOX-QA-0021.

12.10 Flag compound results in the sample that may be affected by ion suppression with a “S” qualifier. When ion suppression of a PFK trace occurs at greater than or equal to 20% of full scale on both the lock mass and QC mass traces, and when the suppression is sustained for greater than 4 seconds, the suppression must be evaluated to determine which, if any, PCB congeners co-elute with the suppression. Samples may be diluted to decrease the effects of the ion suppression and rerun. Congeners that are determined to co-elute with the suppression are flagged with an “S” qualifier.

12.11 Data Review

12.11.1 Refer to Figure 3 for an example data review checklists used to perform and document the review of the data. Using the data review checklist, the analyst also creates a narrative which includes any qualifications of the sample data.

12.11.2 The analyst who performs the initial data calculations must initial and date the front chromatogram of the raw data package to document that they have performed the qualitative and quantitative analysis on the sample data.

12.11.3 A second analyst must verify all qualitative peak identifications. If discrepancies are found, the data must be returned to the analyst who performed the initial peak identification for resolution.

12.11.4 A second analyst must check all hand calculation and data entry into calculation programs, databases, or spreadsheets at a frequency of 100 percent. If discrepancies are found, the data must be returned to the analyst who performed the initial calculation for resolution.

12.11.5 The reviewing analyst must initial and date the front chromatogram of the raw data package to document that they have performed the second level review on the sample data.

12.11.6 All items listed on the data review checklist must be checked by both the analyst who performed the initial qualitative and quantitative analysis and the analyst who performed the second level review. An example data review checklist is shown in Figure 3.

13 Method Performance

13.1 Method Detection Limit (MDL) - An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. The procedure for determination of the method detection limit is given in the SOP CA-Q-S006, current revision, based on 40 CFR Part 136 Appendix B. The result of the MDL determination must support the reporting limit (ML).

13.2 Initial Demonstration of Capability – Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in TestAmerica Knoxville SOP KNOX-QA-0009.

13.3 Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009 current revision for further requirements for performing and documenting initial and on-going demonstrations of capability**.**

14 Pollution Prevention

14.1 All attempts will be made to minimize the use of solvents and standard materials.

15 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 Waste Streams Produced by the Procedure: The following waste streams are produced when this method is carried out.

- Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials shall be placed in the incinerable laboratory waste stream, contained in a steel or poly satellite accumulation container.

16 References

16.1 Knoxville Laboratory Quality Assurance Manual (QAM), current revision.

16.2 Method 1668, Revision A, B and C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-821-R-00-002, December 1999.

16.3 Method 1613: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS [Revision B], EPA#: 821/B-94-005a YEAR: 1994

16.4 Ballschmiter, K. and M. Zell, "Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography", *Fresenius Z. Anal. Chem*., 302:20-31 (1980).

16.5 Schulte, E. and R. Malisch, "Berechnung der Wahren PCB-Gehalte in Umweltproben I. Ermittlung der Zusammensetzung Zweier Technischer PCB-Gemische," *Fresenius Z. Anal. Chem*., 314:545-551 (1983).

16.6 Guitart, R., P. Puig and J. Gómez-Catalán, "Requirement for a Standardized Nomenclature Criterion for PCBs: Computer-Assisted Assignment of Correct Congener Denomination and Numbering," *Chemosphere*, 27(8):1451-1459 (1993).

16.7 Rigaudy, J. and Klesney, S.P., Nomenclature of Organic Chemistry, Pergamon, 1979.

16.8 Pretsch, Clerc, Seibl, Simon, Tables of Spectral Data for Structure Determination of Organic Compounds, Second Edition, Springer-Verlag, 1989.

16.9 CRC Handbook of Chemistry and Physics, 71st edition, CRC Press, 1990-1991.

16.10 Email dated 03/02/10 from Richard Reding Ph.D., Chief U.S. EPA, Engineering & Analytical Support Branch. Engineering & Analysis Division Office of Science and Technology, Office of Water – Subject: Use of QC Acceptance Limits."

17 Miscellaneous

17.1 Deviations from EPA Method 1668, Revisions A, B and C.

17.1.1 Additional recovery standards are used in this procedure. The additional standards are listed in Table 1.

17.1.2 Additional labeled standards are used in this procedure as field sampling surrogates. The additional standards are listed in Table 1

17.1.3 This procedure uses internal standards which are within the same MID group as the native congener that is being calculated. This improvement is used to reduce the effects of full verses reduced accelerating voltage tuning differences which can be exaggerated when crossing MID groups.

17.1.4 The calibration procedure in the method 1668A, revision B, and C call for a single point standard for the non-Toxic/LOC congeners. This procedure uses a multi-point calibration for all 209 congeners.

17.1.5 This procedure uses MID groups that differ from the method. The procedure uses 4 groups, rather than 6, to improve instrument stability, by holding the magnet current steady for longer periods. Therefore alternate PFK lock masses are monitored, to reflect the mass ranges of the procedure's MID groups.

17.1.6 This procedure uses average retention times (and average relative retention times) produced by triplicate analyses of the 5 mixes specified, rather than single analyses of the diluted 209 standard.

17.1.7 The absolute retention times, relative retention times, and relative retention time limits used by the laboratory differ from those listed in all versions of Method 1668. Each SPB-Octyl column lot used by the laboratory has exhibited slightly different retention time characteristics resulting in different absolute retention times than those listed in the method or those observed with another SPB-Octyl column lot. To ensure that the correct peak assignments are made, a retention time study is performed for each new column lot to verify the original study. This study includes the analysis of the five retention time mixes listed in Method 1668A as described in Section 10.2.5 to 10.2.7. If the retention time study is found to be different than the original elution pattern, a new ICAL will be performed. This procedure requires a minimum elution time of 55 minutes for PCB 209.

17.1.8 The calibration verification procedures in the method call for updating the retention times, relative retention times and response factors for non-Toxic compounds during daily calibration and use the retention times, relative retention times and response factors from the initial calibration for Toxic and LOC compounds. This laboratory uses the retention times and relative retention times from triplicate analyses of the 5-mix series, which contains all congeners, and uses response factors from the initial calibration for all 209 compounds. The practice of updating the relative retention times of only a subset of compounds causes significant error in the linear regression prediction formulas used by targeting software to identify the compounds. This procedure has provisions for updating all RTs and RRTs by analyzing a new retention time calibration series.

17.1.9 The EMLs listed in Table 4 differ from those listed in the reference method. The EMLs are set above the mean plus 2 standard deviations for the higher of detections or EDLs for method blanks. In no case is the EML lower than the low calibration limit. The survey period was approximately 14 months.

17.1.10 This procedure uses a 100uL final volume for solid samples, or a variation depending on the protocol assigned to the sample based on screen results prior to extraction.

17.1.11 Method 1668C requires recovery standards be added immediately prior to analysis. In this procedure recovery standards are added to sample extracts at the conclusion of the extraction / clean up process prior to analysis.

17.1.12 Method 1668C implies that all qualitative criteria must be met in order for a positive hit to be reported. This procedure allows positive hits to be reported with “Q” qualifiers.

17.1.13 This procedure uses calculated concentrations for determining if a dilution is required not area counts as suggested by Method 1668C, section 17.5.

17.1.14 This procedure reports positive hits below the estimated minimum level (EML) which are determined to be greater than 2.5 signal to noise. These hits are qualified with a “J”.

17.1.15 This procedure does not report blank corrected values.

17.1.16 When samples are diluted to bring a native analyte within the upper calibration range all 209 congeners are reported from this analysis. This procedure reports all congeners from one analytical analysis.

17.1.17 This procedure uses a daily calibration CCV consisting of all 209 congeners at the beginning of each 12 hour analytical shift.

17.1.18 Extracts are stored in a dark box at room temperature in the same manner as standards.

17.1.19 1668C EML limits are listed in Table 4. However, this laboratory currently uses the 1668A/B limits

17.1.20 The limits established in 1668B, Table 6 for the IPR and OPR performance evaluations have been observed by EPA reviewers to penalize an analyst who achieves better performance than those observed in the inter-laboratory study. Based on EPA's recommendations (section 16.10), TestAmerica Knoxville performs analysis by Method 1668B with the QC acceptance criteria in Table 6 of EPA Method 1668A. Table 10 "Acceptance Criteria for Performance Tests" of this SOP will be used for the IPR and OPR limits.

17.2 List of tables and figures referenced in the body of the SOP.

17.2.1 Table 1 – Polychlorinated Biphenyls Determined by High Resolution Gas Chromatography (HRGC)/High Resolution Mass Spectrometry (HRMS)

17.2.2 Table 2 – RT References, Quantitation References, Retention Times (RT), and Relative Retention Times (RRTs) for the 209 CB congeners on SPB-Octyl

17.2.3 Table 3 – Low Calibration Levels Based on Various Final Extract Volumes

17.2.4 Table 4 – Estimated Minimum Levels EPA 1668A, B & C– Matrix and Concentration

17.2.5 Table 5a – Concentration of Native PCB Congener Stock and Spiking Solutions

17.2.6 Table 5b – Concentration of $^{13}C_{12}$ Labeled PCB Congener Stock and Spiking Solutions

17.2.7 Table 6a – Concentration of PCBs in Calibration Solutions

17.2.8 Table 6b – Preparation of Calibration Solutions

17.2.9 Table 7 – Window Defining Mixture and SPB-Octyl Resolution Test Compounds

17.2.10 Table 8 – Ions Monitored for HRGC/HRMS Analysis of PCBs

17.2.11 Table 9 – Theoretical Ion Abundance Ratios and Control Limits for PCBs

17.2.12 Table 10A – Acceptance Criteria for Performance Tests (1668A & B)

17.2.13 Table 10B – Acceptance Criteria for Performance Tests (1668C)

17.2.14 Table 11 – Retention Times of Isomers on SPB-Octyl Column for PCB Standard Mixes

17.2.15 Table 12 – Assignment of Sample Preparation Protocols

17.2.16 Figure 1 – Recommended GC Operating Conditions

17.2.17 Figure 2 – Recommended MID Descriptors

17.2.18 Figure 3 – Example Data Review Checklist

17.2.19 Table 1 - Polychlorinated Biphenyls Determined by HRGC/HRMS

PCB Number[1]	BZ/IUPAC Number[2].	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	Usage
1	1	2-monochlorobiphenyl	2051-60-7	$^{13}C_{12}$-2-monochlorobiphenyl	234432-85-0	Internal Std
2	2	3-monochlorobiphenyl	2051-61-8			
3	3	4-monochlorobiphenyl	2051-62-9	$^{13}C_{12}$-4-monochlorobiphenyl	208263-77-8	Internal Std
4	4	2,2’-dichlorobiphenyl	13029-08-8	$^{13}C_{12}$-2,2’-dichlorobiphenyl	234432-86-1	Internal Std
5	5	2,3-dichlorobiphenyl	16605-91-7			
6	6	2,3’-dichlorobiphenyl	25569-80-6			
7	7	2,4-dichlorobiphenyl	33284-50-3			
8	8	2,4’-dichlorobiphenyl	34883-43-7	$^{13}C_{12}$-2,4’-dichlorobiphenyl		Surrogate Std
9	9	2,5-dichlorobiphenyl	34883-39-1	$^{13}C_{12}$-2,5-dichlorobiphenyl	250694-89-4	Recovery Std
10	10	2,6-dichlorobiphenyl	33146-45-1			
11	11	3,3’-dichlorobiphenyl	2050-67-1			
12	12	3,4-dichlorobiphenyl	2974-92-7			
13	13	3,4’-dichlorobiphenyl	2974-90-5			
14	14	3,5-dichlorobiphenyl	34883-41-5			
15	15	4,4’-dichlorobiphenyl	2050-68-2	$^{13}C_{12}$-4,4’-dichlorobiphenyl	208263-67-6	Internal Std
16	16	2,2’,3-trichlorobiphenyl	38444-78-9			
17	17	2,2’,4-trichlorobiphenyl	37680-66-3			
18	18	2,2’,5-trichlorobiphenyl	37680-65-2			
19	19	2,2’,6-trichlorobiphenyl	38444-73-4	$^{13}C_{12}$-2,2’,6-trichlorobiphenyl	234432-87-2	Internal Std
20	20	2,3,3’-trichlorobiphenyl	38444-84-7			
21	21	2,3,4-trichlorobiphenyl	55702-46-0			
22	22	2,3,4’-trichlorobiphenyl	38444-85-8			
23	23	2,3,5-trichlorobiphenyl	55720-44-0			
24	24	2,3,6-trichlorobiphenyl	55702-45-9			
25	25	2,3’,4-trichlorobiphenyl	55712-37-3			
26	26	2,3’,5-trichlorobiphenyl	38444-81-4			
27	27	2,3’,6-trichlorobiphenyl	38444-76-7			
28	28	2,4,4’-trichlorobiphenyl	7012-37-5	$^{13}C_{12}$-2,4,4’-trichlorobiphenyl	208263-76-7	Cleanup Std
29	29	2,4,5-trichlorobiphenyl	15862-07-4			
30	30	2,4,6-trichlorobiphenyl	35693-92-6			
31	31	2,4’,5-trichlorobiphenyl	16606-02-3	$^{13}C_{12}$-2,4’,5-trichlorobiphenyl		Recovery Std
32	32	2,4’,6-trichlorobiphenyl	38444-77-8	$^{13}C_{12}$-2,4’,6-trichlorobiphenyl		Recovery Std
33	33	2’,3,4-trichlorobiphenyl (2,3’,4’-trichlorobiphenyl)	38444-86-9			
34	34	2’,3,5-trichlorobiphenyl (2,3’,5’-trichlorobiphenyl)	37680-68-5			
35	35	3,3’,4-trichlorobiphenyl	37680-69-6			
36	36	3,3’,5-trichlorobiphenyl	38444-87-0			
37	37	3,4,4’-trichlorobiphenyl	38444-90-5	$^{13}C_{12}$-3,4,4’-trichlorobiphenyl	208263-79-0	Internal Std
38	38	3,4,5-trichlorobiphenyl	53555-66-1			
39	39	3,4’,5-trichlorobiphenyl	38444-88-1			
40	40	2,2’,3,3’-tetrachlorobiphenyl	38444-93-8			
41	41	2,2’,3,4-tetrachlorobiphenyl	52663-59-9			
42	42	2,2’,3,4’-tetrachlorobiphenyl	36559-22-5			
43	43	2,2’,3,5-tetrachlorobiphenyl	70362-46-8			
44	44	2,2’,3,5’-tetrachlorobiphenyl	41464-39-5			
45	45	2,2’,3,6-tetrachlorobiphenyl	70362-45-7			
46	46	2,2’,3,6’-tetrachlorobiphenyl	41464-47-5			
47	47	2,2’,4,4’-tetrachlorobiphenyl	2437-79-8			
48	48	2,2’,4,5-tetrachlorobiphenyl	70362-47-9			
49	49	2,2’,4,5’-tetrachlorobiphenyl	41464-40-8			
50	50	2,2’,4,6-tetrachlorobiphenyl	62796-65-0			
51	51	2,2’,4,6’-tetrachlorobiphenyl	68194-04-7			
52	52	2,2’,5,5’-tetrachlorobiphenyl	35693-99-3	$^{13}C_{12}$-2,2’,5,5’-tetrachlorobiphenyl	160901-66-6	Recovery Std
53	53	2,2’,5,6’-tetrachlorobiphenyl	41464-41-9			
54	54	2,2’,6,6’-tetrachlorobiphenyl	15968-05-5	$^{13}C_{12}$-2,2’,6,6’-tetrachlorobiphenyl	234432-88-3	Internal Std
55	55	2,3,3’,4-tetrachlorobiphenyl	74338-24-2			
56	56	2,3,3’,4’-tetrachlorobiphenyl	41464-43-1			
57	57	2,3,3’,5-tetrachlorobiphenyl	70424-67-8			
58	58	2,3,3’,5’-tetrachlorobiphenyl	41464-49-7			

PCB Number[1]	BZ/IUPAC Number[2].	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	Usage
59	59	2,3,3’,6-tetrachlorobiphenyl	74472-33-6			
60	60	2,3,4,4’-tetrachlorobiphenyl	33025-41-1			
61	61	2,3,4,5-tetrachlorobiphenyl	33284-53-6			
62	62	2,3,4,6-tetrachlorobiphenyl	54230-22-7			
63	63	2,3,4’,5-tetrachlorobiphenyl	74472-34-7			
64	64	2,3,4’,6-tetrachlorobiphenyl	52663-58-8			
65	65	2,3,5,6-tetrachlorobiphenyl	33284-54-7			
66	66	2,3’,4,4’-tetrachlorobiphenyl	32598-10-0			
67	67	2,3’,4,5-tetrachlorobiphenyl	73575-53-8			
68	68	2,3’,4,5’-tetrachlorobiphenyl	73575-52-7			
69	69	2,3’,4,6-tetrachlorobiphenyl	60233-24-1			
70	70	2,3’,4’,5-tetrachlorobiphenyl	32598-11-1			
71	71	2,3’,4’,6-tetrachlorobiphenyl	41464-46-4			
72	72	2,3’,5,5’-tetrachlorobiphenyl	41464-42-0			
73	73	2,3’,5’,6-tetrachlorobiphenyl	74338-23-1			
74	74	2,4,4’,5-tetrachlorobiphenyl	32690-93-0			
75	75	2,4,4’,6-tetrachlorobiphenyl	32598-12-2			
76	76	2’,3,4,5-tetrachlorobiphenyl (2,3’,4’,5’-tetrachlorobiphenyl)	70362-48-0			
77	77	3,3’,4,4’-tetrachlorobiphenyl	32598-13-3	$^{13}C_{12}$-3,3’,4,4’-tetrachlorobiphenyl	160901-67-7	Internal Std
78	78	3,3’,4,5-tetrachlorobiphenyl	70362-49-1			
79	79	3,3’,4,5’-tetrachlorobiphenyl	41464-48-6	$^{13}C_{12}$-3,3’,4,5’-tetrachlorobiphenyl		Surrogate Std
80	80	3,3’,5,5’-tetrachlorobiphenyl	33284-52-5			
81	81	3,4,4’,5-tetrachlorobiphenyl	70362-50-4	$^{13}C_{12}$-3,4,4’,5-tetrachlorobiphenyl	160901-68-8	Internal Std
82	82	2,2’,3,3’,4-pentachlorobiphenyl	52663-62-4			
83	83	2,2’,3,3’,5-pentachlorobiphenyl	60145-20-2			
84	84	2,2’,3,3’,6-pentachlorobiphenyl	52663-60-2			
85	85	2,2’,3,4,4’-pentachlorobiphenyl	65510-45-4			
86	86	2,2’,3,4,5-pentachlorobiphenyl	55312-69-1			
87	87	2,2’,3,4,5’-pentachlorobiphenyl	38380-02-8			
88	88	2,2’,3,4,6-pentachlorobiphenyl	55215-17-3			
89	89	2,2’,3,4,6’-pentachlorobiphenyl	73575-57-2			
90	90	2,2’,3,4’,5-pentachlorobiphenyl	68194-07-0			
91	91	2,2’,3,4’,6-pentachlorobiphenyl	68194-05-8			
92	92	2,2’,3,5,5’-pentachlorobiphenyl	52663-61-3			
93	93	2,2’,3,5,6-pentachlorobiphenyl	73575-56-1			
94	94	2,2’,3,5,6’-pentachlorobiphenyl	73575-55-0			
95	95	2,2’,3,5’,6-pentachlorobiphenyl	38379-99-6	$^{13}C_{12}$-2,2’,3,5’,6-pentachlorobiphenyl		Surrogate Std
96	96	2,2’,3,6,6’-pentachlorobiphenyl	73575-54-9			
97	97	2,2’,3’,4,5-pentachlorobiphenyl (2,2’,3,4’,5’-pentachlorobiphenyl)	41464-51-1			
98	98	2,2’,3’,4,6-pentachlorobiphenyl (2,2’,3,4’,6’-pentachlorobiphenyl)	60233-25-2			
99	99	2,2’,4,4’,5-pentachlorobiphenyl	38380-01-7			
100	100	2,2’,4,4’,6-pentachlorobiphenyl	39485-83-1			
101	101	2,2’,4,5,5’-pentachlorobiphenyl	37680-73-2	$^{13}C_{12}$-2,2’,4,5,5’-pentachlorobiphenyl	160901-69-9	Recovery Std
102	102	2,2’,4,5,6’-pentachlorobiphenyl	68194-06-9			
103	103	2,2’,4,5’,6-pentachlorobiphenyl	60145-21-3			
104	104	2,2’,4,6,6’-pentachlorobiphenyl	56558-16-8	$^{13}C_{12}$-2,2’,4,6,6’-pentachlorobiphenyl	234432-89-4	Internal Std
105	105	2,3,3’,4,4’-pentachlorobiphenyl	32598-14-4	$^{13}C_{12}$-2,3,3’,4,4’-pentachlorobiphenyl	160901-70-2	Internal Std
106	106	2,3,3’,4,5-pentachlorobiphenyl	70424-69-0			
107	107/109	2,3,3',4',5-pentachlorobiphenyl	70424-68-9			
108	108/107	2,3,3',4,5'-pentachlorobiphenyl	70362-41-3			
109	109/108	2,3,3',4,6-pentachlorobiphenyl	74472-35-8			
110	110	2,3,3’,4’,6-pentachlorobiphenyl	38380-03-9			
111	111	2,3,3’,5,5’-pentachlorobiphenyl	39635-32-0	$^{13}C_{12}$-2,3,3’,5,5’-pentachlorobiphenyl	160901-71-3	Cleanup Std
112	112	2,3,3’,5,6-pentachlorobiphenyl	74472-36-9			
113	113	2,3,3’,5’,6-pentachlorobiphenyl	68194-10-5			
114	114	2,3,4,4’,5-pentachlorobiphenyl	74472-37-0	$^{13}C_{12}$-2,3,4,4’,5-pentachlorobiphenyl	160901-72-4	Internal Std
115	115	2,3,4,4’,6-pentachlorobiphenyl	74472-38-1			
116	116	2,3,4,5,6-pentachlorobiphenyl	18259-05-7			
117	117	2,3,4’,5,6-pentachlorobiphenyl	68194-11-6			

PCB Number[1]	BZ/IUPAC Number[2].	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	Usage
118	118	2,3’,4,4’,5-pentachlorobiphenyl	31508-00-6	$^{13}C_{12}$-2,3’,4,4’,5-pentachlorobiphenyl	160901-73-5	Internal Std
119	119	2,3’,4,4’,6-pentachlorobiphenyl	56558-17-9			
120	120	2,3’,4,5,5’-pentachlorobiphenyl	68194-12-7			
121	121	2,3’,4,5’,6-pentachlorobiphenyl	56558-18-0			
122	122	2’,3,3’,4,5-pentachlorobiphenyl (2,3,3’,4’,5’-pentachlorobiphenyl)	76842-07-4			
123	123	2’,3,4,4’,5-pentachlorobiphenyl (2,3’,4,4’,5’-pentachlorobiphenyl)	65510-44-3	$^{13}C_{12}$-2’,3,4,4’,5-pentachlorobiphenyl	160901-74-6	Internal Std
124	124	2’,3,4,5,5’-pentachlorobiphenyl (2,3’,4’,5’,5-pentachlorobiphenyl)	70424-70-3			
125	125	2’,3,4,5,6’-pentachlorobiphenyl (2,3’,4’,5’,6-pentachlorobiphenyl)	74472-39-2			
126	126	3,3’,4,4’,5-pentachlorobiphenyl	57465-28-8	$^{13}C_{12}$-3,3’,4,4’,5-pentachlorobiphenyl	160901-75-7	Internal Std
127	127	3,3’,4,5,5’-pentachlorobiphenyl	39635-33-1	$^{13}C_{12}$-3,3’,4,5,5’-pentachlorobiphenyl		Recovery Std
128	128	2,2’,3,3’,4,4’-hexachlorobiphenyl	38380-07-3			
129	129	2,2’,3,3’,4,5-hexachlorobiphenyl	55215-18-4			
130	130	2,2’,3,3’,4,5’-hexachlorobiphenyl	52663-66-8			
131	131	2,2’,3,3’,4,6-hexachlorobiphenyl	61798-70-7			
132	132	2,2’,3,3’,4,6’-hexachlorobiphenyl	38380-05-1			
133	133	2,2’,3,3’,5,5’-hexachlorobiphenyl	35694-04-3			
134	134	2,2’,3,3’,5,6-hexachlorobiphenyl	52704-70-8			
135	135	2,2’,3,3’,5,6’-hexachlorobiphenyl	52744-13-5			
136	136	2,2’,3,3’,6,6’-hexachlorobiphenyl	38411-22-2			
137	137	2,2’,3,4,4’,5-hexachlorobiphenyl	35694-06-5			
138	138	2,2’,3,4,4’,5’-hexachlorobiphenyl	35065-28-2	$^{13}C_{12}$-2,2’,3,4,4’,5’-hexachlorobiphenyl	160901-76-8	Recovery Std
139	139	2,2’,3,4,4’,6-hexachlorobiphenyl	56030-56-9			
140	140	2,2’,3,4,4’,6’-hexachlorobiphenyl	59291-64-4			
141	141	2,2’,3,4,5,5’-hexachlorobiphenyl	52712-04-6			
142	142	2,2’,3,4,5,6-hexachlorobiphenyl	41411-61-4			
143	143	2,2’,3,4,5,6’-hexachlorobiphenyl	68194-15-0			
144	144	2,2’,3,4,5’,6-hexachlorobiphenyl	68194-14-9			
145	145	2,2’,3,4,6,6’-hexachlorobiphenyl	74472-40-5			
146	146	2,2’,3,4’,5,5’-hexachlorobiphenyl	51908-16-8			
147	147	2,2’,3,4’,5,6-hexachlorobiphenyl	68194-13-8			
148	148	2,2’,3,4’,5,6’-hexachlorobiphenyl	74472-41-6			
149	149	2,2’,3,4’,5’,6-hexachlorobiphenyl	38380-04-0			
150	150	2,2’,3,4’,6,6’-hexachlorobiphenyl	68194-08-1			
151	151	2,2’,3,5,5’,6-hexachlorobiphenyl	52663-63-5			
152	152	2,2’,3,5,6,6’-hexachlorobiphenyl	68194-09-2			
153	153	2,2’,4,4’,5,5’-hexachlorobiphenyl	35065-27-1	$^{13}C_{12}$-2,2’,4,4’,5,5’-hexachlorobiphenyl		Surrogate Std
154	154	2,2’,4,4’,5,6’-hexachlorobiphenyl	60145-22-4			
155	155	2,2’,4,4’,6,6’-hexachlorobiphenyl	33979-03-2	$^{13}C_{12}$-2,2’,4,4’,6,6’-hexachlorobiphenyl	234432-90-7	Internal Std
156	156	2,3,3’,4,4’,5-hexachlorobiphenyl	38380-08-4	$^{13}C_{12}$-2,3,3’,4,4’,5-hexachlorobiphenyl	160901-77-9	Internal Std
157	157	2,3,3’,4,4’,5’-hexachlorobiphenyl	69782-90-7	$^{13}C_{12}$-2,3,3’,4,4’,5’-hexachlorobiphenyl	160901-78-0	Internal Std
158	158	2,3,3’,4,4’,6-hexachlorobiphenyl	74472-42-7			
159	159	2,3,3’,4,5,5’-hexachlorobiphenyl	39635-35-3			
160	160	2,3,3’,4,5,6-hexachlorobiphenyl	41411-62-5			
161	161	2,3,3’,4,5’,6-hexachlorobiphenyl	74472-43-8			
162	162	2,3,3’,4’,5,5’-hexachlorobiphenyl	39635-34-2			
163	163	2,3,3’,4’,5,6-hexachlorobiphenyl	74472-44-9			
164	164	2,3,3’,4’,5’,6-hexachlorobiphenyl	74472-45-0			
165	165	2,3,3’,5,5’,6-hexachlorobiphenyl	74472-46-1			
166	166	2,3,4,4’,5,6-hexachlorobiphenyl	41411-63-6			
167	167	2,3’,4,4’,5,5’-hexachlorobiphenyl	52663-72-6	$^{13}C_{12}$-2,3’,4,4’,5,5’-hexachlorobiphenyl	161627-18-5	Internal Std
168	168	2,3’,4,4’,5’,6-hexachlorobiphenyl	59291-65-5			
169	169	3,3’,4,4’,5,5’-hexachlorobiphenyl	32774-16-6	$^{13}C_{12}$-3,3’,4,4’,5,5’-hexachlorobiphenyl	160901-79-1	Internal Std
170	170	2,2’,3,3’,4,4’,5-heptachlorobiphenyl	35065-30-6	$^{13}C_{12}$-2,2’,3,3’,4,4’,5-heptachlorobiphenyl	160901-80-4	Internal Std
171	171	2,2’,3,3’,4,4’,6-heptachlorobiphenyl	52663-71-5			
172	172	2,2’,3,3’,4,5,5’-heptachlorobiphenyl	52663-74-8			
173	173	2,2’,3,3’,4,5,6-heptachlorobiphenyl	68194-16-1			
174	174	2,2’,3,3’,4,5,6’-heptachlorobiphenyl	38411-25-5			
175	175	2,2’,3,3’,4,5’,6-heptachlorobiphenyl	40186-70-7			

PCB Number[1]	BZ/IUPAC Number[2].	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	Usage
176	176	2,2’,3,3’,4,6,6’-heptachlorobiphenyl	52663-65-7			
177	177	2,2’,3,3’,4’,5,6-heptachlorobiphenyl (2,2’,3,3’,4,5’,6’-heptachlorobiphenyl)	52663-70-4			
178	178	2,2’,3,3’,5,5’,6-heptachlorobiphenyl	52663-67-9	$^{13}C_{12}$-2,2’,3,3’,5,5’,6-heptachlorobiphenyl	160901-81-5	Cleanup Std
179	179	2,2’,3,3’,5,6,6’-heptachlorobiphenyl	52663-64-6			
180	180	2,2’,3,4,4’,5,5’-heptachlorobiphenyl	35065-29-3	$^{13}C_{12}$-2,2’,3,4,4’,5,5’-heptachlorobiphenyl	160901-82-6	Recovery Std
181	181	2,2’,3,4,4’,5,6-heptachlorobiphenyl	74472-47-2			
182	182	2,2’,3,4,4’,5,6’-heptachlorobiphenyl	60145-23-5			
183	183	2,2’,3,4,4’,5’,6-heptachlorobiphenyl	52663-69-1			
184	184	2,2’,3,4,4’,6,6’-heptachlorobiphenyl	74472-48-3			
185	185	2,2’,3,4,5,5’,6-heptachlorobiphenyl	52712-05-7			
186	186	2,2’,3,4,5,6,6’-heptachlorobiphenyl	74472-49-4			
187	187	2,2’,3,4’,5,5’,6-heptachlorobiphenyl	52663-68-0			
188	188	2,2’,3,4’,5,6,6’-heptachlorobiphenyl	74487-85-7	$^{13}C_{12}$-2,2’,3,4’,5,6,6’-heptachlorobiphenyl	234432-91-8	Internal Std
189	189	2,3,3’,4,4’,5,5’-heptachlorobiphenyl	39635-31-9	$^{13}C_{12}$-2,3,3’,4,4’,5,5’-heptachlorobiphenyl	160901-83-7	Internal Std
190	190	2,3,3’,4,4’,5,6-heptachlorobiphenyl	41411-64-7			
191	191	2,3,3’,4,4’,5’,6-heptachlorobiphenyl	74472-50-7			
192	192	2,3,3’,4,5,5’,6-heptachlorobiphenyl	74472-51-8			
193	193	2,3,3’,4’,5,5’,6-heptachlorobiphenyl	69782-91-8			
194	194	2,2’,3,3’,4,4’,5,5’-octachlorobiphenyl	35694-08-7	$^{13}C_{12}$-2,2’,3,3’,4,4’,5,5’-octachlorobiphenyl	208263-74-5	Recovery Std
195	195	2,2’,3,3’,4,4’,5,6-octachlorobiphenyl	52663-78-2			
196	196	2,2’,3,3’,4,4’,5,6’-octachlorobiphenyl	42740-50-1			
197	197	2,2’,3,3’,4,4’,6,6’-octachlorobiphenyl	33091-17-7			
198	198	2,2’,3,3’,4,5,5’,6-octachlorobiphenyl	68194-17-2			
199	201/199	2,2’,3,3’,4,5,5’,6’-octachlorobiphenyl	52663-75-9			
200	199/200	2,2’,3,3’,4,5,6,6’-octachlorobiphenyl	52663-73-7			
201	200/201	2,2’,3,3’,4,5’,6,6’-octachlorobiphenyl	40186-71-8			
202	202	2,2’,3,3’,5,5’,6,6’-octachlorobiphenyl	2136-99-4	$^{13}C_{12}$-2,2’,3,3’,5,5’,6,6’-octachlorobiphenyl	105600-26-8	Internal Std
203	203	2,2’,3,4,4’,5,5’,6-octachlorobiphenyl	52663-76-0			
204	204	2,2’,3,4,4’,5,6,6’-octachlorobiphenyl	74472-52-9			
205	205	2,3,3’,4,4’,5,5’,6-octachlorobiphenyl	74472-53-0	$^{13}C_{12}$-2,3,3’,4,4’,5,5’,6-octachlorobiphenyl	234446-64-1	Internal Std
206	206	2,2’,3,3’,4,4’,5,5’,6-nonachlorobiphenyl	40186-72-9	$^{13}C_{12}$-2,2’,3,3’,4,4’,5,5’,6-nonachlorobiphenyl	208263-75-6	Internal Std
207	207	2,2’,3,3’,4,4’,5,6,6’-nonachlorobiphenyl	52663-79-3			
208	208	2,2’,3,3’,4,5,5’,6,6’-nonachlorobiphenyl	52663-77-1	$^{13}C_{12}$-2,2’,3,3’,4,5,5’,6,6’-nonachlorobiphenyl	234432-92-9	Internal Std
209	209	2,2’,3,3’,4,4’,5,5’,6,6’-decachlorobiphenyl	2051-24-3	$^{13}C_{12}$-decachlorobiphenyl	160901-84-8	Internal Std

1. The PCB congener number is from Method 1668C and Chemical Abstract Services.
2. The BZ number is from Ballschmiter and Zell (1980). The IUPAC number, when different from the BZ, follows the recommended changes to the BZ number per Schulte and Malisch (1983) and Guitart et al. (1993).
3. The chemical structure names are from Ballschmiter and Zell (1980). IUPAC nomenclature structure names are listed in parenthesis when different from the BZ name (source CAS Registry).
4. Chemical Abstract Service Registry number (source CAS Registry and 1668A Table 1).

Table 2 - RT References, Quantitation References, Retention Times (RTs), and Relative Retention Times (RRTs) for the 209 CB congeners on SPB-Octyl

Cl No[1]	**IUPAC No[2,3]**	**RT Ref[4]**	**Quantitation Reference[5]**	**RT Win[6] (sec)**
1	1L	9L	9L	30
1	3L	9L	9L	30
1	1	1L	1L	10
1	2	3L	1L/3L	6
1	3	3L	3L	6
2	4L	9L	9L	30
2	9L	9L	9L	25
2	8L	4L	4L/15L	6
2	15L	9L	9L	20
2	4	4L	4L	10
2	10	4L	4L/15L	6
2	9	4L	4L/15L	6
2	7	4L	4L/15L	6
2	6	4L	4L/15L	6
2	5	4L	4L/15L	6
2	8	4L	4L/15L	6
2	14	15L	4L/15L	6
2	11	15L	4L/15L	6
2	13	15L	4L/15L	6
2	12	15L	4L/15L	6
2	15	15L	15L	10
3	19L	32L	32L	30
3	32L	32L	32L	6
3	31L	31L	31L	6
3	28L	31L	31L	20
3	37L	31L	31L	30
3	19	19L	19L	10
3	30	19L	19L	6
3	18	19L	19L	6
3	17	19L	19L	6
3	27	19L	19L	6
3	24	19L	19L	6
3	16	19L	19L	6
3	32	19L	19L	6
3	34	19L	37L	6
3	23	19L	37L	6
3	29	19L	37L	10
3	26	19L	37L	10
3	25	37L	37L	6
3	31	37L	37L	6
3	28	37L	37L	10
3	20	37L	37L	10
3	21	37L	37L	10
3	33	37L	37L	10
3	22	37L	37L	6
3	36	37L	37L	6
3	39	37L	37L	6
3	38	37L	37L	6
3	35	37L	37L	6
3	37	37L	37L	6
4	54L	52L	32L	20
4	52L	52L	52L	25
4	79L	81L	81L/77L	6
4	81L	52L	52L	20
4	77L	52L	52L	20
4	54	54L	54L	10
4	50	54L	81L/77L	10
4	53	54L	81L/77L	10
4	45	54L	81L/77L	10
4	51	54L	81L/77L	10
4	46	54L	81L/77L	6
4	52	54L	81L/77L	6
4	73	54L	81L/77L	6
4	43	54L	81L/77L	6
4	69	54L	81L/77L	10
4	49	54L	81L/77L	10
4	48	54L	81L/77L	6
4	65	54L	81L/77L	10
4	47	54L	81L/77L	10
4	44	54L	81L/77L	10
4	62	54L	81L/77L	10
4	75	54L	81L/77L	10

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
4	59	54L	81L/77L	10
4	42	54L	81L/77L	6
4	41	54L	81L/77L	10
4	71	54L	81L/77L	10
4	40	54L	81L/77L	10
4	64	54L	81L/77L	6
4	72	81L	81L/77L	6
4	68	81L	81L/77L	6
4	57	81L	81L/77L	6
4	58	81L	81L/77L	6
4	67	81L	81L/77L	6
4	63	81L	81L/77L	6
4	61	81L	81L/77L	12
4	70	81L	81L/77L	12
4	76	81L	81L/77L	12
4	74	81L	81L/77L	10
4	66	81L	81L/77L	6
4	55	81L	81L/77L	6
4	56	81L	81L/77L	6
4	60	81L	81L/77L	6
4	80	81L	81L/77L	6
4	79	81L	81L/77L	6
4	78	81L	81L/77L	6
4	81	81L	81L	6
4	77	77L	77L	6
5	104L	101L	101L	20
5	95L	104L	104L	10
5	101L	101L	101L	25
5	111L	101L	101L	20
5	123L	101L	127L	20
5	118L	101L	127L	20
5	114L	101L	127L	20
5	105L	101L	127L	20
5	127L	127L	127L	25
5	126L	101L	127L	20
5	104	104L	104L	10
5	96	104L	104L	10
5	103	104L	104L	6
5	94	104L	104L	6
5	95	104L	104L	10
5	100	104L	104L	10
5	93	104L	104L	10
5	102	104L	104L	10
5	98	104L	104L	10
5	88	104L	104L	12
5	91	104L	104L	10
5	84	104L	104L	6
5	89	104L	104L	6
5	121	104L	104L	6
5	92	123L	104L	6
5	113	104L	104L	10
5	90	104L	104L	10
5	101	104L	104L	10
5	83	104L	104L	12
5	99	104L	104L	10
5	112	104L	104L	6
5	119	104L	104L	16
5	108 (109)	104L	104L	16
5	86	104L	104L	16
5	97	104L	104L	16
5	125	104L	104L	16
5	87	104L	104L	10
5	117	104L	104L	12
5	116	104L	104L	12
5	85	104L	104L	10
5	110	104L	104L	10
5	115	104L	104L	10
5	82	104L	104L	6
5	111	104L	104L	6
5	120	104L	104L	6
5	107 (108)	104L	123L/114L/118L/105L/126L	10
5	124	104L	123L/114L/118L/105L/126L	10
5	109 (107)	104L	123L/114L/118L/105L/126L	6
5	123	123L	123L	6
5	106	123L	123L/114L/118L/105L/126L	6
5	118	118L	118L	6
5	122	118L	123L/114L/118L/105L/126L	6

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
5	114	114L	114L	6
5	105	105L	105L	6
5	127	105L	123L/114L/118L/105L/126L	6
5	126	126L	126L	6
6	155L	138L	101L	20
6	153L	167L	156L/157L/167L/169L	10
6	138L	138L	138L	100
6	167L	138L	138L	20
6	156L	138L	138L	6
6	157L	138L	138L	20
6	169L	138L	138L	6
6	155	155L	155L	10
6	152	155L	155L	6
6	150	155L	155L	6
6	136	155L	155L	6
6	145	155L	155L	6
6	148	155L	155L	6
6	151	155L	155L	10
6	135	155L	155L	10
6	154	155L	155L	10
6	144	155L	155L	6
6	147	155L	156L/157L/167L/169L	10
6	149	155L	156L/157L/167L/169L	10
6	134	155L	156L/157L/167L/169L	10
6	143	155L	156L/157L/167L/169L	10
6	139	155L	156L/157L/167L/169L	10
6	140	155L	156L/157L/167L/169L	10
6	131	155L	156L/157L/167L/169L	6
6	142	155L	156L/157L/167L/169L	6
6	132	155L	156L/157L/167L/169L	10
6	133	155L	156L/157L/167L/169L	6
6	165	167L	156L/157L/167L/169L	6
6	146	167L	156L/157L/167L/169L	6
6	161	167L	156L/157L/167L/169L	6
6	153	167L	156L/157L/167L/169L	10
6	168	167L	156L/157L/167L/169L	10
6	141	167L	156L/157L/167L/169L	6
6	130	167L	156L/157L/167L/169L	6
6	137	167L	156L/157L/167L/169L	6
6	164	167L	156L/157L/167L/169L	6
6	138	167L	156L/157L/167L/169L	14
6	163	167L	156L/157L/167L/169L	14
6	129	167L	156L/157L/167L/169L	14
6	160	167L	156L/157L/167L/169L	10
6	158	167L	156L/157L/167L/169L	6
6	166	167L	156L/157L/167L/169L	10
6	128	167L	156L/157L/167L/169L	10
6	159	167L	156L/157L/167L/169L	6
6	162	167L	156L/157L/167L/169L	6
6	167	167L	167L	6
6	156	156L	156L/157L	6
6	157	157L	156L/157L	10
6	169	169L	169L	6
7	188L	180L	180L	20
7	178L	180L	180L	20
7	180L	180L	180L	100
7	170L	180L	180L	20
7	189L	180L	194L	20
7	188	188L	188L	6
7	179	188L	188L/170L	6
7	184	188L	188L/170L	6
7	176	188L	188L/170L	6
7	186	188L	188L/170L	6
7	178	188L	188L/170L	6
7	175	188L	188L/170L	6
7	187	188L	188L/170L	6
7	182	188L	188L/170L	6
7	183	188L	188L/170L	6
7	185	188L	188L/170L	6
7	174	188L	188L/170L	6
7	177	188L	188L/170L	6
7	181	188L	188L/170L	6
7	171	188L	188L/170L	10
7	173	188L	188L/170L	6
7	172	189L	188L/170L	6
7	192	189L	188L/170L	6
7	193	189L	188L/170L	6

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
7	180	189L	188L/170L	6
7	191	189L	188L/170L	6
7	170	189L	170L	6
7	190	189L	188L/170L	6
7	189	189L	189L	6
8	202L	194L	180L	20
8	194L	194L	194L	25
8	205L	194L	194L	30
8	202	202L	202L	10
8	201	202L	202L	6
8	204	202L	202L	6
8	197	202L	202L	6
8	200	202L	202L	6
8	198	202L	202L	10
8	199	202L	202L	6
8	196	205L	202L	6
8	203	205L	202L	6
8	195	205L	205L	6
8	194	205L	205L	6
8	205	205L	205L	6
9	208L	194L	194L	20
9	206L	194L	194L	30
9	208	208L	208L	6
9	207	208L	208L/206L	6
9	206	206L	206L	6
10	209L	194L	194L	30
10	209	209L	209L	6

1. Number of chlorines on congener.
2. Suffix "L" indicates labeled compound.
3. IUPAC Number per Table 2 of Method 1668A. (Numbers in parentheses are PCB Numbers from 1668C)
4. Retention time reference that is used to locate target congener.
5. Quantitation reference that is used to calculate the concentration of the target congener or labeled standard.
6. RT window width for congener or group of two or more congeners.

Table 3 - Low Calibration Levels (LCLs) Based on Final Extract Volumes

		20 µL Extract Volume	100µL Extract Volume
Analyte	Minimum Cal. Level CS 0.5 (ng/mL)	Water 1L (ng/L)	Solids and Tissues 10g[2] (ng/g)
Monochlorobiphenyls	0.5	0.01[1]	0.005
Dichlorobiphenyls	0.5	0.01[1]	0.005
Trichlorobiphenyls	0.5	0.01	0.005
Tetrachlorobiphenyls	0.5	0.01	0.005
Pentachlorobiphenyls	0.5	0.01	0.005
Hexachlorobiphenyls	0.5	0.01	0.005
Heptachlorobiphenyls	0.5	0.01	0.005
Octachlorobiphenyls	0.5	0.01	0.005
Nonachlorobiphenyls	0.5	0.01	0.005
Decachlorobiphenyl	0.5	0.01	0.005

1. This value reflects the LCL. Reliable detection at this level may not be attained due to evaporative loss in adjusting the extract volume to 20 µL for these homolog groups.

2. The values for solids and tissues reflect the LCLs for Protocol 1 as described in Table 12. If the sample is prepared by another protocol described in that table, the LCLs shown in this table must be adjusted appropriately.

Table 4 - Estimated Minimum Levels EPA 1668A, B & C- Matrix and Concentration

	TestAmerica Knoxville EMLs[1] (1668A, B & C)			**1668C Reference Method EMLs[2]**		
Parameter	**Water ng/L**	**Other[3] ng/g**	**Extract ng/mL**	**Water ng/L**	**Other[3] ng/g**	**Extract ng/mL**
PCB 1	0.040	0.01	1.0	0.02	0.002	1
PCB 2	0.040	0.01	1.0	0.02	0.002	1
PCB 3	0.040	0.01	1.0	0.05	0.005	2.5
PCB 4	0.060	0.02	2.0	0.05	0.005	2.5
PCB 5	0.040	0.01	1.0	0.02	0.002	1
PCB 6	0.040	0.01	1.0	0.02	0.002	1
PCB 7	0.040	0.01	1.0	0.02	0.002	1
PCB 8	0.060	0.02	2.0	0.05	0.005	2.5
PCB 9	0.040	0.01	1.0	0.02	0.002	1
PCB 10	0.040	0.01	1.0	0.05	0.005	2.5
PCB 11	0.060	0.02	2.0	0.10	0.01	5
PCB 12	0.060	0.01	1.0	0.05	0.005	2.5
PCB 14	0.040	0.01	1.0	0.02	0.002	1
PCB 13	0.060	0.01	1.0	0.05	0.005	2.5
PCB 15	0.040	0.01	1.0	0.05	0.005	2.5
PCB 16	0.040	0.01	1.0	0.02	0.002	1
PCB 17	0.040	0.01	1.0	0.02	0.002	1
PCB 18	0.060	0.02	2.0	0.05	0.005	2.5
PCB 30	0.060	0.02	2.0	0.05	0.005	2.5
PCB 19	0.040	0.01	1.0	0.02	0.002	1
PCB 20	0.040	0.02	2.0	0.05	0.005	2.5
PCB 28	0.040	0.02	2.0	0.05	0.005	2.5
PCB 21	0.040	0.01	1.0	0.05	0.005	2.5
PCB33	0.040	0.01	1.0	0.05	0.005	2.5
PCB 22	0.040	0.01	1.0	0.02	0.002	1
PCB 23	0.040	0.01	1.0	0.02	0.002	1
PCB 24	0.040	0.01	1.0	0.02	0.002	1
PCB 25	0.040	0.01	1.0	0.02	0.002	1
PCB 26	0.040	0.01	1.0	0.05	0.005	2.5
PCB 29	0.040	0.01	1.0	0.05	0.005	2.5
PCB 27	0.040	0.01	1.0	0.02	0.002	1
PCB 31	0.040	0.02	2.0	0.05	0.005	2.5
PCB 32	0.040	0.01	1.0	0.02	0.002	1
PCB 34	0.040	0.01	1.0	0.02	0.002	1
PCB 35	0.040	0.01	1.0	0.02	0.002	1
PCB 36	0.040	0.01	1.0	0.02	0.002	1
PCB 37	0.040	0.01	1.0	0.02	0.002	1
PCB 38	0.040	0.01	1.0	0.02	0.002	1
PCB 39	0.040	0.01	1.0	0.02	0.002	1
PCB 40	0.040	0.01	1.0	0.1	0.01	5
PCB 41	0.040	0.01	1.0	0.1	0.01	5
PCB 71	0.040	0.01	1.0	0.1	0.01	5
PCB 42	0.040	0.01	1.0	0.05	0.005	2.5
PCB 43	0.040	0.01	1.0	0.05	0.005	2.5
PCB 73	0.040	0.01	1.0	0.05	0.005	2.5
PCB 44	0.040	0.01	1.0	0.1	0.01	5
PCB 47	0.040	0.01	1.0	0.1	0.01	5
PCB 65	0.040	0.01	1.0	0.1	0.01	5
PCB 45	0.040	0.01	1.0	0.05	0.005	2.5
PCB 51	0.040	0.01	1.0	0.05	0.005	2.5
PCB 46	0.040	0.01	1.0	0.02	0.002	1
PCB 48	0.040	0.01	1.0	0.05	0.005	2.5
PCB 49	0.040	0.01	1.0	0.1	0.01	5
PCB 69	0.040	0.01	1.0	0.1	0.01	5
PCB 50	0.040	0.01	1.0	0.1	0.01	5
PCB 53	0.040	0.01	1.0	0.1	0.01	5
PCB 52	0.040	0.01	1.0	0.05	0.005	2.5

	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
Parameter	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**
PCB 54	0.040	0.01	1.0	0.05	0.005	2.5
PCB 55	0.040	0.01	1.0	0.05	0.005	2.5
PCB 56	0.040	0.01	1.0	0.05	0.005	2.5
PCB 57	0.040	0.01	1.0	0.05	0.005	2.5
PCB 58	0.040	0.01	1.0	0.05	0.005	2.5
PCB 59	0.040	0.01	1.0	0.1	0.01	5
PCB 62	0.040	0.01	1.0	0.1	0.01	5
PCB 75	0.040	0.01	1.0	0.1	0.01	5
PCB 60	0.040	0.01	1.0	0.05	0.005	2.5
PCB 61	0.040	0.02	2.0	0.2	0.02	10
PCB 70	0.040	0.02	2.0	0.2	0.02	10
PCB 74	0.040	0.02	2.0	0.2	0.02	10
PCB 76	0.040	0.02	2.0	0.2	0.02	10
PCB 63	0.040	0.01	1.0	0.005	0.05	2.5
PCB 64	0.040	0.01	1.0	0.05	0.005	2.5
PCB 66	0.040	0.01	1.0	0.05	0.005	2.5
PCB 67	0.040	0.01	1.0	0.05	0.005	2.5
PCB 68	0.040	0.01	1.0	0.05	0.005	2.5
PCB 72	0.040	0.01	1.0	0.05	0.005	2.5
PCB 77	0.040	0.01	1.0	0.05	0.005	2.5
PCB 78	0.040	0.01	1.0	0.05	0.005	2.5
PCB 79	0.040	0.01	1.0	0.05	0.005	2.5
PCB 80	0.040	0.01	1.0	0.05	0.005	2.5
PCB 81	0.040	0.01	1.0	0.05	0.005	2.5
PCB 82	0.040	0.01	1.0	0.05	0.005	2.5
PCB 83	0.040	0.01	1.0	0.1	0.01	5
PCB 84	0.040	0.01	1.0	0.02	0.002	1
PCB 85	0.040	0.01	1.0	0.1	0.01	5
PCB 116	0.040	0.01	1.0	0.1	0.01	5
PCB 117	0.040	0.01	1.0	0.1	0.01	5
PCB 86	0.040	0.01	1.0	0.2	0.02	10
PCB 87	0.040	0.01	1.0	0.2	0.02	10
PCB 97	0.040	0.01	1.0	0.2	0.02	10
PCB 109	0.040	0.01	1.0	0.2	0.02	10
PCB 119	0.040	0.01	1.0	0.2	0.02	10
PCB 125	0.040	0.01	1.0	0.2	0.02	10
PCB 88	0.040	0.01	1.0	0.05	0.005	2.5
PCB 91	0.040	0.01	1.0	0.05	0.005	2.5
PCB 89	0.040	0.01	1.0	0.05	0.005	2.5
PCB 90	0.040	0.01	1.0	0.2	0.02	10
PCB 101	0.040	0.01	1.0	0.2	0.02	10
PCB 113	0.040	0.01	1.0	0.2	0.02	10
PCB 92	0.040	0.01	1.0	0.05	0.005	2.5
PCB 93	0.040	0.01	1.0	0.2	0.02	10
PCB 100	0.040	0.01	1.0	0.2	0.02	10
PCB 94	0.040	0.01	1.0	0.05	0.005	2.5
PCB 95	0.040	0.01	1.0	0.2	0.02	10
PCB 96	0.040	0.01	1.0	0.05	0.005	2.5
PCB 98	0.040	0.01	1.0	0.2	0.02	10
PCB 102	0.040	0.01	1.0	0.2	0.02	10
PCB 99	0.040	0.01	1.0	0.1	0.01	5
PCB 112	0.040	0.01	1.0	0.05	0.005	2.5
PCB 103	0.040	0.01	1.0	0.05	0.005	2.5
PCB 104	0.040	0.01	1.0	0.05	0.005	2.5
PCB 105	0.040	0.01	1.0	0.05	0.005	2.5
PCB 106	0.040	0.01	1.0	0.05	0.005	2.5
PCB 107	0.040	0.01	1.0	0.05	0.005	2.5
PCB 108	0.040	0.01	1.0	0.1	0.01	5
PCB 124	0.040	0.01	1.0	0.1	0.01	5
PCB 110	0.040	0.01	1.0	0.1	0.01	5

	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
Parameter	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**
PCB 115	0.040	0.01	1.0	0.1	0.01	5
PCB 111	0.040	0.01	1.0	0.05	0.005	2.5
PCB 114	0.040	0.01	1.0	0.05	0.005	2.5
PCB 118	0.040	0.01	1.0	0.1	0.01	5
PCB 120	0.040	0.01	1.0	0.05	0.005	2.5
PCB 121	0.040	0.01	1.0	0.05	0.005	2.5
PCB 122	0.040	0.01	1.0	0.05	0.005	2.5
PCB 123	0.040	0.01	1.0	0.05	0.005	2.5
PCB 126	0.040	0.01	1.0	0.05	0.005	2.5
PCB 127	0.040	0.01	1.0	0.05	0.005	2.5
PCB 128	0.040	0.01	1.0	0.1	0.01	5
PCB 166	0.040	0.01	1.0	0.1	0.01	5
PCB 129	0.040	0.01	1.0	0.2	0.02	10
PCB 138	0.040	0.01	1.0	0.2	0.02	10
PCB 163	0.040	0.01	1.0	0.2	0.02	10
PCB 130	0.040	0.01	1.0	0.05	0.005	2.5
PCB 131	0.040	0.01	1.0	0.05	0.005	2.5
PCB 132	0.040	0.01	1.0	0.05	0.005	2.5
PCB 133	0.040	0.01	1.0	0.05	0.005	2.5
PCB 134	0.040	0.01	1.0	0.1	0.01	5
PCB 143	0.040	0.01	1.0	0.1	0.01	5
PCB 135	0.040	0.01	1.0	0.1	0.01	5
PCB 151	0.040	0.01	1.0	0.1	0.01	5
PCB 136	0.040	0.01	1.0	0.05	0.005	2.5
PCB 137	0.040	0.01	1.0	0.05	0.005	2.5
PCB 164	0.040	0.01	1.0	0.05	0.005	2.5
PCB 139	0.040	0.01	1.0	0.1	0.01	5
PCB 140	0.040	0.01	1.0	0.1	0.01	5
PCB 141	0.040	0.01	1.0	0.05	0.005	2.5
PCB 142	0.040	0.01	1.0	0.05	0.005	2.5
PCB 144	0.040	0.01	1.0	0.05	0.005	2.5
PCB 145	0.040	0.01	1.0	0.05	0.005	2.5
PCB 146	0.040	0.01	1.0	0.05	0.005	2.5
PCB 147	0.040	0.01	1.0	0.1	0.01	5
PCB 149	0.040	0.01	1.0	0.1	0.01	5
PCB 148	0.040	0.01	1.0	0.05	0.005	2.5
PCB 150	0.040	0.01	1.0	0.05	0.005	2.5
PCB 152	0.040	0.01	1.0	0.05	0.005	2.5
PCB 153	0.040	0.01	1.0	0.1	0.01	5
PCB 168	0.040	0.01	1.0	0.1	0.01	5
PCB 154	0.040	0.01	1.0	0.1	0.01	5
PCB 155	0.040	0.01	1.0	0.05	0.005	2.5
PCB 156	0.040	0.01	1.0	0.1	0.01	5
PCB 157	0.040	0.01	1.0	0.1	0.01	5
PCB 158	0.040	0.01	1.0	0.05	0.005	2.5
PCB 159	0.040	0.01	1.0	0.05	0.005	2.5
PCB 160	0.040	0.01	1.0	0.2	0.02	10
PCB 161	0.040	0.01	1.0	0.05	0.005	2.5
PCB 162	0.040	0.01	1.0	0.05	0.005	2.5
PCB 165	0.040	0.01	1.0	0.05	0.005	2.5
PCB 167	0.040	0.01	1.0	0.05	0.005	2.5
PCB 169	0.040	0.01	1.0	0.05	0.005	2.5
PCB 170	0.040	0.01	1.0	0.05	0.005	2.5
PCB 171	0.040	0.01	1.0	0.1	0.01	5
PCB 173	0.040	0.01	1.0	0.1	0.01	5
PCB 172	0.040	0.01	1.0	0.05	0.005	2.5
PCB 174	0.040	0.01	1.0	0.05	0.005	2.5
PCB 175	0.040	0.01	1.0	0.05	0.005	2.5
PCB 176	0.040	0.01	1.0	0.05	0.005	2.5
PCB 177	0.040	0.01	1.0	0.05	0.005	2.5

	TestAmerica Knoxville EMLs [1] (1668A, B & C)			**1668C Reference Method EMLs [2]**		
Parameter	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**
PCB 178	0.040	0.01	1.0	0.05	0.005	2.5
PCB 179	0.040	0.01	1.0	0.05	0.005	2.5
PCB 180	0.040	0.01	1.0	0.1	0.01	5
PCB 193	0.040	0.01	1.0	0.1	0.01	5
PCB 181	0.040	0.01	1.0	0.05	0.005	2.5
PCB 182	0.040	0.01	1.0	0.05	0.005	2.5
PCB 183	0.040	0.01	1.0	0.1	0.01	5
PCB 185	0.040	0.01	1.0	0.1	0.01	5
PCB 184	0.040	0.01	1.0	0.05	0.005	2.5
PCB 186	0.040	0.01	1.0	0.05	0.005	2.5
PCB 187	0.040	0.01	1.0	0.05	0.005	2.5
PCB 188	0.040	0.01	1.0	0.05	0.005	2.5
PCB 189	0.040	0.01	1.0	0.05	0.005	2.5
PCB 190	0.040	0.01	1.0	0.05	0.005	2.5
PCB 191	0.040	0.01	1.0	0.05	0.005	2.5
PCB 192	0.040	0.01	1.0	0.05	0.005	2.5
PCB 194	0.040	0.01	1.0	0.05	0.005	2.5
PCB 195	0.040	0.01	1.0	0.05	0.005	2.5
PCB 196	0.040	0.01	1.0	0.05	0.005	2.5
PCB 197	0.040	0.01	1.0	0.1	0.01	5
PCB 200	0.040	0.01	1.0	0.1	0.01	5
PCB 198	0.040	0.01	1.0	0.1	0.01	5
PCB 199	0.040	0.01	1.0	0.1	0.01	5
PCB 201	0.040	0.01	1.0	0.05	0.005	2.5
PCB 202	0.040	0.01	1.0	0.1	0.01	5
PCB 203	0.040	0.01	1.0	0.05	0.005	2.5
PCB 204	0.040	0.01	1.0	0.05	0.005	2.5
PCB 205	0.040	0.01	1.0	0.05	0.005	2.5
PCB 206	0.040	0.01	1.0	0.05	0.005	2.5
PCB 207	0.040	0.01	1.0	0.05	0.005	2.5
PCB 208	0.040	0.01	1.0	0.05	0.005	2.5
PCB 209	0.040	0.01	1.0	0.05	0.005	2.5

[1] The estimated minimum level (EML) is defined as the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present assuming a sample is extracted at the recommended weight or volume and is carried through all normal extraction and analysis procedures The values for other matrices (solids, tissues and solid wastes) reflect the EMLs for Protocol 1 as described in Table 12. If the sample is prepared by another protocol described in that section, the EMLs shown in this table must be adjusted appropriately. The EMLs are based on the mean plus 2 standard deviations for matrix-pooled historical blank data and calibration data obtained while performing EPA 1668A. The survey period was fourteen months, ending in February 2004. Individual EMLs may be adjusted to reflect more recent data.

[2] For reference purposes only, not currently used by TestAmerica Knoxville

[3] Other matrices include solids, tissues and solid wastes

Table 5a- Concentration of Native PCB Congener Stock and Spiking Solutions

Native PCB Congener	**PCB**	**Standard Source**	**Catalog Number**	**Vendor Conc (ng/mL)**	**LCS Spiking Solution Conc (ng/mL)**
2-MoCB	1	AccuStd	S-99994-4x	4000	5.0
4-MoCB	3	AccuStd	S-99994-4x	4000	5.0
2,2’-DiCB	4	AccuStd	S-99994-4x	4000	5.0
4,4’-DiCB	15	AccuStd	S-99994-4x	4000	5.0
2,2’,6-TrCB	19	AccuStd	S-99994-4x	4000	5.0
3,4,4’-TrCB	37	AccuStd	S-99994-4x	4000	5.0
2,2’,6,6’-TeCB	54	AccuStd	S-99994-4x	4000	5.0
3,3’,4,4’-TeCB	77	AccuStd	S-99994-4x	4000	5.0
3,4,4’,5-TeCB	81	AccuStd	S-99994-4x	4000	5.0
2,2’,4,6,6’-PeCB	104	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’-PeCB	105	AccuStd	S-99994-4x	4000	5.0
2,3,4,4’,5-PeCB	114	AccuStd	S-99994-4x	4000	5.0
2,3’,4,4’,5-PeCB	118	AccuStd	S-99994-4x	4000	5.0
2’,3,4,4’,5-PeCB	123	AccuStd	S-99994-4x	4000	5.0
3,3’,4,4’,5-PeCB	126	AccuStd	S-99994-4x	4000	5.0
2,2’,4,4’,6,6’-HxCB	155	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’,5-HxCB	156	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’,5’-HxCB	157	AccuStd	S-99994-4x	4000	5.0
2,3’,4,4’,5,5’-HxCB	167	AccuStd	S-99994-4x	4000	5.0
3,3’,4,4’,5,5’-HxCB	169	AccuStd	S-99994-4x	4000	5.0
2,2’,3,3’,4,4’,5-HpCB	170	AccuStd	S-99994-4x	4000	5.0
2,2’,3,4,4’,5,5’-HpCB	180	AccuStd	S-99994-4x	4000	5.0
2,2’,3,4’,5,6,6’-HpCB	188	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’,5,5’-HpCB	189	AccuStd	S-99994-4x	4000	5.0
2,2’,3,3’,5,5’,6,6’-OcCB	202	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’,5,5’,6-OcCB	205	AccuStd	S-99994-4x	4000	5.0
2,2’,3,3’,4,4’,5,5’,6-NoCB	206	AccuStd	S-99994-4x	4000	5.0
2,2’,3,3’,4’,5,5’,6,6’-NoCB	208	AccuStd	S-99994-4x	4000	5.0
DeCB	209	AccuStd	S-99994-4x	4000	5.0
All other CB congeners	NA	AccuStd	S-99994-4x	4000	5.0

Table 5b: Concentration of $^{13}C_{12}$ Labeled PCB Congener Stock and Spiking Solutions

Labeled PCB Congener	**PCB**	**Standard Source**	**Catalog Number**	**Vendor Conc (ng/mL)**	Stock Conc (ng/mL)	Spiking Solution Conc (ng/mL)
Internal Standards						
$^{13}C_{12}$-2-chlorobiphenyl	1L	Cambridge	EC-4908	40,000	1000	10
$^{13}C_{12}$-4-chlorobiphenyl	3L	Cambridge	EC-4990	40,000	1000	10
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	Cambridge	EC-4911	40,000	1000	10
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	Cambridge	EC-1402	40,000	1000	10
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	Cambridge	EC-4909	40,000	1000	10
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	Cambridge	EC-4901	40,000	1000	10
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	Cambridge	EC-4912	40,000	1000	10
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	Cambridge	EC-1404	40,000	1000	10
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	Cambridge	EC-1412	40,000	1000	10
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	Cambridge	EC-4910	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	Cambridge	EC-1420	40,000	1000	10
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	Cambridge	EC-4902	40,000	1000	10
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	Cambridge	EC-1435	40,000	1000	10
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	Cambridge	EC-4904	40,000	1000	10
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	Cambridge	EC-1425	40,000	1000	10
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	Cambridge	EC-4167	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	Cambridge	EC-1422	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	Cambridge	EC-4051	40,000	1000	10
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	Cambridge	EC-4050	40,000	1000	10
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	Cambridge	EC-1416	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	Cambridge	EC-4905	40,000	1000	10
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	Cambridge	EC-4913	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	Cambridge	EC-1409	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	Cambridge	EC-1408	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	Cambridge	EC-4199	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	Cambridge	EC-4900	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	Cambridge	EC-1419	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	Cambridge	EC-1410	40,000	1000	10
Recovery Standards						
$^{13}C_{12}$-2,5-dichlorobiphenyl	9L	Cambridge	EC-4165	40,000	1000	100
$^{13}C_{12}$-2,4',5-trichlorobiphenyl	31L	Wellington	MBP-31	50,000	1000	100
$^{13}C_{12}$-2,4',6-trichlorobiphenyl	32L	Cambridge	EC-4163	40,000	1000	100
$^{13}C_{12}$-2,2',5,5'-tetrachlorobiphenyl	52L	Cambridge	EC-1424	40,000	1000	100
$^{13}C_{12}$-2,2',4,5,5'-pentachlorobiphenyl	101L	Cambridge	EC-1405	40,000	1000	100
$^{13}C_{12}$-3,3',4,5,5'-pentachlorobiphenyl	127L	Cambridge	EC-1421	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5'-hexachlorobiphenyl	138L	Cambridge	EC-1436	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-heptachlorobiphenyl	180L	Cambridge	EC-1407	40,000	1000	100
$^{13}C_{12}$-2,2',3,3',4,4’,5,5'-octachlorobiphenyl	194L	Cambridge	EC-1418	40,000	1000	100
Cleanup Standards						
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	Cambridge	EC-1413	40,000	5000	10
$^{13}C_{12}$-2,3,3’,5,5’-pentachlorobiphenyl	111L	Cambridge	EC-1415	40,000	5000	10
$^{13}C_{12}$-2,2’,3,3’,5,5’,6-heptachlorobiphenyl	178L	Cambridge	EC-1417	40,000	5000	10
Sampling Surrogate Standards						
$^{13}C_{12}$-2,4'-dichlorobiphenyl	8L	Cambridge	EC-5095	40,000	5000	50
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	79L	Cambridge	EC-5048	40,000	5000	50
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	95L	Wellington	MBP-95	50,000	5000	50
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	153L	Cambridge	EC-1406	40,000	5000	50

Table 6a - Concentration of PCBs in Calibration Solutions

		CS 0.5	**CS 1**	**CS 2**	**CS 3[2]**	**CS 4**	**CS 5**
Analyte Type	**PCB[1]**	**ng/mL**	**ng/mL**	**ng/mL**	**ng/mL**	**ng/mL**	**ng/mL**
Congeners							
2-MoCB	1	0.5	1.0	5.0	50	400	2000
4-MoCB	3	0.5	1.0	5.0	50	400	2000
2,2'-DiCB	4	0.5	1.0	5.0	50	400	2000
4,4'-DiCB	15	0.5	1.0	5.0	50	400	2000
2,2',6'-TrCB	19	0.5	1.0	5.0	50	400	2000
3,4,4'-TrCB	37	0.5	1.0	5.0	50	400	2000
2,2',6,6'-TeCB	54	0.5	1.0	5.0	50	400	2000
3,3',4,4'-TeCB	77	0.5	1.0	5.0	50	400	2000
3,4,4',5-TeCB	81	0.5	1.0	5.0	50	400	2000
2,2',4,6,6'-PeCB	104	0.5	1.0	5.0	50	400	2000
2,3,3',4,4'-PeCB	105	0.5	1.0	5.0	50	400	2000
2,3,4,4',5-PeCB	114	0.5	1.0	5.0	50	400	2000
2,3',4,4',5-PeCB	118	0.5	1.0	5.0	50	400	2000
2',3,4,4',5-PeCB	123	0.5	1.0	5.0	50	400	2000
3,3',4,4',5-PeCB	126	0.5	1.0	5.0	50	400	2000
2,2',4,4',6,6'-HxCB	155	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5-HxCB	156	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5'-HxCB	157	0.5	1.0	5.0	50	400	2000
2,3',4,4',5,5'-HxCB	167	0.5	1.0	5.0	50	400	2000
3,3',4,4',5,5'-HxCB	169	0.5	1.0	5.0	50	400	2000
2,2',3,4',5,6,6'-HpCB	188	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5,5'-HpCB	189	0.5	1.0	5.0	50	400	2000
2,2',3,3',5,5',6,6'-OcCB	202	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5,5',6-OcCB	205	0.5	1.0	5.0	50	400	2000
2,2',3,3',4,4',5,5',6-NoCB	206	0.5	1.0	5.0	50	400	2000
2,2',3,3',4',5,5',6,6'-NoCB	208	0.5	1.0	5.0	50	400	2000
DeCB	209	0.5	1.0	5.0	50	400	2000
All other CB congeners		0.5	1.0	5.0	50	400	2000
Labeled Congeners							
$^{13}C_{12}$-2-MoCB	1L	100	100	100	100	100	100
$^{13}C_{12}$-4-MoCB	3L	100	100	100	100	100	100
$^{13}C_{12}$-2,2'-DiCB	4L	100	100	100	100	100	100
$^{13}C_{12}$-4,4'-DiCB	15L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6-TrCB	19L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4’,5-TeCB	81L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	100	100	100	100	100
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	100	100	100	100	100

Analyte Type	PCB[1]	CS 0.5 ng/mL	CS 1 ng/mL	CS 2 ng/mL	CS 3[2] ng/mL	CS 4 ng/mL	CS 5 ng/mL
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4',5,5',6,6'-NoCB	208L	100	100	100	100	100	100
$^{13}C_{12}$-DeCB	209L	100	100	100	100	100	100
Cleanup Standards							
$^{13}C_{12}$-2,4,4'-TriCB	28L			5.0	50	400	--
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L			5.0	50	400	--
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L			5.0	50	400	--
Recovery Standards							
$^{13}C_{12}$-2,5-DiCB	9L	100	100	100	100	100	100
$^{13}C_{12}$-2,4',5-TriCB	31L	100	100	100	100	100	100
$^{13}C_{12}$-2,4',6-TriCB	32L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4',5,5'-PeCB	101L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,5,5'-PeCB	127L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	138L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	100	100	100	100	100	100
Labeled Sampling Surrogates							
$^{13}C_{12}$-2,4'-DiCB	8L			5.0	50	400	--
$^{13}C_{12}$-3,3',4,5'-TeCB	79L			5.0	50	400	--
$^{13}C_{12}$-2,2',3,5',6-PeCB	95L			5.0	50	400	--
$^{13}C_{12}$-2,2',4,4',5,5'-HxCB	153L			5.0	50	400	--

Notes:

1. Suffix “L” indicates labeled compound.
2. The CS 3 standard is also used as the calibration verification solution.

Table 6b – Preparation of Calibration Solutions

Calibration Standard ID	Parent Standards	Parent Conc. (ng/mL)	Volume Added (mL)	Final Volume (mL)	Final Conc (ng/mL)
CS 0.5	Native PCB Congener Stock Solution	40	0.0125	1.0	0.50
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100
CS 1	Native PCB Congener Stock Solution	40	0.0250	1.0	1.0
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100
CS 2	Native PCB Congener Stock Solution	40	0.1250	1.0	5.0
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100
	Cleanup Standard Stock Solution	5000	0.0010		5.0
	Sampling Surrogate Stock Solution	5000	0.0010		5.0
CS 3	Native PCB Congener Standard Mix	4000	0.0375	3.0	50
	Internal Standard Stock Solution	1000	0.3000		100
	Recovery Standard Stock Solution	1000	0.3000		100
	Cleanup Standard Stock Solution	5000	0.0300		50
	Sampling Surrogate Stock Solution	5000	0.0300		50
CS 4	Native PCB Congener Standard Mix	4000	0.1000	1.0	400
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100
	Cleanup Standard Stock Solution	5000	0.0800		400
	Sampling Surrogate Stock Solution	5000	0.0800		400
CS 5	Native PCB Congener Standard Mix	4000	0.5000	1.0	2000
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100

Table 7 - GC Window Defining Mixture and SPB –Octyl Resolution Test Compounds

SPB-Octyl

Congener Group		First Eluted		Last Eluted
Mono	1	2-	3	4-
Di	4	2,2’-	15	4,4’-
Tri	19	2,2’,6-	37	3,4,4’-
Tetra	54	2,2’,6,6’-	77	3,3’,4,4’-
Penta	104	2,2’,4,6,6’-	126	3,3’,4,4’,5-
Hexa	155	2,2’,4,4’,6,6’-	169	3,3’,4,4’,5,5’-
Hepta	188	2,2’,3,4’,5,6,6’-	189	2,3,3’,4,4’,5,5’-
Octa	202	2,2’,3,3’,5,5’,6,6’-	205	2,3,3’,4,4’,5,5’,6-
Nona	208	2,2’,3,3’,4,5,5’,6,6’-	206	2,2’,3,3’,4,4’,5,5’,6-
Deca	209	2,2’,3,3’,4,4’,5,5’,6,6’-	209	2,2’,3,3’,4,4’,5,5’,6,6’-

SPB-Octyl Resolution Test Compounds

23	2,3,5-trichlorobiphenyl
34	2’,3,5-trichlorobiphenyl (2,3’,5’-trichlorobiphenyl)
182	2,2’,3,4,4’,5,6’-heptachlorobiphenyl
187	2,2’,3,4’,5,5’,6-heptachlorobiphenyl

Table 8 - Ions Monitored for HRGC/HRMS Analysis of PCBs

Descriptor	Accurate Mass	Ion ID	Elemental Composition	Analyte
1	180.9888	Lock	C_4F_7	PFK
	188.0393	M	$C_{12}H_9{}^{35}Cl$	Mono
	190.0363	M+2	$C_{12}H_9{}^{37}Cl$	Mono
	200.0795	M	$^{13}C_{12}H_9{}^{35}Cl$	Mono-13C12
	202.0766	M+2	$^{13}C_{12}H_9{}^{37}Cl$	Mono-13C12
	222.0003	M	$C_{12}H_8{}^{35}Cl_2$	Di
	223.9974	M+2	$C_{12}H_8{}^{35}Cl^{37}Cl$	Di
	234.0406	M	$^{13}C_{12}H_8{}^{35}Cl_2$	Di-13C
	236.0376	M+2	$^{13}C_{12}H_8{}^{35}Cl^{37}Cl$	Di-13C
	255.9613	M	$C_{12}H_7{}^{35}Cl_3$	Tri
	257.9584	M+2	$C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	Tri-13C
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri-13C
	280.9824	QC	C_6F_{11}	PFK
	289.9224	M	$C_{12}H_6{}^{35}Cl_4$	Tetra
	291.9194	M+2	$C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra
	301.9626	M	$^{13}C_{12}H_6{}^{35}Cl_4$	Tetra-13C
	303.9597	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra-13C
2	255.9613	M	$C_{12}H_7{}^{35}Cl_3$	Tri
	257.9584	M+2	$C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	Tri-13C
	268.9824	Lock	C_5F_{11}	PFK
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri-13C
	289.9224	M	$C_{12}H_6{}^{35}Cl_4$	Tetra
	291.9194	M+2	$C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra
	301.9626	M	$^{13}C_{12}H_6{}^{35}Cl_4$	Tetra-13C
	303.9597	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra-13C
	325.8804	M+2	$C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta
	327.8775	M+4	$C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta-13C
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta-13C
	359.8415	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa
	361.8385	M+4	$C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa-13C
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa-13C
	380.9760	QC	$C_{10}F_{14}$	PFK

Table 8 - Ions Monitored for HRGC/HRMS Analysis of PCBs (continued)

Descriptor	Accurate Mass	Ion ID	Elemental Composition	Analyte
3	325.8804	M+2	$C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta
	327.8775	M+4	$C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta
	337.9207	M+2	${}^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta-13C
	339.9178	M+4	${}^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta-13C
	342.9792	Lock	C_8F_{13}	PFK
	359.8415	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa
	361.8385	M+4	$C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa
	371.8817	M+2	${}^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa-13C
	373.8788	M+4	${}^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa-13C
	393.8025	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta
	395.7995	M+4	$C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta
	405.8428	M+2	${}^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta-13C
	407.8398	M+4	${}^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta-13C
	427.7635	M+2	$C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa
	429.7606	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa
	430.9728	QC	C_9F_{17}	PFK
	439.8038	M+2	${}^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa-13C
	441.8008	M+4	${}^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa-13C
4	393.8025	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta
	395.7995	M+4	$C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta
	404.9760	Lock	$C_{10}F_{15}$	PFK
	405.8428	M+2	${}^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta-13C
	407.8398	M+4	${}^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta-13C
	427.7635	M+2	$C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa
	429.7606	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa
	439.8038	M+2	${}^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa-13C
	441.8008	M+4	${}^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa-13C
	461.7246	M+2	$C_{12}H{}^{35}Cl_7{}^{37}Cl_2$	Nona
	463.7216	M+4	$C_{12}H{}^{35}Cl_6{}^{37}Cl_3$	Nona
	473.7648	M+2	${}^{13}C_{12}H{}^{35}Cl_7{}^{37}Cl_2$	Nona-13C
	475.7619	M+4	${}^{13}C_{12}H{}^{35}Cl_6{}^{37}Cl_3$	Nona-13C
	495.6856	M+2	$C_{12}{}^{35}Cl_8{}^{37}Cl_2$	Deca
	497.6826	M+4	$C_{12}{}^{35}Cl_7{}^{37}Cl_3$	Deca
	504.9697	QC	$C_{12}F_{19}$	PFK
	507.7258	M+2	${}^{13}C_{12}{}^{35}Cl_8{}^{37}Cl_2$	Deca-13C
	509.7229	M+4	${}^{13}C_{12}{}^{35}Cl_7{}^{37}Cl_3$	Deca-13C

1. Nuclidic masses used: H = 1.007825 C = 12.00000 ${}^{13}C$ = 13.003355 F = 18.9984

O = 15.994915 ${}^{35}Cl$ = 34.968853 ${}^{37}Cl$ = 36.965903

Table 9 - Theoretical Ion Abundance Ratios and Control Limits for PCBs

Chlorine Atoms	mass's Forming Ratios	Theoretical Ratio	Lower QC Limit	Upper QC Limit
1	m/m+2	3.13	2.66	3.60
2	m/m+2	1.56	1.33	1.79
3	m/m+2	1.04	0.88	1.20
4	m/m+2	0.77	0.65	0.89
5	m+2/m+4	1.55	1.32	1.78
6	m+2/m+4	1.24	1.05	1.43
7	m+2/m+4	1.05	0.89	1.21
8	m+2/m+4	0.89	0.76	1.02
9	m+2/m+4	0.77	0.65	0.89
10	m+2/m+4	0.69	0.59	0.79

Table 10A - Acceptance Criteria for Performance Tests (1668A & B)

			IPR (IDOC)			
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL)[1]**	**%RSD**	**%R**	**VER %R**	**LCS %R**
2-chlorobiphenyl	1	50	40	60-140	70-130	50-150
4-chlorobiphenyl	3	50	40	60-140	70-130	50-150
2,2’-dichlorobiphenyl	4	50	40	60-140	70-130	50-150
4,4’-dichlorobiphenyl	15	50	40	60-140	70-130	50-150
2,2’,6-trichlorobiphenyl	19	50	40	60-140	70-130	50-150
3,4,4’-trichlorobiphenyl	37	50	40	60-140	70-130	50-150
2,2’,6,6’-tetrachlorobiphenyl	54	50	40	60-140	70-130	50-150
3,3’,4,4’-tetrachlorobiphenyl	77	50	40	60-140	70-130	50-150
3,4,4’,5-tetrachlorobiphenyl	81	50	40	60-140	70-130	50-150
2,2’4,6,6’-pentachlorobiphenyl	104	50	40	60-140	70-130	50-150
2,3,3’,4,4’-pentachlorobiphenyl	105	50	40	60-140	70-130	50-150
2,3,4,4’,5-pentachlorobiphenyl	114	50	40	60-140	70-130	50-150
2,3’,4,4’,5-pentachlorobiphenyl	118	50	40	60-140	70-130	50-150
2’,3’,4,4’,5-pentachlorobiphenyl	123	50	40	60-140	70-130	50-150
3,3’,4,4’,5-pentachlorobiphenyl	126	50	40	60-140	70-130	50-150
2,2’,4,4’,6,6’-hexachlorobiphenyl	155	50	40	60-140	70-130	50-150
2,3,3’,4,4’,5-hexachlorobiphenyl	156	50	40	60-140	70-130	50-150
2,3’,4,4’,5,5’-hexachlorobiphenyl	157	50	40	60-140	70-130	50-150
2,3,3’,4,4’,5’-hexachlorobiphenyl	167	50	40	60-140	70-130	50-150
3,3’,4,4’,5,5’-hexachlorobiphenyl	169	50	40	60-140	70-130	50-150
2,2’,3,4’5,6,6’-heptachlorobiphenyl	188	50	40	60-140	70-130	50-150
2,3,3’,4,4’,5,5’-heptachlorobiphenyl	189	50	40	60-140	70-130	50-150
2,2’,3,3',5,5',6,6’-octachlorobiphenyl	202	50	40	60-140	70-130	50-150
2,3,3',4,4',5,5',6-octachlorobiphenyl	205	50	40	60-140	70-130	50-150
2,2’,3,3’,4,4’,5,5’,6-nonachlorobiphenyl	206	50	40	60-140	70-130	50-150
2,2’,3,3’,4,5,5’,6,6’-nonachlorobiphenyl	208	50	40	60-140	70-130	50-150
2,2’,3,3’,4,4’,5,5’,6,6’-decachlorobiphenyl	209	50	40	60-140	70-130	50-150
Internal Standards						
$^{13}C_{12}$-2-chlorobiphenyl	1L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-4-chlorobiphenyl	3L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2’-dichlorobiphenyl	4L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-4,4’-dichlorobiphenyl	15L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2’,6-trichlorobiphenyl	19L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,4,4’-trichlorobiphenyl	37L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2’,6,6’-tetrachlorobiphenyl	54L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,3’,4,4’-tetrachlorobiphenyl	77L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,4,4’,5-tetrachlorobiphenyl	81L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2’,4,6,6’-pentachlorobiphenyl	104L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3’,4,4’-pentachlorobiphenyl	105L	100	50	35-135	50-150	30-140
$^{13}C_{12}$2,3,4,4’,5-pentachlorobiphenyl -	114L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3’,4,4’,5-pentachlorobiphenyl	118L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2’,3,4,4’,5-pentachlorobiphenyl	123L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,3’,4,4’,5-pentachlorobiphenyl	126L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2’,4,4’,6,6’-hexachlorobiphenyl	155L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3’,4,4’,5-hexachlorobiphenyl	156L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3’,4,4’,5’-hexachlorobiphenyl	157L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3’,4,4’,5,5’-hexachlorobiphenyl	167L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,3’,4,4’,5,5’-hexachlorobiphenyl	169L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2’,3,4’,5,6,6’-heptachlorobiphenyl	188L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3’,4,4’,5,5’-heptachlorobiphenyl	189L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2’,3,3’,5,5’,6,6’-octachlorobiphenyl	202L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3’,4,4’,5,5’,6-octachlorobiphenyl	205L	100	50	35-135	50-150	30-140

			IPR (IDOC)			
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL)** [1]	**%RSD**	**%R**	**VER %R**	**LCS %R**
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	100	50	35-135	50-150	30-140
Cleanup Standards						
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	50	45	45-120	60-130	40-125
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	111L	50	45	45-120	60-130	40-125
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	178L	50	45	45-120	60-130	40-125
Sampling Surrogate Standards						
$^{13}C_{12}$-2,4'-dichlorobiphenyl	8L	50			50-150	50-150
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	79L	50			50-150	50-150
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	95L	50			50-150	50-150
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	153L	50			50-150	50-150

[1] - Test concentrations are based on ng/mL in the sample extract or standard solution.

Table 10B - Acceptance Criteria for Performance Tests (1668C)

			IPR (IDOC)				
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL)** [1]	**%RSD**	**Mean %R**	**VER %R**	**OPR (LCS) %R**	**Labeled Compound %R in Samples**
2-chlorobiphenyl	1	50	25	70-130	75-125	60-135	
4-chlorobiphenyl	3	50	25	70-130	75-125	60-135	
2,2’-dichlorobiphenyl	4	50	25	70-130	75-125	60-135	
4,4’-dichlorobiphenyl	15	50	25	70-130	75-125	60-135	
2,2’,6-trichlorobiphenyl	19	50	25	70-130	75-125	60-135	
3,4,4’-trichlorobiphenyl	37	50	25	70-130	75-125	60-135	
2,2’,6,6’-tetrachlorobiphenyl	54	50	25	70-130	75-125	60-135	
3,3’,4,4’-tetrachlorobiphenyl	77	50	25	70-130	75-125	60-135	
3,4,4’,5-tetrachlorobiphenyl	81	50	25	70-130	75-125	60-135	
2,2’4,6,6’-pentachlorobiphenyl	104	50	25	70-130	75-125	60-135	
2,3,3’,4,4’-pentachlorobiphenyl	105	50	25	70-130	75-125	60-135	
2,3,4,4’,5-pentachlorobiphenyl	114	50	25	70-130	75-125	60-135	
2,3’,4,4’,5-pentachlorobiphenyl	118	50	25	70-130	75-125	60-135	
2’,3’,4,4’,5-pentachlorobiphenyl	123	50	25	70-130	75-125	60-135	
3,3’,4,4’,5-pentachlorobiphenyl	126	50	25	70-130	75-125	60-135	
2,2’,4,4’,6,6’-hexachlorobiphenyl	155	50	25	70-130	75-125	60-135	
2,3,3’,4,4’,5-hexachlorobiphenyl	156	50	25	70-130	75-125	60-135	
2,3’,4,4’,5,5’-hexachlorobiphenyl	157	50	25	70-130	75-125	60-135	
2,3,3’,4,4’,5’-hexachlorobiphenyl	167	50	25	70-130	75-125	60-135	
3,3’,4,4’,5,5’-hexachlorobiphenyl	169	50	25	70-130	75-125	60-135	
2,2’,3,4’5,6,6’-heptachlorobiphenyl	188	50	25	70-130	75-125	60-135	
2,3,3’,4,4’,5,5’-heptachlorobiphenyl	189	50	25	70-130	75-125	60-135	
2,2’,3,3',5,5',6,6’-octachlorobiphenyl	202	50	25	70-130	75-125	60-135	
2,3,3',4,4',5,5',6-octachlorobiphenyl	205	50	25	70-130	75-125	60-135	
2,2’,3,3’,4,4’,5,5’,6-nonachlorobiphenyl	206	50	25	70-130	75-125	60-135	
2,2’,3,3’,4,5,5’,6,6’-nonachlorobiphenyl	208	50	25	70-130	75-125	60-135	
2,2’,3,3’,4,4’,5,5’,6,6’-decachlorobiphenyl	209	50	25	70-130	75-125	60-135	
Internal Standards							
$^{13}C_{12}$-2-chlorobiphenyl	1L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-4-chlorobiphenyl	3L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-2,2’-dichlorobiphenyl	4L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-4,4’-dichlorobiphenyl	15L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-2,2’,6-trichlorobiphenyl	19L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-3,4,4’-trichlorobiphenyl	37L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-2,2’,6,6’-tetrachlorobiphenyl	54L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-3,3’,4,4’-tetrachlorobiphenyl	77L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-3,4,4’,5-tetrachlorobiphenyl	81L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,4,6,6’-pentachlorobiphenyl	104L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3’,4,4’-pentachlorobiphenyl	105L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$2,3,4,4’,5-pentachlorobiphenyl -	114L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3’,4,4’,5-pentachlorobiphenyl	118L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2’,3,4,4’,5-pentachlorobiphenyl	123L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-3,3’,4,4’,5-pentachlorobiphenyl	126L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,4,4’,6,6’-hexachlorobiphenyl	155L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3’,4,4’,5-hexachlorobiphenyl	156L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3’,4,4’,5’-hexachlorobiphenyl	157L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3’,4,4’,5,5’-hexachlorobiphenyl	167L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-3,3’,4,4’,5,5’-hexachlorobiphenyl	169L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,3,4’,5,6,6’-heptachlorobiphenyl	188L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3’,4,4’,5,5’-heptachlorobiphenyl	189L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,3,3’,5,5’,6,6’-octachlorobiphenyl	202L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3’,4,4’,5,5’,6-octachlorobiphenyl	205L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,3,3’,4,4’,5,5’,6-	206L	100	50	45-135	50-145	40-145	10-145

			IPR (IDOC)				
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL)[1]**	**%RSD**	**Mean %R**	**VER %R**	**OPR (LCS) %R**	**Labeled Compound %R in Samples**
nonachlorobiphenyl							
$^{13}C_{12}$-2,2’,3,3’,4,5,5’,6,6’-nonachlorobiphenyl	208L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,3,3’,4,4’,5,5’,6,6’-decachlorobiphenyl	209L	100	50	45-135	50-145	40-145	10-145
Cleanup Standards							
$^{13}C_{12}$-2,4,4’-trichlorobiphenyl	28L	50	70	20-135	65-135	15-145	5-145
$^{13}C_{12}$-2,3,3’,5,5’-pentachlorobiphenyl	111L	50	50	45-135	75-125	40-145	10-145
$^{13}C_{12}$-2,2’,3,3’,5,5’,6-heptachlorobiphenyl	178L	50	50	45-135	75-125	40-145	10-145
Sampling Surrogate Standards							
$^{13}C_{12}$-2,4'-dichlorobiphenyl	8L	50			50-150	50-150	50-150
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	79L	50			50-150	50-150	50-150
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	95L	50			50-150	50-150	50-150
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	153L	50			50-150	50-150	50-150

[1] - Test concentrations are based on ng/mL in the sample extract or standard solution.

Table 11 - Retention Times of Isomers on the SPB-Octyl Column for the PCB Standard Mixes[1]

PCB Congener Mix 1 (AccustandardM-1668-1)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
1	2	4	78	6	161
2	10	4	81	6	153
2	9	5	96	6	130
2	6	5	103	6	129
2	8	5	95	6	166
2	14	5	88	6	159
2	11	5	89	6	167
3	30	5	92	6	156
3	27	5	113	7	179
3	32	5	83	7	176
3	34	5	119	7	178
3	26	5	87	7	175
3	31	5	85	7	183
3	33	5	82	7	177
3	36	5	120	7	171
3	38	5	124	7	172
3	35	5	106	7	191
4	50	5	122	7	170
4	45	5	105	7	190
4	52	5	127	8	200/201
4	49	6	152	8	204
4	75	6	136	8	199/200
4	41	6	148	8	198
4	72	6	151	8	196
4	57	6	144	8	195
4	63	6	143	8	194
4	66	6	142	9	207
4	79	6	133		

PCB Congener Mix 2 (Accustandard M-1668-2)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
2	7	4	55	6	139
2	5	4	60	6	132
2	12	5	94	6	165
3	18	5	100	6	168
3	24	5	91	6	137
3	23	5	121	6	160
3	28	5	90	6	128
3	22	5	99	6	162
3	39	5	109/108	6	157
4	53	5	117	7	184
4	51	5	111	7	186
4	73	5	108/107	7	187
4	48	5	118	7	185
4	62	5	114	7	181
4	71	6	150	7	192
4	68	6	145	8	197
4	58	6	135	8	201/199
4	61	6	149	8	203

Table 11 - Retention Times of Isomers on the SPB-Octyl Column for the PCB Standard Mixes[1] (Continued)

PCB Congener Mix 3 (Accustandard M-1668-3)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
2	13	4	80	6	140
3	17	5	93	6	146
3	29	5	84	6	141
3	20	5	101	6	164
4	46	5	112	6	158
4	65	5	86	7	182
4	59	5	116	7	174
4	40	5	107/109	7	173
4	67	6	154	7	193
4	76	6	147		

PCB Congener Mix 4 (Accustandard M-1668-4)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
3	25	4	64	5	123
3	21	4	70	6	134
4	69	5	102	6	131
4	47	5	97	6	163
4	42	5	115	7	180

PCB Congener Mix 5 (AccustandardM-1668-5)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
1	1	4	74	6	169
1	3	4	56	7	188
2	4	4	77	7	189
2	15	5	104	8	202
3	19	5	98	8	205
3	16	5	125	9	208
3	37	5	110	9	206
4	54	5	126	10	209
4	43	6	155		
4	44	6	138		

Notes:

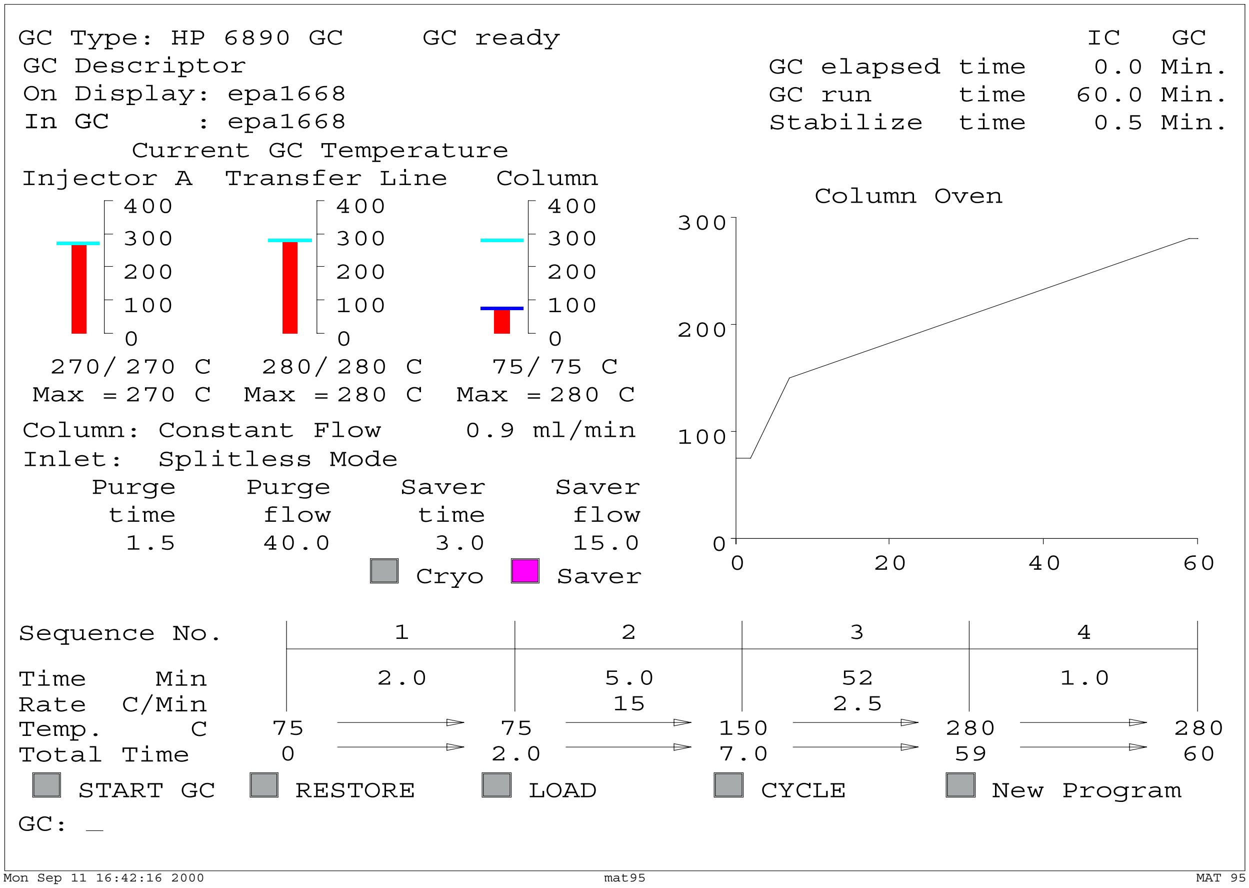
[1] Each congener mix is analyzed in triplicate to establish the retention times of the PCB isomers in the absence of co-eluting isomers. The elution order listed here is used to assign peak identifications in the separate mixture analysis. The average retention time established in the analysis of the separate mixtures is then used to establish relative retention times. (See sections 10.2.3)

[2] BZ/IUPAC Number, if different.

Table 12: Assignment of Sample Preparation Protocols

1668 Protocol	Protocol Name	Sample Amount Extracted (g)	Fraction of Extract Cleaned	IS Added (mL)	Cleanup Std Added (mL)	Recovery Std Added (µL)	Extract Delivery Volume (µL)	Prep Split Factor	Prep Dilution Factor	QuantIMS Final Volume
P1	**Clean**	**10**	**1/2**	**1**	**0.5 (added aftersplit)**	**50**	**50**	**2**	**1**	**100**
P2	**Low**	**2**	**1**	**1**	**1**	**100**	**100**	**1**	**1**	**100**
P3	**Medium**	**1.25**	**1/2**	**2**	**1 (added after split)**	**100**	**250**	**2**	**2.5**	**200**
P4	**High**	**1**	**1/4**	**4**	**1 (added after split)**	**100**	**500**	**4**	**5**	**400**

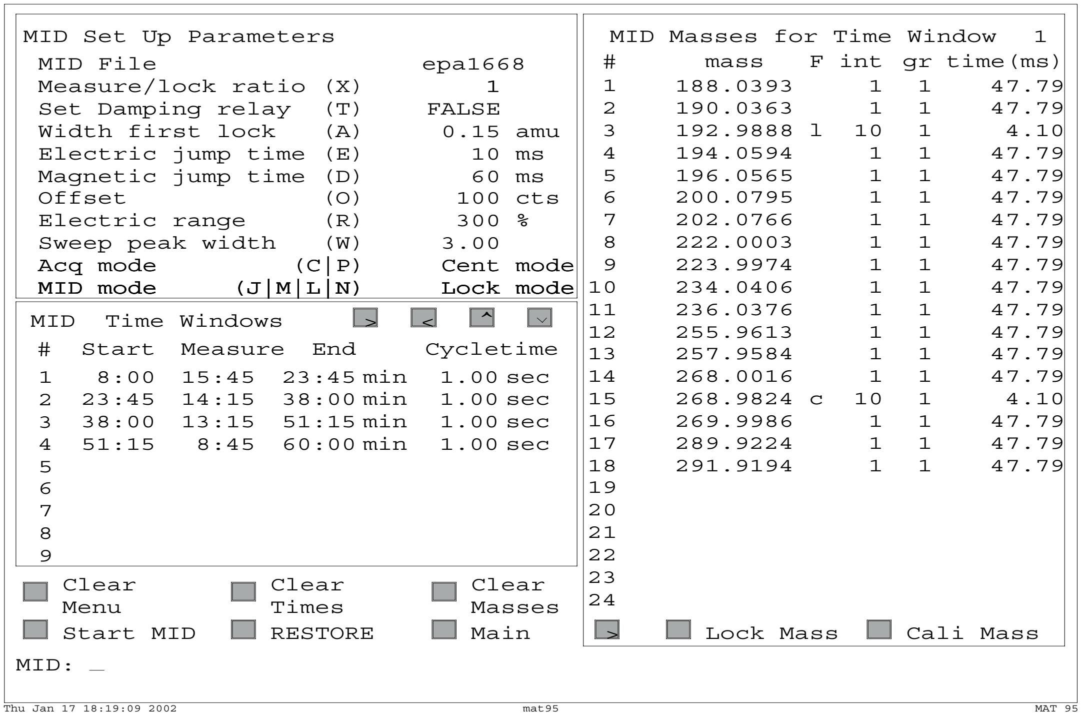
Figure 1 - Recommended GC Operating Conditions



```
GC Type: HP 6890 GC     GC ready                                IC    GC
GC Descriptor                              GC elapsed time     0.0 Min.
On Display: epa1668                        GC run     time    60.0 Min.
In GC      : epa1668                       Stabilize  time     0.5 Min.
       Current GC Temperature
Injector A  Transfer Line   Column
      400           400          400                 Column Oven
      300           300          300
      200           200          200
      100           100          100
      0             0            0
 270/270 C     280/280 C     75/75 C
Max =270 C    Max =280 C    Max =280 C
Column: Constant Flow       0.9 ml/min
Inlet:  Splitless Mode
     Purge      Purge      Saver      Saver
      time       flow       time       flow
       1.5       40.0        3.0       15.0
                         ☐ Cryo     ■ Saver

Sequence No.          1            2            3            4
Time     Min         2.0          5.0          52          1.0
Rate   C/Min                       15          2.5
Temp.      C   75 ------->  75 -------> 150 -------> 280 -------> 280
Total Time     0  ------->  2.0 ------> 7.0 ------>  59  ------->  60
☐ START GC   ☐ RESTORE    ☐ LOAD    ☐ CYCLE    ☐ New Program
GC: _
Mon Sep 11 16:42:16 2000            mat95                      MAT 95
```

Figure 2 - Recommended MID Descriptors



```
MID Set Up Parameters
 MID File                       epa1668
 Measure/lock ratio  (X)          1
 Set Damping relay   (T)      FALSE
 Width first lock    (A)       0.15 amu
 Electric jump time  (E)         10 ms
 Magnetic jump time  (D)         60 ms
 Offset              (O)        100 cts
 Electric range      (R)        300 %
 Sweep peak width    (W)       3.00
 Acq mode           (C|P)      Cent mode
 MID mode       (J|M|L|N)      Lock mode

 MID  Time Windows      >   <   ^   v
 #  Start  Measure  End      Cycletime
 1   8:00  15:45  23:45 min  1.00 sec
 2  23:45  14:15  38:00 min  1.00 sec
 3  38:00  13:15  51:15 min  1.00 sec
 4  51:15   8:45  60:00 min  1.00 sec
 5
 6
 7
 8
 9

☐ Clear       ☐ Clear     ☐ Clear
  Menu          Times       Masses
☐ Start MID   ☐ RESTORE   ☐ Main
MID: _
```

```
 MID Masses for Time Window  1
 #     mass    F int  gr time(ms)
 1    188.0393     1   1    47.79
 2    190.0363     1   1    47.79
 3    192.9888 l  10   1     4.10
 4    194.0594     1   1    47.79
 5    196.0565     1   1    47.79
 6    200.0795     1   1    47.79
 7    202.0766     1   1    47.79
 8    222.0003     1   1    47.79
 9    223.9974     1   1    47.79
10    234.0406     1   1    47.79
11    236.0376     1   1    47.79
12    255.9613     1   1    47.79
13    257.9584     1   1    47.79
14    268.0016     1   1    47.79
15    268.9824 c  10   1     4.10
16    269.9986     1   1    47.79
17    289.9224     1   1    47.79
18    291.9194     1   1    47.79
19
20
21
22
23
24
 >    ☐ Lock Mass   ☐ Cali Mass
```

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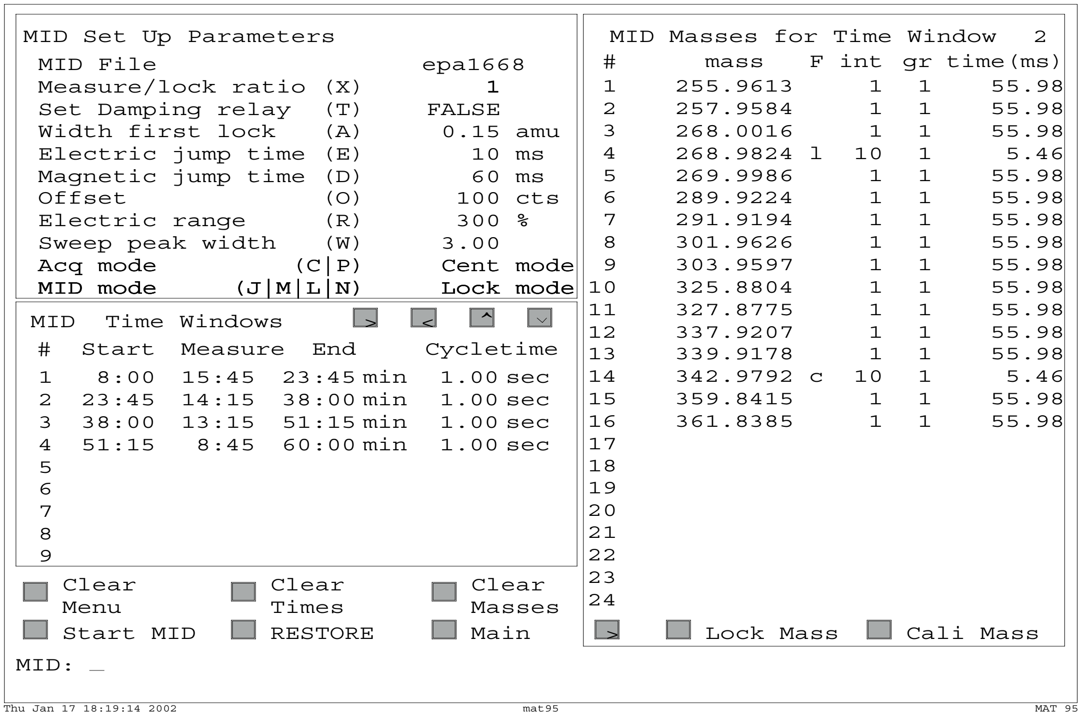

```
MID Set Up Parameters
 MID File                       epa1668
 Measure/lock ratio  (X)          1
 Set Damping relay   (T)      FALSE
 Width first lock    (A)       0.15 amu
 Electric jump time  (E)         10 ms
 Magnetic jump time  (D)         60 ms
 Offset              (O)        100 cts
 Electric range      (R)        300 %
 Sweep peak width    (W)       3.00
 Acq mode           (C|P)      Cent mode
 MID mode       (J|M|L|N)      Lock mode

 MID  Time Windows      >   <   ^   v
 #  Start  Measure  End      Cycletime
 1   8:00  15:45  23:45 min  1.00 sec
 2  23:45  14:15  38:00 min  1.00 sec
 3  38:00  13:15  51:15 min  1.00 sec
 4  51:15   8:45  60:00 min  1.00 sec
 5
 6
 7
 8
 9

☐ Clear       ☐ Clear     ☐ Clear
  Menu          Times       Masses
☐ Start MID   ☐ RESTORE   ☐ Main
MID: _
```

```
 MID Masses for Time Window  2
 #     mass    F int  gr time(ms)
 1    255.9613     1   1    55.98
 2    257.9584     1   1    55.98
 3    268.0016     1   1    55.98
 4    268.9824 l  10   1     5.46
 5    269.9986     1   1    55.98
 6    289.9224     1   1    55.98
 7    291.9194     1   1    55.98
 8    301.9626     1   1    55.98
 9    303.9597     1   1    55.98
10    325.8804     1   1    55.98
11    327.8775     1   1    55.98
12    337.9207     1   1    55.98
13    339.9178     1   1    55.98
14    342.9792 c  10   1     5.46
15    359.8415     1   1    55.98
16    361.8385     1   1    55.98
17
18
19
20
21
22
23
24
 >    ☐ Lock Mass   ☐ Cali Mass
```

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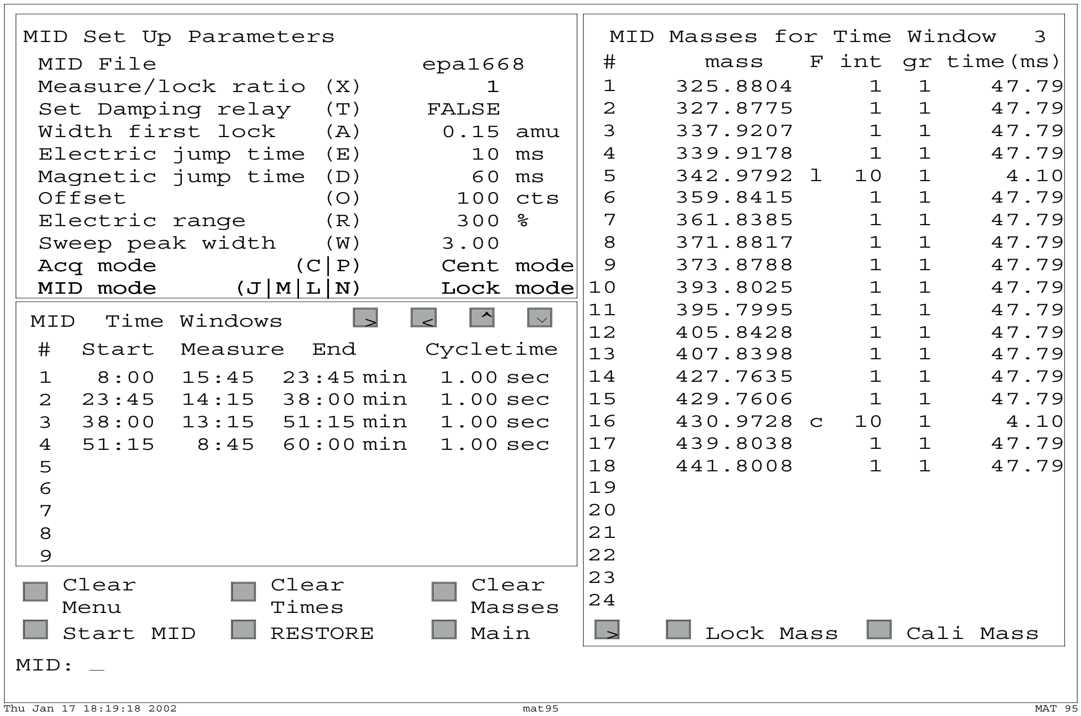
Figure 2 - Recommended MID Descriptors (Continued)

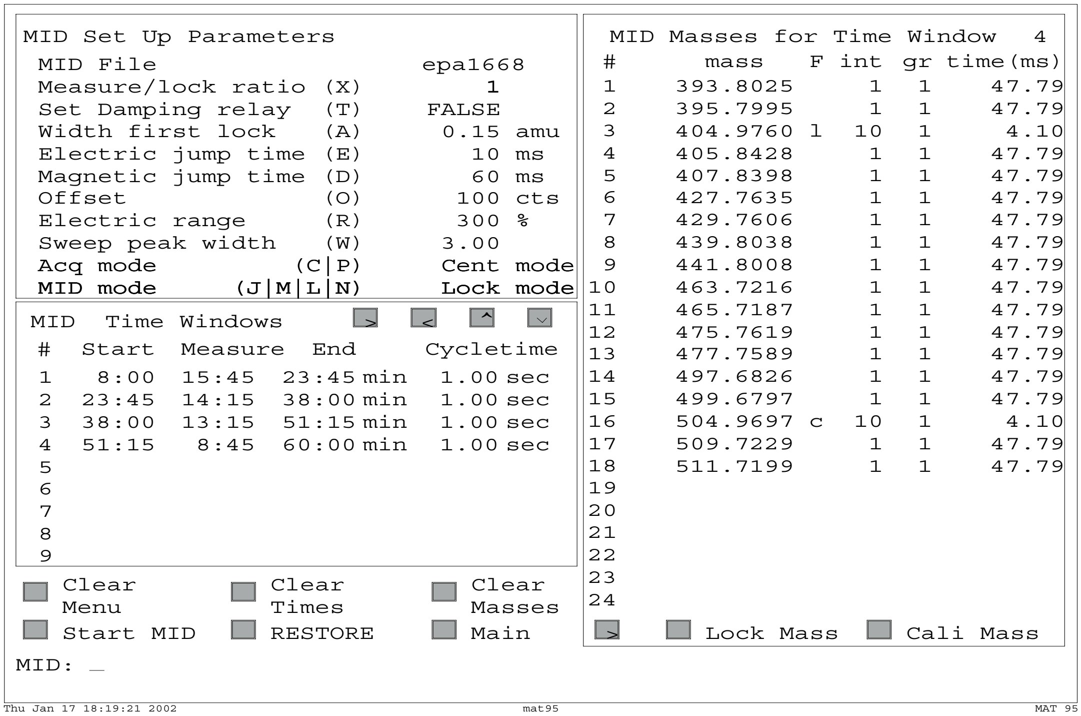


```
MID Set Up Parameters
 MID File                         epa1668
 Measure/lock ratio  (X)             1
 Set Damping relay   (T)         FALSE
 Width first lock    (A)          0.15 amu
 Electric jump time  (E)            10 ms
 Magnetic jump time  (D)            60 ms
 Offset              (O)           100 cts
 Electric range      (R)           300 %
 Sweep peak width    (W)          3.00
 Acq mode          (C|P)          Cent mode
 MID mode        (J|M|L|N)        Lock mode

MID  Time Windows
 #  Start  Measure  End      Cycletime
 1   8:00  15:45   23:45 min  1.00 sec
 2  23:45  14:15   38:00 min  1.00 sec
 3  38:00  13:15   51:15 min  1.00 sec
 4  51:15   8:45   60:00 min  1.00 sec
 5
 6
 7
 8
 9

MID:  _
```

```
 MID Masses for Time Window   3
 #      mass    F  int  gr  time(ms)
 1    325.8804       1   1     47.79
 2    327.8775       1   1     47.79
 3    337.9207       1   1     47.79
 4    339.9178       1   1     47.79
 5    342.9792  l   10   1      4.10
 6    359.8415       1   1     47.79
 7    361.8385       1   1     47.79
 8    371.8817       1   1     47.79
 9    373.8788       1   1     47.79
10    393.8025       1   1     47.79
11    395.7995       1   1     47.79
12    405.8428       1   1     47.79
13    407.8398       1   1     47.79
14    427.7635       1   1     47.79
15    429.7606       1   1     47.79
16    430.9728  c   10   1      4.10
17    439.8038       1   1     47.79
18    441.8008       1   1     47.79
19
20
21
22
23
24
```



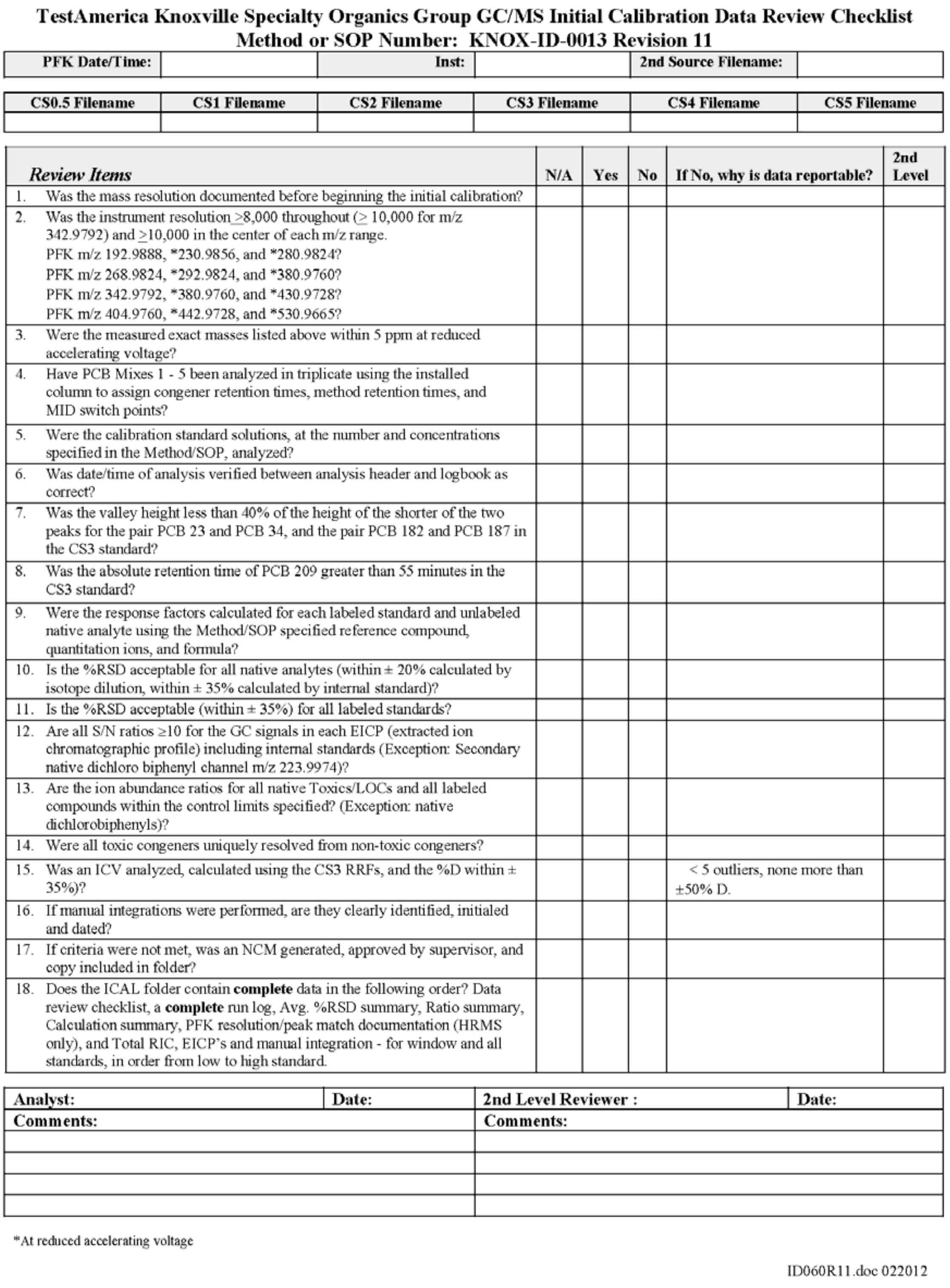
```
MID Set Up Parameters
 MID File                         epa1668
 Measure/lock ratio  (X)             1
 Set Damping relay   (T)         FALSE
 Width first lock    (A)          0.15 amu
 Electric jump time  (E)            10 ms
 Magnetic jump time  (D)            60 ms
 Offset              (O)           100 cts
 Electric range      (R)           300 %
 Sweep peak width    (W)          3.00
 Acq mode          (C|P)          Cent mode
 MID mode        (J|M|L|N)        Lock mode

MID  Time Windows
 #  Start  Measure  End      Cycletime
 1   8:00  15:45   23:45 min  1.00 sec
 2  23:45  14:15   38:00 min  1.00 sec
 3  38:00  13:15   51:15 min  1.00 sec
 4  51:15   8:45   60:00 min  1.00 sec
 5
 6
 7
 8
 9

MID:  _
```

```
 MID Masses for Time Window   4
 #      mass    F  int  gr  time(ms)
 1    393.8025       1   1     47.79
 2    395.7995       1   1     47.79
 3    404.9760  l   10   1      4.10
 4    405.8428       1   1     47.79
 5    407.8398       1   1     47.79
 6    427.7635       1   1     47.79
 7    429.7606       1   1     47.79
 8    439.8038       1   1     47.79
 9    441.8008       1   1     47.79
10    463.7216       1   1     47.79
11    465.7187       1   1     47.79
12    475.7619       1   1     47.79
13    477.7589       1   1     47.79
14    497.6826       1   1     47.79
15    499.6797       1   1     47.79
16    504.9697  c   10   1      4.10
17    509.7229       1   1     47.79
18    511.7199       1   1     47.79
19
20
21
22
23
24
```

Figure 3 - Example Data Review Checklist



TestAmerica Knoxville Specialty Organics Group GC/MS Initial Calibration Data Review Checklist
Method or SOP Number: KNOX-ID-0013 Revision 11

PFK Date/Time:		**Inst:**		**2nd Source Filename:**	

CS0.5 Filename	CS1 Filename	CS2 Filename	CS3 Filename	CS4 Filename	CS5 Filename

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd Level
1. Was the mass resolution documented before beginning the initial calibration?					
2. Was the instrument resolution ≥8,000 throughout (≥ 10,000 for m/z 342.9792) and ≥10,000 in the center of each m/z range. PFK m/z 192.9888, *230.9856, and *280.9824? PFK m/z 268.9824, *292.9824, and *380.9760? PFK m/z 342.9792, *380.9760, and *430.9728? PFK m/z 404.9760, *442.9728, and *530.9665?					
3. Were the measured exact masses listed above within 5 ppm at reduced accelerating voltage?					
4. Have PCB Mixes 1 - 5 been analyzed in triplicate using the installed column to assign congener retention times, method retention times, and MID switch points?					
5. Were the calibration standard solutions, at the number and concentrations specified in the Method/SOP, analyzed?					
6. Was date/time of analysis verified between analysis header and logbook as correct?					
7. Was the valley height less than 40% of the height of the shorter of the two peaks for the pair PCB 23 and PCB 34, and the pair PCB 182 and PCB 187 in the CS3 standard?					
8. Was the absolute retention time of PCB 209 greater than 55 minutes in the CS3 standard?					
9. Were the response factors calculated for each labeled standard and unlabeled native analyte using the Method/SOP specified reference compound, quantitation ions, and formula?					
10. Is the %RSD acceptable for all native analytes (within ± 20% calculated by isotope dilution, within ± 35% calculated by internal standard)?					
11. Is the %RSD acceptable (within ± 35%) for all labeled standards?					
12. Are all S/N ratios ≥10 for the GC signals in each EICP (extracted ion chromatographic profile) including internal standards (Exception: Secondary native dichloro biphenyl channel m/z 223.9974)?					
13. Are the ion abundance ratios for all native Toxics/LOCs and all labeled compounds within the control limits specified? (Exception: native dichlorobiphenyls)?					
14. Were all toxic congeners uniquely resolved from non-toxic congeners?					
15. Was an ICV analyzed, calculated using the CS3 RRFs, and the %D within ± 35%)?				< 5 outliers, none more than ±50% D.	
16. If manual integrations were performed, are they clearly identified, initialed and dated?					
17. If criteria were not met, was an NCM generated, approved by supervisor, and copy included in folder?					
18. Does the ICAL folder contain **complete** data in the following order? Data review checklist, a **complete** run log, Avg. %RSD summary, Ratio summary, Calculation summary, PFK resolution/peak match documentation (HRMS only), and Total RIC, EICP's and manual integration - for window and all standards, in order from low to high standard.					

Analyst:	**Date:**	**2nd Level Reviewer :**	**Date:**
Comments:		**Comments:**	

*At reduced accelerating voltage

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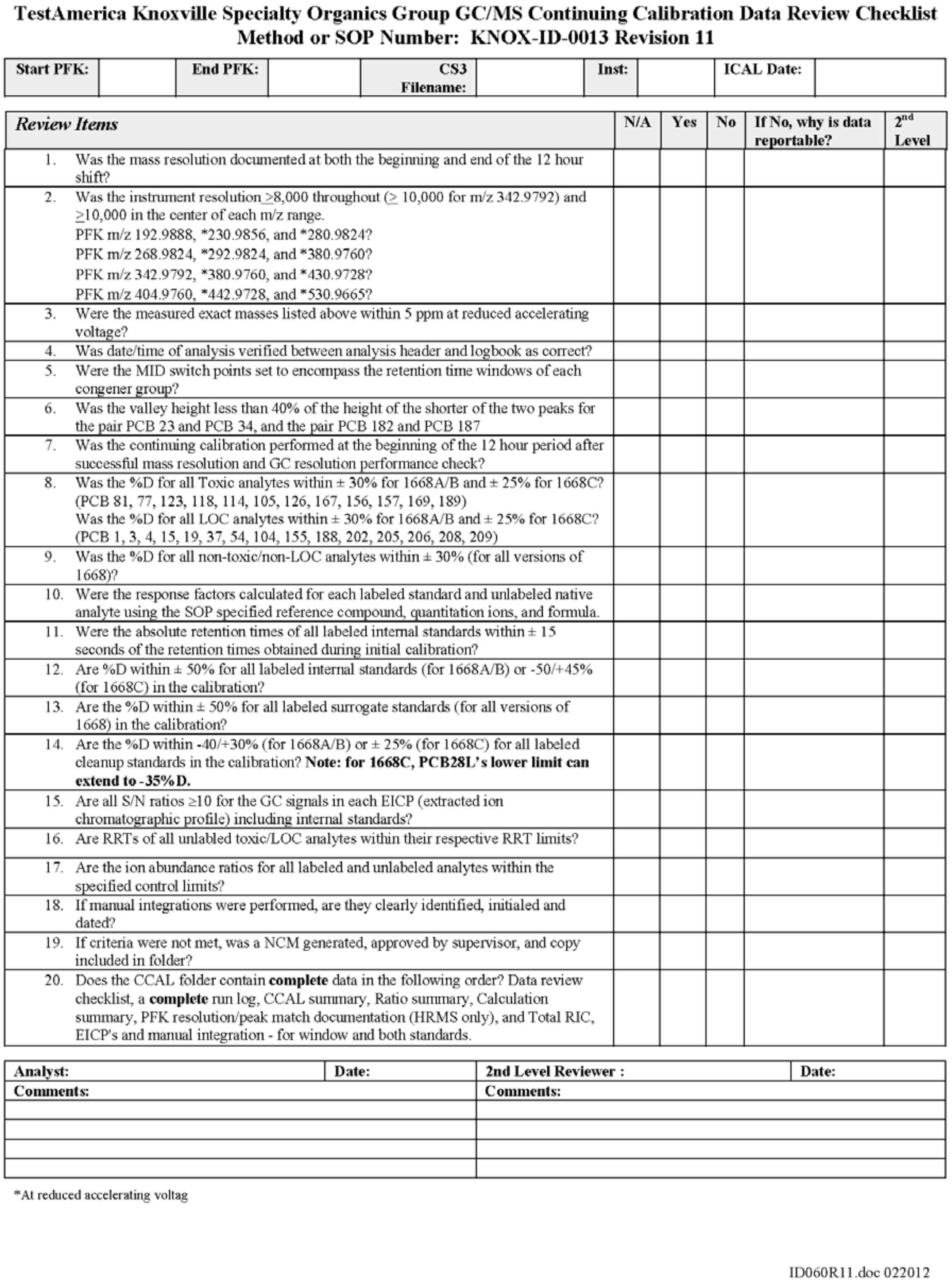
Figure 3- Example Data Review Checklist (Continued)



TestAmerica Knoxville Specialty Organics Group GC/MS Continuing Calibration Data Review Checklist
Method or SOP Number: KNOX-ID-0013 Revision 11

Start PFK:		**End PFK:**		**CS3 Filename:**		**Inst:**		**ICAL Date:**	

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd Level
1. Was the mass resolution documented at both the beginning and end of the 12 hour shift?					
2. Was the instrument resolution ≥8,000 throughout (≥ 10,000 for m/z 342.9792) and ≥10,000 in the center of each m/z range. PFK m/z 192.9888, *230.9856, and *280.9824? PFK m/z 268.9824, *292.9824, and *380.9760? PFK m/z 342.9792, *380.9760, and *430.9728? PFK m/z 404.9760, *442.9728, and *530.9665?					
3. Were the measured exact masses listed above within 5 ppm at reduced accelerating voltage?					
4. Was date/time of analysis verified between analysis header and logbook as correct?					
5. Were the MID switch points set to encompass the retention time windows of each congener group?					
6. Was the valley height less than 40% of the height of the shorter of the two peaks for the pair PCB 23 and PCB 34, and the pair PCB 182 and PCB 187					
7. Was the continuing calibration performed at the beginning of the 12 hour period after successful mass resolution and GC resolution performance check?					
8. Was the %D for all Toxic analytes within ± 30% for 1668A/B and ± 25% for 1668C? (PCB 81, 77, 123, 118, 114, 105, 126, 167, 156, 157, 169, 189) Was the %D for all LOC analytes within ± 30% for 1668A/B and ± 25% for 1668C? (PCB 1, 3, 4, 15, 19, 37, 54, 104, 155, 188, 202, 205, 206, 208, 209)					
9. Was the %D for all non-toxic/non-LOC analytes within ± 30% (for all versions of 1668)?					
10. Were the response factors calculated for each labeled standard and unlabeled native analyte using the SOP specified reference compound, quantitation ions, and formula.					
11. Were the absolute retention times of all labeled internal standards within ± 15 seconds of the retention times obtained during initial calibration?					
12. Are %D within ± 50% for all labeled internal standards (for 1668A/B) or -50/+45% (for 1668C) in the calibration?					
13. Are the %D within ± 50% for all labeled surrogate standards (for all versions of 1668) in the calibration?					
14. Are the %D within -40/+30% (for 1668A/B) or ± 25% (for 1668C) for all labeled cleanup standards in the calibration? **Note: for 1668C, PCB28L's lower limit can extend to -35%D.**					
15. Are all S/N ratios ≥10 for the GC signals in each EICP (extracted ion chromatographic profile) including internal standards?					
16. Are RRTs of all unlabled toxic/LOC analytes within their respective RRT limits?					
17. Are the ion abundance ratios for all labeled and unlabeled analytes within the specified control limits?					
18. If manual integrations were performed, are they clearly identified, initialed and dated?					
19. If criteria were not met, was a NCM generated, approved by supervisor, and copy included in folder?					
20. Does the CCAL folder contain **complete** data in the following order? Data review checklist, a **complete** run log, CCAL summary, Ratio summary, Calculation summary, PFK resolution/peak match documentation (HRMS only), and Total RIC, EICP's and manual integration - for window and both standards.					

Analyst:	**Date:**	**2nd Level Reviewer :**	**Date:**
Comments:		**Comments:**	

*At reduced accelerating voltag

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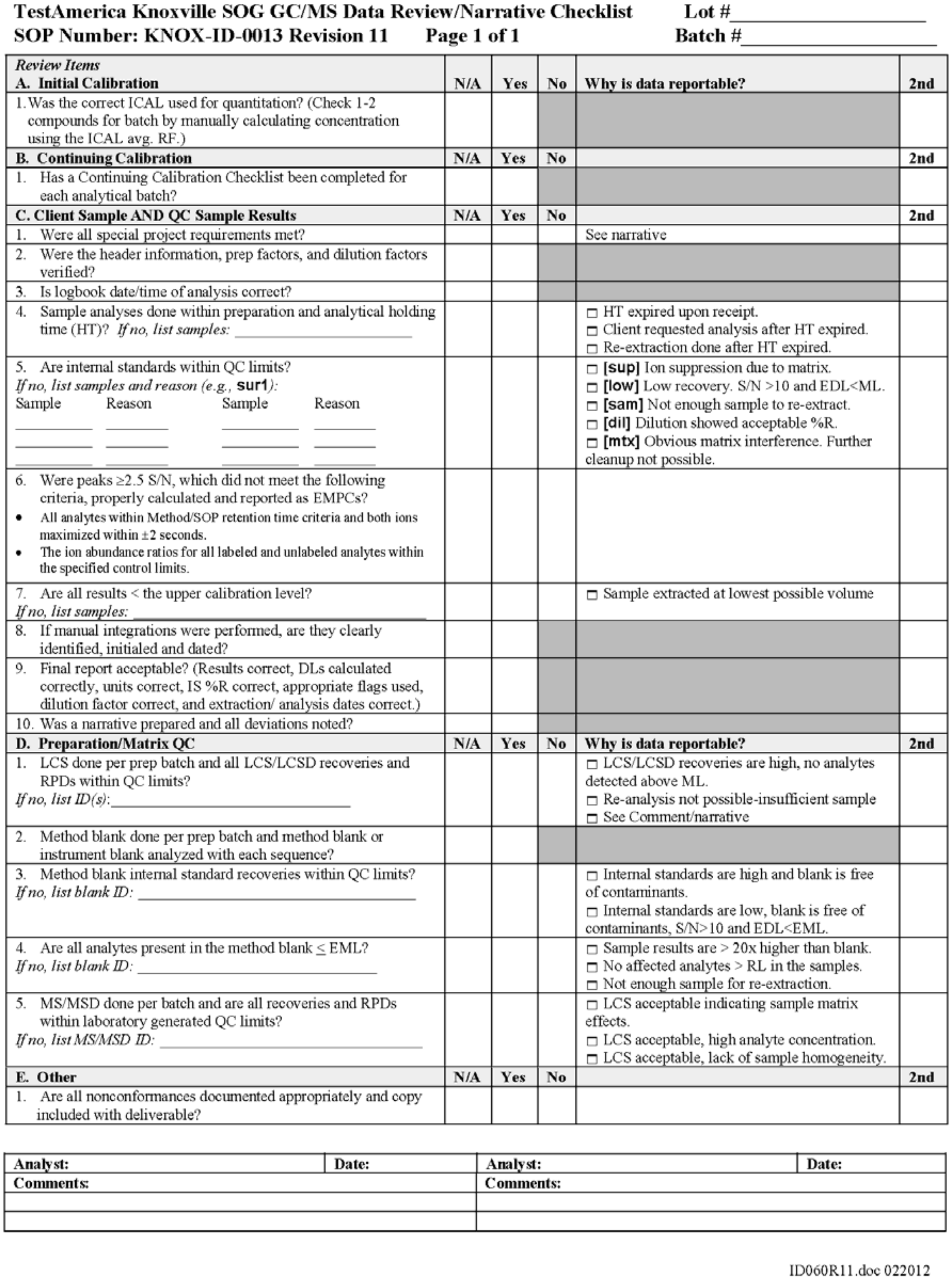
Figure 3 - Example Data Review Checklist (Continued)



TestAmerica Knoxville SOG GC/MS Data Review/Narrative Checklist **Lot #**___________________
SOP Number: KNOX-ID-0013 Revision 11 **Page 1 of 1** **Batch #**___________________

Review Items **A. Initial Calibration**	**N/A**	**Yes**	**No**	**Why is data reportable?**	**2nd**
1. Was the correct ICAL used for quantitation? (Check 1-2 compounds for batch by manually calculating concentration using the ICAL avg. RF.)					
B. Continuing Calibration	**N/A**	**Yes**	**No**		**2nd**
1. Has a Continuing Calibration Checklist been completed for each analytical batch?					
C. Client Sample AND QC Sample Results	**N/A**	**Yes**	**No**		**2nd**
1. Were all special project requirements met?				See narrative	
2. Were the header information, prep factors, and dilution factors verified?					
3. Is logbook date/time of analysis correct?					
4. Sample analyses done within preparation and analytical holding time (HT)? *If no, list samples:* ______________________				☐ HT expired upon receipt. ☐ Client requested analysis after HT expired. ☐ Re-extraction done after HT expired.	
5. Are internal standards within QC limits? *If no, list samples and reason (e.g.,* **sur1***):* Sample Reason Sample Reason _________ ________ _________ ________ _________ ________ _________ ________ _________ ________ _________ ________				☐ **[sup]** Ion suppression due to matrix. ☐ **[low]** Low recovery. S/N >10 and EDL<ML. ☐ **[sam]** Not enough sample to re-extract. ☐ **[dil]** Dilution showed acceptable %R. ☐ **[mtx]** Obvious matrix interference. Further cleanup not possible.	
6. Were peaks ≥2.5 S/N, which did not meet the following criteria, properly calculated and reported as EMPCs? • All analytes within Method/SOP retention time criteria and both ions maximized within ±2 seconds. • The ion abundance ratios for all labeled and unlabeled analytes within the specified control limits.					
7. Are all results < the upper calibration level? *If no, list samples:* ________________________________				☐ Sample extracted at lowest possible volume	
8. If manual integrations were performed, are they clearly identified, initialed and dated?					
9. Final report acceptable? (Results correct, DLs calculated correctly, units correct, IS %R correct, appropriate flags used, dilution factor correct, and extraction/ analysis dates correct.)					
10. Was a narrative prepared and all deviations noted?					
D. Preparation/Matrix QC	**N/A**	**Yes**	**No**	**Why is data reportable?**	**2nd**
1. LCS done per prep batch and all LCS/LCSD recoveries and RPDs within QC limits? *If no, list ID(s)*:____________________________				☐ LCS/LCSD recoveries are high, no analytes detected above ML. ☐ Re-analysis not possible-insufficient sample ☐ See Comment/narrative	
2. Method blank done per prep batch and method blank or instrument blank analyzed with each sequence?					
3. Method blank internal standard recoveries within QC limits? *If no, list blank ID:* ________________________________				☐ Internal standards are high and blank is free of contaminants. ☐ Internal standards are low, blank is free of contaminants, S/N>10 and EDL<EML.	
4. Are all analytes present in the method blank ≤ EML? *If no, list blank ID:* ___________________________				☐ Sample results are > 20x higher than blank. ☐ No affected analytes > RL in the samples. ☐ Not enough sample for re-extraction.	
5. MS/MSD done per batch and are all recoveries and RPDs within laboratory generated QC limits? *If no, list MS/MSD ID:* _____________________________				☐ LCS acceptable indicating sample matrix effects. ☐ LCS acceptable, high analyte concentration. ☐ LCS acceptable, lack of sample homogeneity.	
E. Other	**N/A**	**Yes**	**No**		**2nd**
1. Are all nonconformances documented appropriately and copy included with deliverable?					

Analyst:	**Date:**	**Analyst:**	**Date:**
Comments:		**Comments:**	

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TESTAMERICA KNOXVILLE
STANDARD OPERATING PROCEDURE

TITLE: GPC Cleanup

(SUPERSEDES: KNOX-OP-0022, Rev. 2)

Prepared By: Michael S. Patty

Reviewed By: 1/29/14
Technical Specialist

Approved By: Kevin McGee 1/29/14
Quality Assurance Manager

Approved By: 1-29-14
Environmental, Health and Safety Coordinator

Approved By: 01/29/14
Laboratory Director

Copyright Information:

1. Scope and Application

1.1 The purpose of this procedure is to describe the GPC (gel permeation chromatography) cleanup technique used to prepare sample extracts for analysis by Gas Chromatography (GC) and Gas Chromatography/Mass Spectrometry (GC/MS) and HRGC/HRMS. GPC is capable of separating high molecular weight material from the sample analytes, and so is particularly useful if tissue or vegetable matter is part of the sample, and for many soil and sediment samples.

1.2 GPC is recommended for the elimination of the following type of materials from sample extracts: Lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular weight compounds. GPC is appropriate for both polar and non-polar analytes, therefore, it can be effectively used to cleanup extracts containing a broad range of analytes.

1.3 This procedure applies to sample extracts that will be analyzed for PCBs by GC-ECD, semivolatile organics by GC/MS, PAHs by GC/MS, as well as PCB congeners and dioxins/furans by HRMS.

1.4 Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a GC or the front of the GC column. This residue will ultimately reduce the chromatographic separation efficiency or column capacity because of adsorption of the target analytes on the active sites. Pentachlorophenol is especially susceptible to this problem. GPC, operating on the principal of size exclusion, will not usually remove interference peaks that appear in the chromatogram since the molecular size of these compounds is relative similar to the target analytes. Separation cleanup techniques, based on other molecular characteristics (i.e., polarity), may be used to eliminate this type of interference.

1.5 This procedure is based on SW-846 Method 3640A, “Gel-Permeation Cleanup”.

2. Summary of Method

2.1 The column is packed with the required amount of pre-swelled absorbent, and is flushed with solvent for an extended period. The column is calibrated and checked, then loaded with the sample extract to be cleaned up. Elution is effected with a suitable solvent (i.e., methylene chloride) and the product is then concentrated.

3. Definitions

3.1 Definitions of terms used in this SOP may be found in the glossary of the TestAmerica Knoxville Quality Assurance Manual (QAM).

4. Interferences

4.1 A reagent blank should be analyzed for the compound of interest prior to the use of this method. The level of interferences must be below the reporting limits of the analytes of interest before this method is performed on actual samples.

5. Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

5.2 Primary Material Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. 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 the method can be found in the reagents and materials section. 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.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Methanol	Flammable, Poison, Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure can include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and can cause skin to become dry and cracked. Skin absorption can occur; symptoms can parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3 Chemicals that have been classified as carcinogens or potential carcinogens under OSHA include methylene chloride, PCBs, semivolatile organics, PAHs (i.e., benz(a)anthracene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, dibenz(a,h)anthracene, and indeno(1,2,3-cd)pyrene), 2,3,7,8-TCDD and all other 2,3,7,8- substituted PCDD or PCDF isomers..

Note: The 2,3,7,8-TCDD isomer has been found to be acnegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in positions 2,3,7,8 are known to have toxicities comparable to that of 2,3,7,8-TCDD. The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.

5.4 Exposure to hazardous chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.6 All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported immediately to a laboratory supervisor.

6. Equipment and Supplies

6.1 Gel permeation chromatography system (J2 Scientific Accu Prep or equivalent).

6.2 Bio Beads: (S-X3) -200-400 mesh, 70 gm (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750 or equivalent).

6.3 Chromatographic column: 700 mm x 25 mm ID glass column. Flow is upward.

6.4 Ultraviolet detector: Fixed wavelength (254 nm) and a semi-prep flow-through cell.

6.5 Laboratory data system: GPC Data Systems: AccuPrep 307D 10-24-2005, Firmware version 20.8 and PrepLinc 1.0.0.25

6.6 Syringe: 10 mL with Luerlok fitting.

6.7 Syringe filter assembly, with disposable 25mm, 0.45 um filter discs, Millipore No. LSWP 01300 or equivalent.

6.8 GPC collection tubes, 200mm x 37mm, 200ml capacity or equivalent, available from J2 Scientific.

6.9 12 ml screw top sample vials, 100mm x 15mm.

6.10 16mm black caps, part no. BV016 or equivalent.

6.11 13mm PTFE Discs, part no. BV016T-PK or equivalent.

7. Reagents and Standards

7.1 Methylene chloride, methanol, pesticide quality, or equivalent.

7.2 GPC calibration solution – Restek, Catalog # 32142, containing the following analytes in the concentrations listed below:

Analyte	**Stock Standard Conc. (mg/mL)**	**Prepared Standard Conc. (mg/mL)**
Corn Oil	250	25
Bis (2-ethylhexyl) phthalate	5	0.5
Methoxychlor	1	0.1
Perylene	0.2	0.02
Sulfur	0.8	0.08

7.2.1 Store the calibration solution in an amber glass bottle with a Teflon lined screw-cap at 4^{o}C, and protect from light. (Refrigeration may cause the corn oil to precipitate. Before use, allow the calibration solution to stand at room temperature until the corn oil dissolves.) Replace the prepared calibration standard solution every 6 months, or more frequently if necessary.

7.3 GPC Recovery Check Standards

7.3.1 PCB GPC Recovery Check Standard – A 100µg/L standard mix containing PCB 1, PCB 3 and PCB 209. Prepared using 35 µg/mL individual standards from Accustandard. 1.0 mL of the 100 µg/L standard mix is used to prepare the PCB GPC check standard. This standard uses the manufacturer’s expiration date.

7.3.2 Semivolatiles GPC Recovery Check Standard – BNA LCS Spike (Restek Cat. No’s. 567672, 567673, 567674, 567675, 567676, 567677). This standard contains the BNA MS/LCS analytes at 100 µg/mL in methanol. 1.0 mL is used to prepare the semivolatiles GPC check standard.

7.3.3 SIM PAH GPC Recovery Check Standard – SIM PAH LCS Spike and internal standards (refer to KNOX-ID-0016 for details). 1.0 mL of the 0.250 ug/mL (LCS) and 1.0mL of internal standard is used to prepare the SIM PAH GPC check standard.

8. Sample Collection, Preservation and Storage

8.1 Not applicable.

9. Quality Control

9.1 GPC Calibration Solution – This solution is used each week to verify the GPC is still functioning in an acceptable manner. It also serves to set the appropriate collection and dump times to ensure collection of all applicable analytes. See section 7.2 for analytes and their concentrations in the calibration solution. Refer to section 11.4 for the acceptance criteria.

9.2 GPC Recovery Check Standard – These standards contain representative analytes from the determinative methods and are injected into the GPC after calibration to verify that the appropriate collection and dump periods have been established to ensure collection of all target analytes.

9.3 GPC Blank Check – 10 mL of methylene chloride that is processed through the GPC to ensure that the system is clean. This system blank is run through the GPC right after the GPC Recovery Check Standard. The GPC blank must be processed before the samples and must not contain any of the compounds of interest at a concentration above the reporting limit.

10. Calibration and Standardization

10.1 Refer to sections 11.4 through 11.5 for detailed calibration instructions.

11. Procedure

11.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure, except those specified by project specific instructions, shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist, Project Manager and QA Manager. If contractually required, the client shall be notified.

11.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3 GPC Column Packing Preparation

11.3.1 Weigh out 70 g of Bio Beads into a 400 mL beaker or an appropriate container.

11.3.2 Add approximately 300 mL of methylene chloride and stir gently.

11.3.3 Cover and allow the beads to swell for a minimum of two hours. Maintain enough solvent to sufficiently cover the beads at all times.

11.3.4 Position and tighten the outlet bed support (top) plunger assembly in the tube by inserting the plunger and turning it clockwise until snug. Install the plunger near the column end but no closer than 5 cm (measured from the gel packing to the collar).

11.3.5 Turn the column upside down from its normal position with the open end up. Place the tubing from the top plunger assembly into a waste beaker below the column.

11.3.6 Swirl the bead/solvent slurry to get a homogeneous mixture and pour the mixture into the open end of the column. Transfer as much as possible with one continuous pour in order to minimize bubble formation. Pour enough to fill the column. Wait for the excess solvent to drain out before pouring in the rest. Add additional methylene chloride to transfer the remaining beads and to rinse the beaker or container and the sides of the column. If the top of the gel begins to look dry, add more methylene chloride to re-wet the beads.

11.3.7 Wipe any remaining beads and solvent from the inner walls of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

11.3.8 Push the plunger until it meets the gel, then compress the column bed about 4 cm.

11.3.9 Connect the column inlet to the GPC port and place the column outlet tube into the bottom of the UV detector.

11.3.10 Turn on the UV detector, the autoinjector , GPC pump and autosampler.

11.3.11 On the desktop of the PC, double click the icon to bring up the GPC software. While the software is opening, choose “Use Autoinjector” when prompted for GPC 1.

11.3.12 Start the main pump, make sure the column is in line to start the solvent flow through the column.

11.3.13 Wash the column for at least 1-2 hours. Check the back pressure. Adjust the inlet bed support plunger until approximately 6-12 psi back-pressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

11.3.14 If the GPC column has not been used in a while, check the column for dryness. If dryness has occurred, cycle methylene chloride through the column to re-swell the beads or repack the GPC column.

11.4 Initial Calibration and Resolution Requirements of the GPC System

11.4.1 Before use, the GPC must be calibrated based on monitoring the elution of standards with a UV detector connected to the GPC column.

11.4.2 Prepare a calibration solution by placing 10 mL of the calibration solution in a 12 mL screw cap sample vial and placing it in the sample rack of the GPC.

11.4.3 Inject 10 mL of the calibration solution and retain a UV trace that meets the following requirements (see resolution calculation in section 12.1):

11.4.3.1 Peaks must be observed and should be symmetrical for all compounds in the calibration solution.

11.4.3.2 Corn oil and phthalate peaks must exhibit >85% resolution.

11.4.3.3 Phthalate and methoxychlor peaks must exhibit >85% resolution.

11.4.3.4 Methoxychlor and perylene peaks must exhibit >85% resolution.

11.4.3.5 Perylene and sulfur peaks must not be saturated and must exhibit >85% baseline resolution.

11.4.4 A UV trace that does not meet the criteria above indicates the need for system maintenance and/or the need for a new column.

11.4.5 Determine appropriate collect and dump cycles.

11.4.6 The calibrated GPC program for PCBs (called OP PCB in the methods section) should dump >85% of the phthalate and should collect >95% of the methoxychlor and perylene. Stop collection before sulfur elutes. Use a wash time of 15 minutes.

11.4.7 The calibrated GPC program for semivolatiles (called OP Semi Vol in the methods section) should start collecting column eluate just before the elution of bis(2-ethyl-hexyl)phthalate and after the elution of the corn oil. Stop collection ~1.5 minutes past the apex of the sulfur peak to ensure collection of benzo(ghi)perylene. Use a wash time of 15 minutes.

11.4.8 The retention times for both bis(2-ethylhexyl)phthalate and perylene must not vary more than ±5% between calibrations.

11.5 Procedure for Running a GPC Initial Calibration

11.5.1 Pump solvent through the GPC column for 1-2 hours. Verify that the flow rate is 4.5-5.5 mL/min. Corrective action must be taken if the flow rate is outside this range. The column pressure should be between 6-12 psi.

Note: Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared.

11.5.2 Prepare 2 GPC calibration solutions by placing two separate 10 mL aliquots of GPC calibration solution in 12 mL screw top vials using the 10 mL GPC standards-only syringe.

11.5.3 Place a Teflon septa inside the caps and place the vials with the calibration solution into the sample tray on the autosampler. Load the calibration standards into positions 1 and 2 in the autosampler.

11.6 Load the Sequence into the GPC software.

11.6.1 Start the sequence and save the sequence using the date of the analysis and which GPC was used, e.g., 021204_GPC1_Initial_Calibration.

11.6.2 No collection tubes will be needed for calibration as all of the solvent flowing through the column will be directed to waste.

11.7 Calibration Interpretation and Method Update in AccuPrep or PrepLincSoftware

11.7.1 Start AccuChrome or PrepLinc software and import the data file from calibration run.

11.7.2 If it does not default to the data folder, then choose the down arrow in the “look in” field and point to the data folder. The import of this data may take several minutes.

11.7.3 Once opened then view the chromatogram on screen.

11.7.4 Set the peaks and print the chromatogram. Do this for both “Data Plots” for each calibration run.

11.7.5 Run a comparison report for the runs.

11.7.6 Using the timeline on the report, calculate the new dump and collect times based on information in sections 11.4.6 and 11.4.7. Refer to section 12 concerning dump and collect times.

11.8 Updating Methods in the GPC software

11.8.1 Open the method that needs updating.

11.8.2 Enter the new dump time and collect time with a 15 min wash time.

11.8.3 Save the changes.

11.9 GPC Continuing Calibration Check: Check the calibration of the GPC immediately after the initial calibration and every 7 days while the column is in use. To perform this, follow the instructions in 11.9.1.

11.9.1 Inject one calibration solution, and obtain a UV trace. If the retention times of bis(2-ethyl hexyl)phthalate or perylene have changed by more than ± 5%, use this run as the start of a new initial calibration. Run a comparison report comparing the calibration solution you just ran to one of the calibration solutions run on the previous calibration. If the comparison report passes you may proceed with the recovery check.

Excessive retention time shifts may be caused by poor laboratory temperature control or system leaks, an unstabilized column, or high laboratory temperature causing out-gassing of methylene chloride. Corrective actions may need to be taken to correct the problem. If this calibration check fails, perform an initial calibration after corrective actions have been taken.

11.10 GPC Recovery Check for PCBs

11.10.1 The recovery from the GPC must be verified after the initial calibration and prior to the processing of samples. The PCB GPC recovery check standard is 1.0 mL of the 100 µg/L standard mix (refer to section 7.3.1).

Note: Surrogates are not added to the GPC recovery check standard.

11.10.2 A GPC blank that consists of only methylene chloride (no surrogates) must also be loaded onto the GPC after the check standard.

11.10.3 After creating your sequence, run the check and blank through the GPC using the PCB method, collecting the correct fraction for analysis in a 200 mL collection tube.

11.10.4 After collecting the GPC check fraction and GPC blank, concentrate and solvent exchange to hexane. Adjust the final volume to 1.0 mL and analyze by GC/ECD.

11.10.5 If the recovery of each of the analytes is 60 to 140%, then the analyst may use the column. The GPC blank must not contain any of the compounds of interest at a concentration above the reporting limit. If the above criteria are not met, there may be a need for system maintenance.

11.11 GPC Recovery Check for Semivolatiles

11.11.1 The recovery from the GPC must be verified immediately after the initial calibration and prior to the processing of samples. Dilute 1.0 mL of the semivolatiles GPC recovery check standard (refer to section 7.3.2) to 10 mL in methylene chloride.

Note: Surrogates are not added to the GPC recovery check standard.

11.11.2 Load the GPC check followed by a GPC blank (methylene chloride blank - no surrogates) onto the GPC.

11.11.3 After collecting the GPC check and GPC blank fractions, concentrate to 1.0 mL, and analyze by GC/MS.

11.11.4 Recovery of the analytes should meet historical LCS limits. The blank should not contain any analytes at or above the reporting limit. If these conditions are met, the column may be used for sample analysis. If the above criteria are not met, there may be a need for system maintenance.

11.12 GPC Recovery Check for SIM PAH

11.12.1 The recovery from the GPC must be verified immediately after the initial calibration and prior to the processing of samples. Dilute 1.0 mL of the SIM PAH GPC recovery check standard (refer to section 7.3.3) and 1.0 mL of SIM PAH internal standard to 10 mL in methylene chloride.

11.12.2 Load the GPC check followed by a GPC blank (methylene chloride blank – 1.0mL of SIM PAH internal standard) onto the GPC.

11.12.3 After collecting the GPC check and GPC blank fractions, concentrate to 0.5 mL and analyze by GC/MS.

11.12.4 Recovery of the analytes should meet historical LCS limits. The blank should not contain any analytes at or above the reporting limit. If these conditions are met, the column may be used for sample analysis. If the above criteria are not met, there may be a need for system maintenance.

11.13 Sample Extract Cleanup

11.13.1 Reduce the sample extract volume to approximately 6 to 8 mL and add cleanup standard where applicable.

11.13.2 To perform GPC cleanup of sample extracts, follow the same steps for running a calibration sequence as described in section 11.6.

11.13.3 Add sample to syringe with a 0.45 µm filter. Rinse sample vial with approximately 1 to 2 mL of methylene chloride and add to syringe. Dilute to a final volume of 10 mL. After filtering each extract, the syringe filtering apparatus should be rinsed with methylene chloride. Use a new 0.45 µm filter for each extract.

Note: Other solvents may be used to rinse the syringe, however, methylene chloride must be the last rinse solvent.

11.13.4 Select the correct method for the samples being run. The Semi Volatiles method is used for samples that are being analyzed for GCMS semivolatiles and PAHs. The PCB method is used for samples that are being analyzed for: PCB’s by 8082 and 8082A, PCB’s by HRMS, and Dioxins.

11.13.5 Start the pump and let the flow stabilize for 1-2 hours. The solvent flow rate should be 4.5-5.5 mL/min. The ideal laboratory temperature to prevent out-gassing of the methylene chloride is 22^{o}C. The normal backpressure is 6-12 psi.

11.13.6 In order to prevent overloading of the GPC column, highly viscous sample extracts, as well as extracts which are known to contain >10% lipids, must be diluted prior to cleanup. Any sample extract with > 10% lipids or with a viscosity greater than that of a 1:1 glycerol: water solution (by visual comparison) must be diluted and loaded into multiple sample vials.

11.13.7 Collect each sample in a suitable glass collection tube. Monitor sample volumes collected. The collection tubes used for 1668 solid sample analysis, D/F analysis should be marked with a “LL”, “L”, or “S” to designate it for low level analysis glassware. KNOXID0016 PAH analysis should be marked with a “LL”, “L”, “S”, or regular. 1668 water sample tubes should be marked with a “P”. All collection tubes should be solvent rinsed with acetone, methylene chloride, and hexane before use. Toluene will be added to the glassware rinsing if used for HRMS samples.

Note: Ensure that each collection container is in the appropriate position.

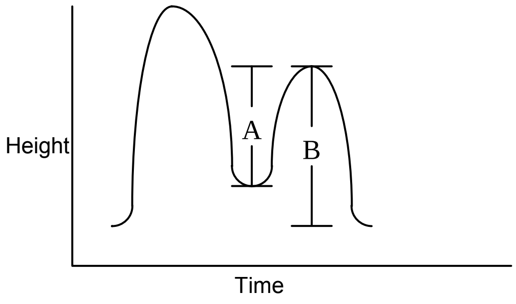
11.13.8 The collection volumes of any samples that were loaded into 2 or more positions must be combined after GPC cleanup.

11.13.9 Concentrate and solvent exchange (as needed) the extracts after GPC cleanup. (Refer to the appropriate SOP for the sample preparation steps needed following GPC cleanup.)

12. Data Analysis and Calculations

12.1 Resolution: To calculate the resolution between two peaks on a chromatograph, divide the depth of the valley between the peaks by the peak height of the smaller peak being resolved and multiply by 100.

Resolution Calculation



$$\% \text{ Resolution } = \frac{A}{B} \times 100$$

Where: A = depth of valley to height of smaller peak

B = peak height of smaller peak

12.2 Dump Time: Mark on the chromatograph the point where collection is to begin. Measure the distance from the injection point. Divide the distance by the chart speed. Alternatively the collect and dump times may be measured by means of an integrator or data system.

$$\text{Dump time (min)} = \frac{\text{Distance (cm) from injection to collection start}}{\text{Chart speed (cm / min)}}$$

12.3 Collection Time

$$\text{Collection time (min)} = \frac{\text{Distance (cm) between collection start and stop}}{\text{Chart speed (cm / min)}}$$

13. Method Performance

13.1 Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009, current revision, for further requirements for performing and documenting initial and on-going demonstrations of capability.

14. Pollution Prevention

14.1 Within the constraints of following the methodology in this SOP, use of organic solvents should be minimized.

15. Waste Management

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention”.

15.2 The following waste streams are produced when this method is carried out.

15.2.1 Solvent waste shall be placed in the flammable waste stream, contained in a steel SAC container type or flammable solvent container.

15.2.2 Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials that might or might not be contaminated/ hazardous must be placed in the incinerable laboratory waste stream, contained in a HDPE satellite accumulation container.

15.2.3 Used bio-beads contaminated with solvents must be placed in the incinerable laboratory waste stream, contained in a HDPE satellite accumulation container.

16. References

16.1 SW-846 Method 3640A, “Gel-Permeation Cleanup”, Revision 1, September 1994.

16.2 TestAmerica Quality Assurance Manual (QAM), current revision.

17. Miscellaneous

17.1 Modifications from Reference Method

17.1.1 The procedure in method 3640A states that only 5 mL of a 10 mL extract is processed through the GPC, resulting in a dilution factor of 2. The entire 10 mL of extract are processed using this procedure.

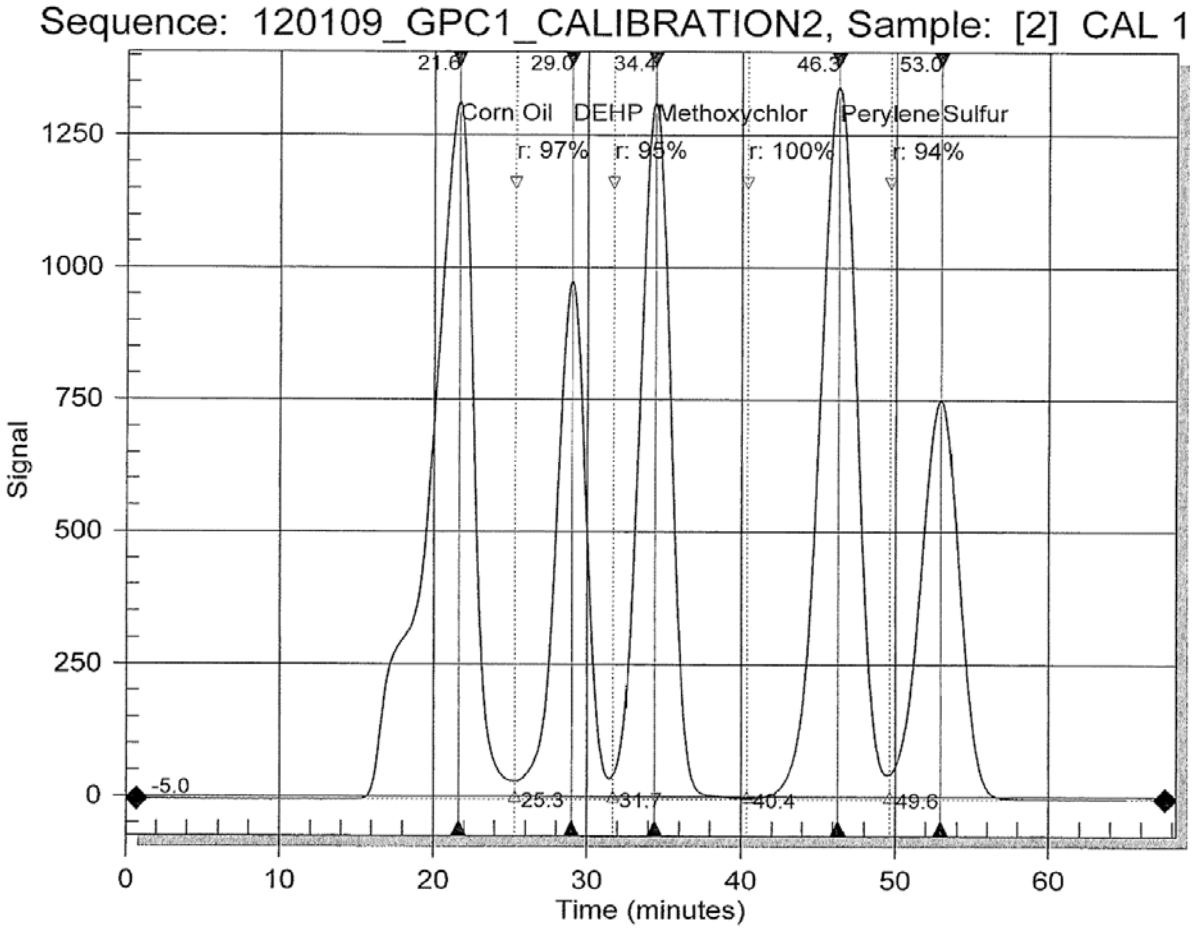
17.1.2 There are minor differences in GPC conditions between this SOP and method 3640A.

17.1.3 The procedure in 3640A states that the resolution between perylene and sulfur shall be at 90%. This procedure uses 85% resolution between perylene and sulfur. A full analyte spike is used for 8270 and SIM PAH analyses to ensure the system is recovering all analytes. Aroclors 1016 and 1260 are used for 8082A analysis.

17.2 Figures

17.2.1 Figure 1 – Example UV Chromatogram of the Calibration Solution

Figure 1 – Example UV Chromatogram of the Calibration Solution



TestAmerica
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Pittsburgh
SOP Change Form
PT-QA-W-023 R0
Effective Date: 01/09/2013

SOP # with Revision PT-GC-005 R4
Title: Polychlorinated Biphenyls (PCBs) and PCB's as Congeners by GC/ECD Method SW846 8082A

Effective Date of Amendment: 2/15/13

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The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the <u>front</u> of the SOP copy.***

1. Briefly explain the reason for the update.
More clearly explain RT Window Studies and how they are to be maintained

2. Category for SOP Change:
☐ Typographical Corrections (Non-Technical) – Retraining Not Required.
☒ Typographical Corrections (Technical- define below) – Retraining is required.
☐ Procedural Changes (Define Below) – Retraining Required.

3. Summary of Procedure Change (circle to indicate if there are attachments to this form: [✓ No] / Yes: # pages attached = ___)
In section 9.20.2.1 added text to clarify when RT studies are to be done and how they are to be maintained
Does this change result in a Method Modification? Yes [✓ No]
If yes, please explain below. Modification section of SOP must be corrected upon full revision.

2/15/13
Approval Signature/Date: Technical

2-15-13
Approval Signature/Date: QA Manager or Designee

Title: Polychlorinated Biphenyls (PCBs) and PCBs as Congeners by GC/ECD

Method: SW-846 8082A

Approvals (Signature/Date):

	10/16/2012		9/26/2012
Sharon Bacha	Date	Steve Jackson	Date
Technical Manager		Health & Safety Manager/Coordinator	
Pamela A. Dudeck	9/25/2012		9/26/2012
Pamela A. Dudeck	Date	Deborah L. Lowe	Date
Quality Assurance Manager (Interim)		Laboratory Director	

Copyright Information:

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure for the determination of concentrations of polychlorinated biphenyls (PCB) as Aroclors and as individual Congeners (see Appendix A) using the methodology prescribed in EPA SW-846 Method 8082A.

1.2 This procedure is applicable to the analysis of aqueous, solid, sediment, biological, and waste/oil samples. When utilized for the analysis of waste/oils, additional cleanup procedures may be required.

1.3 This SOP does not include the procedures for extracting environmental samples. Refer to TestAmerica SOP PT-OP-001 for sample preparation procedures. For specific DoD quality control requirements refer to SOP # PT-QA-025. For DoD V4.2 refer to SOP PT-QA-029.

1.4 Analytes, Matrix(s), and Reporting Limits

1.5 Table 1 lists the specific Aroclors that are determined using this procedure and their associated reporting limits (RLs).

2.0 SUMMARY OF METHOD

2.1 Preparation

2.1.1 Aqueous Samples

PCBs are extracted from a one-liter aqueous sample with methylene chloride using a separatory funnel (SW-846 Method 3510). The extract is evaporated to dryness and exchanged to hexane. The final extract volume is 40 mL for normal PCB analysis and 1.0 ml for low-level PCB analysis. The extraction procedure is detailed in SOP PT-OP-001.

2.1.2 Soil, Sediment and Tissue Samples

PCBs are extracted from soil, sediment and tissue samples using a 50:50 acetone - hexane mixture by the automated Soxtherm (Method 3541). The extract is evaporated to dryness and exchanged to hexane. The final extract volume varies depending on the type of “solid” matrix extracted. The extraction procedure is detailed in SOP PT-OP-001.

2.1.3 Oil or Waste Samples

Oil/waste samples are typically prepared by diluting 1 gram of sample to a final volume of 10 mL with hexane. The extraction procedure is detailed in SOP PT-OP-001.

2.1.4 Wipe Samples

Wipes are typically collected using either filter paper or gauze. These samples can then be extracted using the procedure outlined in SOP PT-OP-001.

2.1.5 Cleanup Procedures

Cleanup options are discussed in Section 4 below. Instructions for performing various cleanup procedures are detailed in SOP PT-OP-001.

2.2 Analysis

Samples are analyzed using a gas chromatograph with dual electron capture detectors (ECDs). Specific Aroclor mixtures are identified by the pattern of peaks compared to chromatograms of reference standards. The concentrations of Aroclors in the sample extract are determined using an external standard calibration.

3.0 DEFINITIONS

3.1 Polychlorinated biphenyls (PCBs): PCBs are a class of organic compounds with 1 to 10 chlorine atoms attached to biphenyl, with a general chemical formula of $C_{12}H_{10-x}Cl_x$. There are 209 possible congeners.

3.2 Aroclor: PCBs were produced as technical mixtures by the chlorination of biphenyl. Production processes were designed to produce mixtures with characteristic chlorine contents. In the United States, most of the PCBs in the environment are in the form of Aroclors, which were produced by Monsanto from the 1930s through 1977. Each Aroclor mixture is identified by a four-digit number, the first two digits of which indicate the number of carbons in the biphenyl ring, i.e., 12, and the second two of which indicate the weight percent of chlorine. For example, Aroclor 1254 has 12 carbons and 54% by weight chlorine. The exception is Aroclor 1016, which has 12 carbons and 42% by weight chlorine.

NOTE: Each specific Aroclor produces a characteristic gas chromatographic pattern that represents the relative amounts of PCB congeners in the formulation. The formulation of the mixtures from batch to batch was fairly consistent, but never exactly the same. In almost all cases, the gas chromatogram can be used as a fingerprint to identify the specific Aroclor. Exceptions occurred for Aroclors 1254 and 1221. In each case, at least one batch was produced under different conditions, which resulted in an Aroclor mixture with the same approximate chlorine content , but with a significantly different distribution of congeners. These odd batches of 1254 and 1221 produce chromatographic patterns that are very different from the typical formulations. Standards for these odd batch Aroclors can be used to aid in the qualitative identification of Aroclors in environmental samples.

4.0 INTERFERENCES

4.1 Hydrocarbons can co-elute and thereby mask the Aroclor pattern. The laboratory uses acid cleanup with concentrated sulfuric acid to remove hydrocarbons from solid and oil sample extracts, and for water samples when extracts have noticeable color or whenever there is clear evidence of interferences in the initial sample chromatograms. Acid cleanup removes low-to-medium molecular weight polar organic interferences from sample extracts. Detailed instructions for performing acid cleanup are provided in SOP PT-OP-001.

4.2 Sulfur will interfere and can be removed using procedures described in SOP PT-OP-001.

4.3 Carboprep 90 Cleanup

4.3.1 This cleanup may be performed prior to analyses for PCBs if the sample extract has some color.

4.3.2 Cartridge Method

4.3.2.1 Put approximately 2 ml of sample extract into a test tube and mark the sample volume on the tube.

4.3.2.2 Condition the cartridge by adding 2 ml of methylene chloride and allowing it to drip through the cartridge. Do not allow the cartridge packing to go dry in this or any subsequent step, until the final rinse has been completed.

4.3.2.3 Add 2 ml of hexane/methylene chloride (80%/20%) mixture and allow it to drip through the cartridge until almost empty.

4.3.2.4 Add the sample extract to the cartridge and place the test tube under the cartridge to collect the liquid as it drips through.

4.3.2.3 Rinse 3 times with 2 ml aliquots of hexane/methylene chloride (80%/20%) mixture, while not allowing the cartridge to go dry. After the final rinse, use a pipette bulb to force out all of the remaining liquid in the cartridge.

4.3.2.4 Concentrate the sample extract back down to the original volume according to the mark on the test tube. The extract is now ready for analysis.

4.3.3 Quick Method

4.3.3.1 Add a half scoop of Carboprep 90 to approximately 2 ml of sample extract.

4.3.3.2 Shake for one minute and allow the extract to settle.

4.3.3.3 Pipette out an aliquot of the clear extract for analysis.

4.4 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Any affected samples are re-analyzed.

4.5 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.6 Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD).

4.7 Note: A standard of the DDT analogs should be injected to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, 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 of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.2.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2.4 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the TestAmerica Denver Radiation Safety Officer and the TestAmerica Corporate EH&S Director shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment.

5.3 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

Note: This list does not include all materials used in the method. 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 the method can be found in the reagents and materials section. 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.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hydrogen Gas	Explosive	None	The main hazard is flammability. Exposure to moderate concentrations may cause dizziness, headache, nausea, and unconsciousness. Exposures to atmospheres less than 8 to 10% oxygen will bring about sudden unconsciousness, leaving individuals unable to protect themselves. Lack of sufficient oxygen may cause serious injury or death.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Instrumentation

6.1.1 A gas chromatographic system with dual columns and dual ECD (^{63}Ni) detectors, and a data system capable of measuring peak area and/or height.

6.2 Supplies

6.2.1 Columns

6.2.2 Primary Column: MR1, 30 m x 0.53 mm id, 0.5 µm coating.

6.2.3 Secondary Column: MR2, 30 m x 0.53 mm id, 0.5 µm coating.

6.2.4 Additional columns that can be used for confirmation include 30m x 0.53mm id RTx5, RTx50 or RTx-1701.

6.3 Autosampler vials, crimp caps with PTFE-faced septa.

6.4 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Acetone, 99.4% for organic residue analysis

7.1.2 Hexane, pesticide grade.

7.1.3 Carrier Gas: ≥ 99.99999% pure hydrogen

7.1.4 Make-up Gas: ≥ 99.99980% pure nitrogen

7.2 Standards

7.2.1 Stock Standards

7.2.1.1 All standards must be refrigerated at 4 ± 2 ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using

7.2.1.2 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of receipt or earlier if the vendor indicates an earlier date.

7.2.1.3 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. The standards must be replaced at least every six months, or sooner, if comparison with check standards indicates a problem.

7.2.2 PCB and Surrogate Stock Calibration Standards

7.2.2.1 Stock A

For each of the Aroclors listed in Table 1, a commercially prepared stock standard solution is obtained. Each stock standard contains the specific Aroclor in isooctane at a concentration of 1,000 µg/mL. (Note: At times, specific Aroclor stock standards may also be purchased at various concentration levels, for example 2nd source stock standards are purchased at 200 ug/ml.)

7.2.2.2 Surrogate Stock B

A commercially prepared stock standard solution is obtained that contains the surrogate compounds tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in acetone, each at a concentration of 200 µg/mL.

7.2.3 Intermediate and Working Level Calibration Standard Solutions

7.2.3.1 **Stock C** (Level 7 Calibration) Standard Solutions

A Stock C standard solution is prepared for the various Aroclors or combination of Aroclors as summarized in the following table. In each case, the Stock C standard solution is also the highest concentration (i.e., Level 7) calibration standard.

Stock C	**Recipe**	**Conc (µg/mL)**	**Final Vol (mL)**	**Final Concentrations (µg/mL)**	
AR1660	0.1 mL of Aroclor 1016 Stock A 0.1 mL of Aroclor 1260 Stock A	100	10	Aroclor 1016 Aroclor 1260	1.0
	0.2 mL of surrogate Stock B	200	10	TCMX	4.0
		200	10	DCB	4.0
AR2154	0.125 mL of Aroclor 1221 Stock A		250	Aroclor 1221	0.5
	0.125 mL of Aroclor 1254 Stock A	1000	250	Aroclor 1254	0.5
AR1232	0.125 mL of Aroclor 1232 Stock A	1000	250	Aroclor 1232	0.5
AR1262	0.125 mL of Aroclor 1262 Stock A	1000	250	Aroclor 1262	0.5
AR1242	0.125 mL of Aroclor 1242 Stock A	1000	250	Aroclor 1242	0.5
AR1268	0.125 mL of Aroclor 1268 Stock A	1000	250	Aroclor 1268	0.5
AR1248	0.125 mL of Aroclor 1248 Stock A	1000	250	Aroclor 1248	0.5

7.2.4 AR1660 Calibration Levels

7.2.4.1 A total of 7 calibration standards are prepared for AR1660 as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the Aroclors, but only the AR1660 calibration standards include the surrogates. In all cases, measured volumes of the Stock C standard are diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Vol of Stock C Used (mL)	Final Volume (mL)	Final PCB Conc (µg/mL)	Final Surrogate Conc (µg/mL)*
1	0.01	100	0.01	0.0004
2	0.05	100	0.05	0.002
3	0.20	100	0.20	0.008
4 (CCV)	1.0	200	0.50	0.020
5	2.0	200	1.0	0.040
6	2.0	100	2.0	0.080
7	4.0	100	4.0	0.160
*Surrogates are in the AR1660 calibration solutions only. None of the other Aroclor calibration solutions contain the surrogate compounds.				

7.2.5 Working Single-Point PCB Calibration Standards

The Level 4 standard in the table above is used for single-point calibrations of the individual Aroclors. These standards are also used as pattern recognition standards.

7.3 Second-Source Standards for Initial Calibration Verification (ICV)

These standards are purchased from a vendor different from the one that supplied the stock calibration standards.

7.3.1 Second-Source Stock A' Aroclor Standard Solutions

Commercially prepared solutions in pesticide-grade hexane (or isooctane) are routinely obtained for Aroclors 1016 and 1260. The Aroclor concentration in each solution is 200 µg/mL. A second source may be obtained for the other Aroclors, if necessary.

7.3.2 Second-Source Working Level Standards

The working level second-source ICV standard is prepared by combining 0.5 mL of Aroclor 1016 Stock A' and 0.5 mL of Aroclor 1260 Stock A' diluting to a final volume of 200 mL with pesticide-grade hexane. This results in a concentration of 0.5 µg/mL for each of the Aroclors. Note: Surrogates are not added to any 2nd source working level standards.

7.4 Continuing Calibration Verification Standard (CCV), 0.5 µg/mL

The working CCV solution is the same as the Level 4 or Level 5 initial calibration standard, as shown in the table in Section 7.2.4.1.

7.5 Laboratory Control Standard (LCS) Spiking Solution (AR1660)

NOTE: The LCS/MS spiking solution is prepared and used as part of the scope of the organic preparation SOP PT-OP-001. The following information is provided for reference only.

The working LCS spike solution is prepared in a volumetric flask by combining 1 mL of the Aroclor 1016 Stock standard (10,000 ug/ml) and 1 mL of the Aroclor 1260 Stock standard (10,000 ug/ml), and diluting to a 250 mL final volume with acetone. The final concentration of each Aroclor is 40.0 µg/mL.

7.6 Matrix Spike (MS) Spiking Solution:

The working matrix spike solution is the same as the LCS spike solution. The MS duplicate (MSD) is prepared in the same way using a third aliquot of the selected sample.

Matrix Spike and LCS Solutions		
Analyte Group	Matrix spike Solution ID	Volume added in mL
AR1660	40 ug/mL PCB Spike	1.0 - waters
		0.5 - soils
		0.025 - low-level waters

7.7 Surrogate Spike Solution

7.7.1 Stock Surrogate Spike Solution:

A commercially prepared solution containing 200 µg/mL each of decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX) in acetone is purchased.

7.7.2 Working Surrogate Spike Solution

NOTE: Samples are spiked with the surrogate compounds during sample preparation, which is described in the organic preparation SOP PT-OP-001. The following information is provided for reference only.

The working surrogate spike solution is prepared in a volumetric flask by adding 1.6 mL of the DCB (1000 ug/mL) stock surrogate spike solution, 0.80 mL of the TCMX (2000 ug/mL) stock surrogate spike solution and diluting to a final volume of 2000 mL with acetone. The surrogate compounds are added to all field and QC samples as follows:

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume added in mL
PCB Aroclors	0.8 ug/mL DCB/TCMX	1.0 - Waters
		0.2 - Tissues
		0.5 - Solids
		0.025 low-level waters

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Water samples are collected in pre-cleaned amber glass bottles fitted with a Teflon-lined caps. To achieve routine reporting limits, a full one liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

8.2 Soil samples are collected in 8-ounce, wide-mouth jars with a Teflon-lined lid.

Matrix	Sample Container	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	Amber glass	Cool 4 ± 2°C	7 Days	40 Days from extraction	40 CFR Part 136.3
Soils	Glass	Cool 4 ± 2°C	14 Days	40 Days from extraction	N/A
Tissues	Borosilicate glass, PTFE, quartz, aluminum foil	Freeze at - 20 ± 5°C can be stored this way for 1 year prior to extraction [3]	14 Days from date of thawing	40 Days from extraction	PSEP[1],[2]

[1] This maximum holding time is also recommended by the PSEP (1990). The California Department of Fish and Game (1990), and the USGS National Water Quality Assessment Program (Crawford and Luoma, 1993) recommend a more conservative maximum holding time of 1 year at < 10°C for dioxins/furans.

[2] NOAA recommends a maximum holding time of < 3 months.

[3] NOTE: EPA (1995) lists the storage temperature as < -20ºC. However, the acceptance range at some labs is -20ºC ± 5ºC. TestAmerica applies this acceptance range, having assessed it as functionally equivalent, since no phase change is expected between -25ºC and -15ºC.

9.0 QUALITY CONTROL

9.1 Refer to the TestAmerica Pittsburgh QC program document (PT-QA-021) for further details on criteria and corrective actions. Refer to “project checklist” for project specific requirements.

9.2 For specific DoD quality control requirements refer to SOP # PT-QA-025, Implementation of the DoD QSM Version 3, January 2006 and Appendix I in this SOP. For DoD V4.2 refer to SOP PT-QA-029. Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.2.1 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP PT-QA-016. This is in addition to the corrective actions described in the following sections.

9.3 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.4 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument. See SOP PT-QA-021 for further details.

9.5 Method blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water (for aqueous sample batches) or sodium sulfate (for solid sample batches) to which the surrogate compounds are added (see SOP PT-OP-001). The method blank is subject to the entire extraction and analysis process.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher. Wherever blank contamination is greater than 1/10 the concentrations found in the samples and/or 1/10 of the regulatory limit it is potentially at a level of concern and should be handled as a non-conformance. Blank contamination should always be assessed against project specific requirements. If the analyte is a common laboratory contaminant (ex. phthalate esters) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

9.6 **Corrective Action**: Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.7 Laboratory Control Sample (LCS)

One LCS is prepared and analyzed with each batch of samples. The LCS is prepared as described in Section 7.5. The LCS is subject to the entire extraction and analysis process. See Table 7 for Laboratory generated Control Limits.

Acceptance Criteria: The LCS recovery must be within the established control limits. The laboratory's standard control limits are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. Current control limits are maintained in the LIMS. For each batch of samples, analyze a LCS. The LCS contains Aroclor 1016 and 1260, the same analytes are contained in the matrix spike. **For specific DoD quality control requirements refer to SOP # PT-QA-025.** For DoD V4.2 refer to SOP PT-QA-029.

When there are more than 11 target analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

Allowed Marginal Exceedances

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken. When using list of control compounds, all those compounds must be within acceptable limits.

9.8 **Corrective Action:** If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch. Refer to the SOP PT-QA-021 for further details of the corrective action.

9.9 If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. If the LCS recovery is high and the samples are ND for that analyte the data is reported with a NCM.

9.10 If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 11.0) unless one column is out of control, in which case the in control result is reported.

9.11 Use of marginal exceedances is not permitted for South Carolina work for LCS. For SC work the LCS limits must be 70-130% at a maximum. Same limits or lab calculated limits can be used for MS/MSD. If limits are calculated for LCS, they must be within 70-130 % range.

9.12 Matrix Spike (MS) and Matrix Spike Duplicate Samples (MSD)

One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require preparation and analysis of an MS/MSD pair at a 10% frequency. Preparation of the MS is described in Section 7.6. The MSD is another aliquot of the sample selected for the MS that is spiked in the same manner as the MS. See Table 7 for Laboratory generated Control Limits.

Acceptance Criteria: The MS and MSD recoveries must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. The relative percent difference (RPD) between the MS and MSD must be less than the established limit, which is based on statistical analysis of past results, unless otherwise dictated by project requirements. Current control limits are maintained in the LIMS. For SC work lab generated limits can be used for MS/MSD or same limits as LCS can be used for MS/MSD, 70-130%.

Corrective Actions: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

If the recovery for any component is outside control limits for both the MS and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.

If an MS/MSD is not possible due to limited sample, then an LCS duplicate (LCSD) should be analyzed.

The MS must be analyzed at the same dilution level as the unspiked sample, unless the matrix spike components would then be above the calibration range.

If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 11.4) unless one column is out of control, in which case the in control result is reported.

9.13 Surrogates

Each field sample, QC sample, and each calibration standard that is used for the AR1660 initial calibration, is spiked with surrogate compounds decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). The surrogate spike solution is prepared as described in Section 7.7.2.

Acceptance Criteria: The surrogate recoveries must be within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise.

Corrective Action: If recoveries of the surrogates in blanks are outside of the control limits, check for calculation or instrument problems. High recoveries might be acceptable if the surrogate recoveries for the samples and other QC in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples.

For field samples, surrogate recovery is calculated and reported for both DCB and TCMX. If one surrogate fails to fall within the control limits and the other surrogate is within the control limits, the data is considered reportable with an NCM and narration in the final report.

If matrix interference is not obvious from the initial analysis, it is only necessary to re-prepare and reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effects, as long as the extraction/instrument system is proven to be working properly. **For specific DoD quality control requirements refer to SOP # PT-QA-025.** For DoD V4.2 refer to SOP PT-QA-029.

9.14 Instrument QC

9.14.1 TestAmerica Pittsburgh gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

9.14.2 Use the ChemStation chromatography data system to set up GC conditions for calibration. See Table 2 for typical operating conditions.

9.14.3 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the ChemStation software to set up the analytical sequence.

9.14.4 Unprocessed calibration data are transferred to the TARGET DB database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist (GC and HPLC Data Review Checklist - ICAL). Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration reports are scanned and stored as Adobe Acrobat files on the Public Drive.

9.14.5 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new type of column and replacing the ECD detector.

9.15 Initial Calibration (ICAL)

9.15.1 An external standard calibration using seven concentration levels of the AR1660 mixture is routinely performed. (At least five calibration levels are required.) This provides concentration levels for Aroclor 1016, Aroclor 1260, and the surrogate compounds DCB and TCMX.

9.15.2 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

9.15.3 The calibration curves for Aroclors 1016 and 1260 and the surrogate compounds are modeled either as average calibration factors (CF) or as calibration curves using a systematic approach to selecting the optimum calibration function.

9.15.4 The calibration for each of the other Aroclors (see Table 1) is initially determined using a single, mid-level calibration standard. As needed, the laboratory may generate a multi-point calibration for other commonly detected Aroclors, such as 1221, 1254, and 1248. When additional multi-point calibrations are developed for the other Aroclors, a second-source ICV standard is also analyzed. For specific DoD quality control requirements refer to SOP # PT-QA-025. For DoD V4.2 refer to SOP PT-QA-029.

NOTE: Samples from sites known to be contaminated with specific Aroclors should be analyzed using a multi-point calibration curve for the identified Aroclors. This information is provided to the analyst through special instructions in the LIMS.

NOTE: Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or the linear range is adjusted accordingly. A minimum of five levels must remain in the calibration for a linear regression. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration.

9.15.5 The high and low standard for the initial calibration of the AR1660 mixture defines the acceptable quantitation range for all of the Aroclors. If a sample extract contains any Aroclor at a concentration that exceeds the upper range of the calibration, then the extract must be diluted and reanalyzed.

9.15.6 Select 5 major peaks in the analyte pattern (only 3 peaks are usable for Aroclor 1221). Calculate the response of each of the major peaks for each Aroclor, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be co-eluting with contaminant peaks from the quantitation (i.e. peaks that are significantly larger than would be expected from the rest of the pattern).

NOTE: A minimum of three accurate peaks must be used to quantify an Aroclor.

9.16 External Standard Calibration

External standard calibration involves the comparison of instrument responses from the samples to the responses from the target compounds in the calibration standards. The area (or height) of a peak in a sample chromatogram is compared to the area (or height) of the peak in the standard chromatograms that appears at the same retention time. The ratio of the detector response to the concentration of the target analyte in the calibration standard is defined as the calibration factor (CF) and is calculated as follows:

$$CF = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected}$$

Equation1

Note: It is also possible to use the concentration of the standard rather than the mass injected.

9.17 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors (CF) or as linear regression curves, using a systematic approach to select the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until calibration acceptance criteria are met.

9.17.1 Linear Calibration Using Average Calibration Factor (CF)

The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

The average calibration factor is calculated as follows:

Average CF = $\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$ Equation 2

Where:

CF_i = The calibration factor for the i^{th} calibration level.

n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{\overline{CF}}} \times 100\% \qquad \text{Equation 3}$$

Where SD is the standard deviation of the average RF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)^2}{n-1}} \qquad \text{Equation 4}$$

9.17.2 Evaluation of the Average Calibration Factor

Plot the calibration curve using the average CF as the slope of a line that passes through the origin. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Acceptance Criteria: The RSD must be ≤ 20%. SW-846 Method 8000B allows evaluation of the grand average across all compounds, but some programs (e.g., South Carolina) require evaluation of each compound individually. Check project requirements.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

9.17.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). A weighted least squares regression may be used if at least three multi-point calibrations have been performed. The weighting used is the reciprocal of the square of the standard deviation. The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b$$ Equation 5

Where:

y = Instrument response (peak area or height).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b$$ Equation 6

Where:

A_s = Peak area (or height) of the target analyte in the calibration standard.

C_s = Concentration of the target analyte in the calibration standard (µg/mL).

To calculate the concentration in an unknown sample extract, the regression equation 6 is solved for concentration, resulting in the following equation, where C_s is now C_e, the concentration of the target analyte in the unknown sample extract, and A_s is now A_e, the peak area (or height) of the target analyte in the sample extract.

$$C_e = \frac{A_e - b}{a}$$ Equation 7

9.17.4 Evaluation of the Linear Least-Squares Regression Calibration Function

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum “Clarification Regarding Use of SW-846

Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than reporting limit (RL), and preferably less than the MDL.

Also examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, the calibration should be repeated since a higher order regression is not allowed for this method.

The linear regression must have a correlation coefficient (r) ≥ 0.995.

Corrective Action: If the correlation coefficient falls below the acceptance limit, the linear regression is unacceptable and the calibration should be repeated since a higher order regression is not allowed for this method.

9.17.5 Non-Linear Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c \qquad \textit{Equation 8}$$

Where a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

Evaluation of Second-Order Regression Calibration

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria: The coefficient of determination (COD) must be (r^2) ≥ 0.99.

Second-order regressions should be the last option, and note that some programs (e.g., South Carolina) do not allow the use of second-order regressions. Before selecting a second-order regression calibration model, it is important to ensure the following:

- The absolute value of the intercept is not large relative to the lowest concentrations being reported.
- The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).
- The distribution of concentrations is adequate to characterize the curvature.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the problem should be investigated and the instrument recalibrated.

9.17.6 Polynomial regression fits of third order or higher are not allowed for this method.

9.18 Second-Source Initial Calibration Verification (ICV)

The second-source ICV standard usually consists of Aroclors 1016 and 1260 only. The stock standards are obtained from a source different than that of the standards used for the calibration. The preparation of the ICV standard is described in Section 7.3. The concentration of each Aroclor in the ICV is 0.25 µg/mL; the concentration of each surrogate is 0.125 µg/mL. The ICV standard is analyzed immediately following the initial calibration.

If it is necessary to generate a multi-point calibration for any of the other Aroclors, then an ICV standard containing the specific Aroclor(s) is analyzed immediately following the calibration.

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 20% of the expected value.

Corrective Action: If this is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

9.19 Continuing Calibration Verification (CCV)

12-Hour Calibration Verification

The 12-hour calibration verification sequence consists of, at a minimum, an instrument blank and the mid-level calibration standard. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter when samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration sequence.

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

9.19.1 Continuing Calibration Verification (CCV)

It may be appropriate to analyze a mid-level (i.e. Level 3, 4 or 5) standard more frequently than every 12 hours. The mid-level calibration standard is analyzed as the continuing calibration verification (CCV) standard. At a minimum, this is analyzed after every <u>20</u> samples, including matrix spikes, LCSs, and method blanks. Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns. If 12 hours elapse, analyze the 12-hour standard sequence instead.

9.19.1.1 The daily CCV analysis, at a concentration other than the mid level (to meet NELAC requirements) will consist of Aroclors 1016/1260. All other CCVs will be a mid level Aroclor 1016/1260 standard. The CCV requirements for DoD work are listed in SOP PT-QA-025. For DoD V4.2 refer to SOP PT-QA-029.

9.19.1.2 A mid level calibration standard of the other five Aroclors (1221, 1232, 1242, 1248, 1254), to verify the initial calibration, is analyzed at least once every 72 hours.

9.19.2 Acceptance Criteria for Continuing Calibration Verification (CCV)

Detected Analytes (≥ RL):

For any analyte <u>detected</u> at or above the reporting limit (RL) in client samples, the percent difference (%D) for that analyte in the preceding and following CCVs (i.e., bracketing CCVs) or 12-hour calibration, on the column used for quantitation, must be within ± 20%.

In some cases, the nature of the samples being analyzed may be the cause of a failing %D. When the %D for an analyte falls outside of ± 20% in the CCV, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect if so desired by the client.

Refer to Section 11 for which result to report.

The %D is calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Theoretical Conc}}{\text{Theoretical Conc}} \times 100 \qquad \text{Equation 8}$$

Analytes Not Detected (< RL):

For any analyte not detected (ND) in client samples, the %D for that analyte in the bracketing CCVs should also be within ± 20%.

However, if the CCV %D exceeds +20% and the sample results are ND, it still may be possible to report sample results. In this case, the client should be consulted and an NCM written.

If the CCV % D falls below -20% and sample results are ND, but the target analytes are detected in the RL Standard, it may still be possible to report sample results, since the detection of the analyte(s) in the RL Standard indicate that there was sufficient sensitivity to detect the analyte(s) in the samples. In this case, the client should be consulted and an NCM written.

9.20 Retention Time (RT) Windows

Absolute retention times are used for the identification of PCBs as Aroclors. However, in addition to retention times, peak patterns play a large role in the identification of Aroclors.

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

Before establishing windows, make sure the GC system is within optimum operating conditions. Make 3 injections of all standard mixtures throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

Determine the retention time (RT) windows for the 3-5 major peaks selected for each Aroclor. The AR1016 windows will be used to establish retention time windows for PCBs AR1221, AR1016, AR1232, AR1242, and AR1248. The AR1260 windows will be used to establish retention time windows for PCBs AR1254, AR1260, AR1262, and AR1268.

When conducting either Aroclor or congener analysis, it is important to determine that common single-component pesticides such as DDT, DDD and DDE do not elute at the same retention times as the target congeners. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples. Therefore, in conjunction with determining the retention time windows of the congeners, the analyst should analyze a standard containing the DDT analogs. This standard need only be analyzed when the retention time windows are determined. It is not considered part of the routine initial calibration or calibration verification steps in the method, nor are there any performance criteria associated with the analysis of this standard.

If Aroclor analysis is performed and any of the DDT analogs elute at the same retention time as an Aroclor peak that was chosen for use in quantitation, then the analyst must either adjust the GC conditions to achieve better resolution, or choose another peak that is characteristic of that Aroclor and does not correspond to a peak from a DDT analog. If PCB congener analysis is performed and any of the DDT analogs elute at the same retention time as a PCB congener of interest, then the analyst must adjust the GC conditions to achieve better resolution.

Calculate the mean and standard deviation of the three RTs for each analyte as follows:

$$\text{Mean RT} = \overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RT_i - \overline{RT}\right)^2}{n-1}} \qquad \text{Equations 9 and 10}$$

Where:

RT_i = Retention time for the i^{th} injection.

n = Number of injections (typically 3).

SD = Standard deviation.

If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).

The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. For multi-response analytes, use the RT of major peaks. If the default standard deviation is employed, the width of the window will be 0.03 minutes. This allows for slight variations in RTs caused by sample matrix. Typically for PCBs the width of the window is 0.05 minutes.

The center of the RT window for each analyte is the RT from the last of the three analyses of the standard. The center of the window for each analyte is updated with the RT from the level 4 standard of the ICAL, the CCV at the beginning of the analytical sequence, and with each subsequent 12-hour calibration verification. The width of each window remains the same until new windows are generated following the installation of a new column different from those mention in Section 6 or in response to an RT failure.

Acceptance Criteria for Retention Times:

The RT for each compound in each CCV analysis must be within the RT windows established by the daily initial CCV.

Corrective Action for Retention Times:

If a target analyte falls outside the established RT window in a CCV standard, either adjust the center of the window based on the CCV, or investigate the problem and calculate new RT windows. All samples analyzed after the last acceptable CCV must be reanalyzed.

9.20.1 Sample Retention Time Criteria

The surrogate must fall within the established RT window. Target analyte peaks must be within the established RT window to be reported as such. If the surrogate

RT indicates an RT shift, it may be possible to accept a target analyte peak if it has not shifted relative to the surrogate peak.

9.20.2 Daily Retention Time Windows

9.20.2.1 Establish the center of the retention time windows from the calibration verification at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. Retention time windows can be updated every 12 hours. However, they are usually only updated at the onset of a continuing calibration sequence or after maintenance has been performed.

The laboratory must calculate retention time windows for each analyte on each GC column and whenever a new type of GC column is installed. The data must be retained by the laboratory and available for review.

9.21 Control Limits: Control Limits: For DoD work the control limits are listed in the SOP PT-QA-025. For DoD V4.2 refer to SOP PT-QA-029. For DoD QC acceptance criteria refer to DoD SOP listed above. In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be reviewed at least annually. The recovery limits are mean recovery +/- 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Refer to policy PT-QA-021 for more details.

9.22 These limits do not apply to dilutions, but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.23 Percent Moisture

9.23.1 Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight.

9.23.2 Procedural Variations

9.23.2.1 Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 Samples are extracted and prepared for analysis as described in SOP PT-OP-001.

10.2 Calibration

10.2.1 Chromatographic conditions for this method are presented in Table 2.

10.2.2 Use the ChemStation interface to establish instrument operating conditions for the GC.

10.2.3 Raw data obtained by the ChemStation software is transferred to the TARGET DB database for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the TARGET DB software.

10.2.4 The instrument is set up and calibrated as described in Section 10.2.1 above.

Calibration Controls	Sequence	Control Limit
Calibration Standards	7-point (minimum) linearity	<u><</u>20% RSD
Cont. Cal. Verif. (CCV)	Prior to / after every 20 samples	% D ± 20%
RT Windows (RTW)	Init. CCV determines midpt. of RTW – updated daily	<u>+</u>3X SD

10.3 Sample Analysis

10.3.1 An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract.

10.3.2 Samples, standards, and QC samples must be introduced using the same procedure.

10.3.3 All extracts and standards are allowed to warm to room temperature before injection.

10.3.4 Use the ChemStation interface to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

10.4 Analytical Sequence

10.4.1 n analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. The daily run sequence is generated electronically using the ChemStation software.

10.4.2 The daily calibration verification includes analysis of the 12-hour calibration sequence and updating the retention time windows.

10.4.3 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration verification.

10.4.4 Following is the typical analytical sequence for routine sample analysis:

Suggested Analytical Sequence

Initial Calibration		
Injection No.		
1	Solvent Blank (optional)	
2	Aroclor 1221/1254	Level 4
3	Aroclor 1232	Level 4
4	Aroclor 1242	Level 4
5	Aroclor 1248	Level 4
6	Aroclor 1016/1260	Level 1
7	Aroclor 1016/1260	Level 2
8	Aroclor 1016/1260	Level 3
9	Aroclor 1016/1260	Level 4

Suggested Analytical Sequence		
Initial Calibration		
Injection No.		
10	Aroclor 1016/1260	Level 5
11	Aroclor 1016/1260	Level 6
12	Aroclor 1016/1260	Level 7
13-17	ICV's (2nd source standard of all Aroclors)	
18	Solvent Blank (optional)	
19-38	Sample 1-20 (or 12 hours)	
39	Aroclor 1016/1260	Level 4
At least every 12 hours, counting from the start of the initial calibration or from the start of the last daily calibration, the retention time windows must be updated using the 1016/1260 mix. Mid-level standards of any other Aroclors expected to be present in the samples are also injected.		

10.5 Retention Times - Samples

The centers of the retention time windows are updated with the mid point of the initial calibration and each 12 hour calibration.

Acceptance Criteria for Retention Times: The retention times of all compounds in each continuing calibration must be within the established retention time windows.

Corrective Action for Retention Times: If retention times do not fall within the established RT windows, then all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

10.6 When a sample result exceeds the upper calibration range, then that sample extract is diluted to obtain a result in the upper half of the calibration range and reanalyzed. Any samples that were analyzed immediately following the high sample are evaluated for carryover. If the samples had target analyte detections at or above the RL, the samples must be reanalyzed to rule out carryover.

10.7 Upon completion of the analytical sequence, transfer the raw chromatography data to the TARGET DB database for further processing. Review chromatograms online and determine whether manual data manipulations are necessary. All manual integrations must be justified and documented. See CA-Q-S-002 for requirements for manual integration. Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature. Alternatively, the manual integration may be processed manually. In the latter case, print both the both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration. **For specific DoD quality control requirements refer to SOP # PT-QA-025. For DoD V4.2 refer to SOP PT-QA-029.**

10.8 Case Narrative: For DoD the case narrative shall provide: identification of **samples and analytes** for which manual integration was necessary. DoD QSM, Version 3, Appendix DoD-A. For DoD V4.2 refer to SOP PT-QA-029

10.9 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD. Perform a level 1 data review and document the review on the data review checklist (GC and HPLC Data Review Checklist). Submit the data package and review checklist to a peer analyst for the level 2 review. The data review process is explained in SOP PT-QA-018.

11.0 CALCULATIONS / DATA REDUCTION

11.1 Qualitative Identification of Aroclors

Retention time windows are used for identification of Aroclors, but the “fingerprint” produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

11.2 Quantitation of Aroclors

Quantitation of Aroclors is accomplished using 3-5 major peaks. The peaks must be within the established retention time windows. If there is an interference that affects the accuracy of results, the analyst may use as few as 3 major peaks (2 peaks for Aroclor 1221). The same peaks that are used for sample quantitation must be used for standards and QC quantitation.

11.3 Second column confirmation of Aroclors is performed only when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: USACE projects require the use of second-column confirmation of Aroclors unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis. See also the DoD SOP PT-QA-025. For DoD V4.2 refer to SOP PT-QA-029.

NOTE: South Carolina requires second column confirmation.

11.4 Dual Column Quantitation

11.4.1 NOTE: Dual column quantitation is not routinely performed for PCB analysis. This section is included for those clients/projects that require dual column confirmation.

11.4.2 When reporting the analytical results for field samples of dual column analysis, the higher of the two results is normally reported. For specific DoD quality control requirements refer to SOP # PT-QA-025. For DoD V4.2 refer to SOP PT-QA-029. The result from the second column confirmation is reported if any of the following is true:

- There is obvious chromatographic interference on the initial quantitation column.
- The difference between the result on the initial quantitation column and the result on the second column is > 40% and chromatographic interference is evident.
- A continuing or bracketing standard fails on the initial quantitation column, but is acceptable on the second column. However, if the difference

between the initial quantitation column and second column results is > 40% and the initial quantitation column calibration verification fails, then the sample must be evaluated for reanalysis.

11.4.3 Dual Column Results With > 40% RPD:

11.4.3.1 If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the higher of the two results is reported unless there is obvious interference documented on the chromatogram.

11.4.3.2 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

11.4.4 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

11.4.5 If the relative percent difference (RPD) between the results on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. The RPD is calculated as follows:

$$\%RPD = \frac{|R_1 - R_2|}{1/2(R_1 + R_2)} \times 100\% \qquad \text{Equation 11}$$

Where R_1 is the result for the initial quantitation column, and R_2 is the result for the second column.

11.5 Surrogate Recovery

11.5.1 Surrogate recovery results are calculated and reported for tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in all samples. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

11.6 Calibration Range and Sample Dilutions

11.7 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for any analyte, they must be reanalyzed to rule out carryover. It may also be necessary to dilute samples because of matrix interferences.

11.7.1 If the initial diluted run has no hits or hits below 20% of the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.7.2 Guidance for Dilutions Due to Matrix Interference

11.7.3 If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

11.7.4 Reporting Dilutions

11.7.5 Some programs (e.g., South Carolina) and some projects require reporting of multiple dilutions (check special requirements in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported. Dilutions 3-5X report the data and narrate. Dilutions greater than 5X then reported diluted out.

11.8 Interferences in Observed in Samples

11.8.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

11.8.2 Suspected Negative Interferences

11.8.3 If peak detection is prevented by interferences, further cleanup should be attempted. Elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

11.8.4 Suspected Positive Interferences

11.8.4.1 If no further cleanup is reasonable and interferences are evident that are suspected of causing false positive results, consult with the laboratory Project Manager to determine if analysis using additional confirmation techniques is appropriate for the project. Use of additional confirmation columns is another possible option.

11.9 Calculations

11.9.1 Concentration of Analyte in Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10 for details on establishing the calibration function):

Average Calibration Factor: $C_e = \frac{A_e}{\overline{CF}}$ Equation 12

Linear Regression: $C_e = \frac{[A_e - b]}{a}$ Equation 13

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).

A_e = Peak area for the analyte in the sample extract injection.

b = y-intercept of the calibration fit.

a = Slope of the calibration fit.

11.9.2 Concentration of Analyte in Original Sample

The concentration of the analyte in the original sample is calculated as follows:

$$C_{sample} = \frac{C_e}{1000\frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF \qquad \text{Equation 14}$$

Where:

C_{sample}	=	Concentration of analyte in original sample (µg/L or µg/kg).
C_e	=	Concentration of analyte in sample extract injected in GC (ng/mL).
$1000\frac{ng}{\mu g}$	=	Factor to convert ng/mL to µg/mL.
V_e	=	Volume of sample extract (mL).
V_s	=	Volume (or weight) of original sample (L or kg).
DF	=	Dilution Factor (post extraction dilutions)

11.9.3 Spike Recovery Calculation

LCS, MS, and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Measured Concentration}}{\text{Spiked Concentration}} \times 100\%$$

MS/MSD RPD Calculation

The percent difference between the analyte concentration in the MS and the MSD is calculated as follows:

$$RPD = \frac{|MS - MSD|}{1/2(MS + MSD)} \times 100\%$$

11.10 For manual integration practices refer to TestAmerica SOP, CA-Q-S-002, Acceptable Manual Integration Practices. For DoD and all other projects the following criteria must be met:

11.10.1 When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and signature/initials of person performing manual integration operation (electronic signature is acceptable). DoD QSM, Version 3, Clarification 50 and 57. For DoD V4.2 refer to SOP PT-QA-029.

11.11 Case Narrative: For DoD the case narrative shall provide: identification of samples and analytes for which manual integration was necessary. DoD QSM, Version 3, Appendix DoD-A. For DoD V4.2 refer to SOP PT-QA-029.

11.12 All data are subject to two levels of review, which is documented on a checklist, as described in SOP PT-QA-018.

12.0 METHOD PERFORMANCE

12.1 Method Detection Limit Study (MDL)

12.2 An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy PT-QA-007. An MDL verification is performed once a year to satisfy NELAC 2005 requirement. For DoD, an MDL verification is performed quarterly.

12.3 Demonstration of Capabilities

12.3.1 An initial demonstration of capability for each method must be performed prior to analyzing samples in accordance with PT-QA-001.

12.3.2 For the standard analyte list, the initial demonstration consists of the preparation and analysis of an LCS sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study (described in Section 12.1).

12.3.3 Four LCS samples are analyzed with the same procedures used to analyze samples, including sample preparation.

12.3.4 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.

12.3.5 For non-standard analytes an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is successful analysis of an extracted standard at the reporting limit and a single point calibration.

12.4 Training Requirements

12.4.1 The Group/Team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP PT-QA-021 and PT-QA-001.

12.4.2 Each analyst performing the method must complete an initial demonstration of capability (IDOC) by successfully preparing and/or analyzing four consecutive LCS samples or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the IDOC study are summarized in the NELAC format, as described in SOPs PT-QA-021 and PT-QA-001. IDOCs are approved by the Quality Assurance Manager and the Technical Director/Lab Director. IDOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 POLLUTION CONTROL

13.1 It is TestAmerica’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention.”

14.0 WASTE MANAGEMENT

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.2 Methylene Chloride in vials. This waste is placed in waste container identified as "Vials & Extracts", Waste #7.

14.3 Flammable solvents in vials. This waste is placed in waste container identified as "Vials & Extracts", Waste #7.

14.4 Waste flammable solvents. This waste is collected in a waste container identified as "Mixed Flammable Solvent Waste", Waste #3.

14.5 Expired primary and working PCB standards. This waste is placed in a waste container identified as "PCB Standard Waste", Waste #8.

14.6 Samples containing polychlorinated biphenyls (PCB's) at concentrations $\geq$50 ppm are regulated under the Toxic Substance Control Act (TSCA) and must be segregated from all other waste streams. Analysts are responsible for contacting the Group Leader, Sample Control, and the Health and Safety Coordinator <u>immediately</u> if a sample falls into the TSCA category.

15.0 REFERENCES / CROSS-REFERENCES

15.1 Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatograph, Revision 1, February, 2007, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.2 Method 8000B, Determinative Chromatographic Separations, Revision 2, December, 1996, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.3 SOP PT-OP-001, Extraction and Cleanup of Organic Compounds from Waters and Solids, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series Methods, current version.

15.4 SOP PT-QA-001, Employee Orientation and Training, current version.

15.5 SOP CA-Q-S-002, Manual Integration, current version.

15.6 SOP PT-QA-007, Determination of Method Detection Limits (MDLs), current version.

15.7 SOP PT-QA-016, Nonconformance & Corrective Action System, current version.

15.8 SOP PT-QA-018, Technical Data Review Requirements, current version.

15.9 SOP PT-QA-021, Quality Control Program, current version.

15.10 PT-LQAM, Pittsburgh Laboratory Quality Assurance Manual, current version.

15.11 SOP PT-QA-025, Implementation of the DoD QSM Version 3, January 2006, current version.

15.12 SOP PT-QA-029, DoD Version 4.2 Requirements, current version.

16.0 METHOD MODIFICATIONS:

16.1 Method 8082A includes an internal standardization option. Because of the high probability of interferences affecting internal standards, this is strictly an external standard SOP.

16.2 Method 8082A references 8000B, which allows the use of third-order calibration curves. This SOP does not allow third-order calibration curves.

17.0 ATTACHMENTS

17.1	Table 1.	Analyte List and Standard Reporting Limits
17.2	Table 2.	Typical Instrument Conditions
17.3	Table 3.	Calibration Levels (µg/mL)
17.4	Table 4.	LCS/Matrix Spike and Surrogate Spike Levels (µg/L)
17.5	Table 5.	Preparation of Calibration Standards
17.6	Table 6.	Surrogate Recovery Limits

17.7 Table 7. LCS and MS/MSD Control Limits

18.0 REVISION HISTORY

18.1 Revision 1:

18.1.1 Added section 9.12: Use of marginal exceedances are not permitted for South Carolina work. For SC work the LCS limits must be 70-130% at a maximum. Same limits or lab calculated limits can be used for MS/MSD. If limits are calculated for LCS, they must be within 70-130 % range. Modified section 9.13 to reflect SC requirements.

18.1.2 Added reference to SOP PT-QA-029.

18.2 Revision 2

18.2.1 Added Appendix A for PCB Congener analysis by SW-846 Method 8082A at the end of this SOP using the information formerly found in PT-GC-001 concerning Congener Analysis.

18.3 Revision 3

18.3.1 Updated reference throughout the SOP from DoD QSM 4.1 to 4.2.

18.4 Revision 4

18.4.1 On the cover page, removed Nasreen DeRubeis as QAM and replaced with Pam Dudeck as Interim QAM.

18.4.2 Corrected the Table under section 7.2.3.1 so that 1260 and TCMX are referencing the proper concentrations.

18.4.3 Added text under section 9.20 to indicate that a DDT check is analyzed when a new retention time window is established; also added text on the determination of the RT windows.

18.4.4 In section 9.20.2.1, added text to clarify when RT studies are to be done and how they are to be maintained.

Table 1

Standard Analyte List and Reporting Limits

Compound	**CAS #**	**Reporting Limit, µg/L, µg/wipe or µg/kg**		
		Water/Wipe Regular/ Low Level	**Soil/ Tissue Regular/Low Level**	**Waste**
Aroclor 1016	12674-11-2	0.4/0.01	16.67/0.833	500
Aroclor 1221	11104-28-2	0.4/0.01	16.67/0.833	500
Aroclor 1232	11141-16-5	0.4/0.01	16.67/0.833	500
Aroclor 1242	53469-21-9	0.4/0.01	16.67/0.833	500
Aroclor 1248	12672-29-6	0.4/0.01	16.67/0.833	500
Aroclor 1254	11097-69-1	0.4/0.01	16.67/0.833	500
Aroclor 1260	11096-82-5	0.4/0.01	16.67/0.833	500
Optional Compounds:				
Aroclor 1262	37324-23-5	0.4/0.01	16.67/0.833	500
Aroclor 1268	11100-14-4	0.4/0.01	16.67/0.833	500

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	**Final Vol.**	**Low Level Vol.**
Groundwater	1000 mL	40 mL	1 mL
Wipe	1 wipe	40 mL	NA
Low-Level Soil	15 g	20 mL	1 mL
High-Level Soil/Waste	1 g	40 mL	NA
Tissue	6 g	2 mL (1 mL with GPC clean-up)	NA

Table 2 Typical Instrument Conditions	
Parameter	Recommended Conditions
Injection Port Temperature:	220 °C
Detector Temperature:	325 °C
Temperature Program:	120 °C for 0.75 minute 18 °C/min to 260 °C 1 minute hold 20 °C/min to 300 °C, 1.5 minute hold
Column 1:	MR1, 30 m x 0.53 mm id, 0.5 µm
Column 2:	MR2, 30 m x 0.53 mm id, 0.5 µm
Injection:	1 or 2 µL
Carrier Gas:	Hydrogen or Helium
Make-up Gas:	Nitrogen

Table 3

Calibration Levels ng/mL

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7[1]
AR1660	10	50	200	500	1000	2000	4000
AR1242[2]				500			
AR2154[2]				500			
AR1232[2]				500			
AR1248[2]				500			
Surrogates are included with all the calibration mixes at the following levels:							
TCMX	0.5	2.5	10	25	50	100	200
DCB	0.5	2.5	10	25	50	100	200

[1] Level 7 is optional and should only be used if linearity can be maintained on the instrument to this level.

[2] Aroclors may be quantitated within the range 100 to 2000 ng/mL (4000 ng/mL if the level 6 1016/1260 standard is included). If the Aroclor is more concentrated, it must be reanalyzed at a dilution.

Table 4

LCS/Matrix Spike and Surrogate Spike Levels for Aroclor Analysis
µg/L, µg/wipe or µg/kg

	Aqueous/Wipe	Soil/Tissue	Waste
Aroclor 1016/1260	10	333	10,000
TCMX (surrogate)	0.20	6.67	200
DCB (surrogate)	0.20	6.67	200

Table 5

Preparation of Calibration Standards

Calibration Level	1016/1260 Intermediate (µL)	1221 + 1254 Stock (µL)	1232 Stock (µL)	1242 Stock (µL)	1248 Stock (µL)
Level 1	4				
Level 2	20				
Level 3	80				
Level 4	1000	250	250	250	250
Level 5	400				
Level 6	800				
Level 7	1600				

The surrogate stock is purchased (Decachlorobiphenyl and Tetrachloro-m-xylene) at 200 ug/mL.

The Aroclor 1016 and 1260 stock standards are purchased as certified standards in isooctane at 1000 ug/mL. The other five Aroclor stock standards are purchased at 200 ug/mL.

For Aroclors 1016 and 1260, an intermediate standard is prepared by diluting 1.0 mL of each of the stock standards and 0.25 mL of the surrogate stock standard to 10.0 mL in hexane. The intermediate Aroclor 1016/1260 standard concentrations are 100 ug/mL for each Aroclor and 5 ug/mL for each surrogate.

The Aroclor 1016/1260 calibration standards are prepared by diluting the volumes noted in Table C-6 to a 40.0 mL final volume in hexane except for the Level 3 standard, which is taken to a 200mL final volume in hexane.

The mid level (Level 3) calibration standards for each of the other five Aroclors (1221, 1232, 1242, 1248, 1254) are prepared by diluting 0.25 mL of the appropriate stock standard to a final volume of 100 mL in hexane. Aroclors 1221 and 1254 are combined into one standard and Aroclors 1232, 1242, and 1248 are prepared individually.

Table 6		
Surrogate Recovery Limits[1]		
Surrogate	Water Recovery Limits	Soil Recovery Limits
Decachlorobiphenyl	35-140	35-140
Tetrachloro-m-xylene	35-140	35-140

[1]**NOTE:** The Surrogate Recovery Limits are subject to change as in-house control limits are evaluated and updated by the QA Department.

Table 7						
LCS and MS/MSD Control Limits[1]						
	Soil			**Water**		
	LCS and MS/MSD			**LCS and MS/MSD**		
Compound	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Aroclor 1016	55	130	35	60	130	27
Aroclor 1248	30	150	35	30	150	35
Aroclor 1254	50	150	35	50	150	35
Aroclor 1260	54	130	29	60	130	24

[1]**NOTE:** The Surrogate Recovery Limits are subject to change as in-house control limits are evaluated and updated by the QA Department.

Appendix A

Analysis of PCB Congeners Based on Method 8082A

1.0 SCOPE OF METHOD

1.1 This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) congeners by GC/ECD. This Appendix is to be applied when SW-846 Method 8082A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate TestAmerica sample extraction SOP (PT-OP-001). The PCBs are determined and quantitated as individual PCB congeners.

1.2 Table A-1 lists the congeners, which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

2.0 SUMMARY OF METHOD

2.1 This method presents conditions for the analysis of prepared extracts for PCB congeners. The PCBs are injected onto the GC column(s) and separated and detected by electron capture detection. Quantitation is by the external standard method.

3.0 DEFINITIONS

3.1 Refer to the PT-LQAM for definitions of terms used in this document.

4.0 INTERFERENCES

4.1 Refer to Section 4 of the main body of this SOP for information regarding chromatographic interferences.

4.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3 Sulfur will interfere and can be removed using procedures described in SOP PT-OP-001.

4.4 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), Sulfur cleanup (Method 3660), and Acid Cleanup (Method 3665). These cleanup procedures are included in SOP PT-OP-001.

5.0 SAFETY

5.1 Refer to Section 5 of the main body of this SOP for general safety requirements.

5.2 PCB congeners have been classified as a potential carcinogen under OSHA. Concentrated solutions of PCB congeners must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.3 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.4 All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

6.0 EQUIPMENT AND SUPPLIES

6.1 Refer to Section 6 of the main body of this SOP. A GC equipped with a 63Ni electron capture detector is required.

6.2 Refer to Table A-2 for analytical columns.

6.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Acetone, 99.4% for organic residue analysis.

7.1.2 Hexane, pesticide grade.

7.1.3 Carrier Gas: ≥ 99.99999% pure hydrogen.

7.1.4 Make-up Gas: ≥ 99.99980% pure nitrogen.

7.2 Standards

7.2.1 Stock Standards

7.2.1.1 All standards must be refrigerated at 4 ± 2°C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before use.

7.2.1.2 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually or earlier if the vendor indicates an earlier date.

7.2.1.3 Dilutions from stock standards can not have a later expiration date than the date assigned to the parent stock solutions. The standards must be replaced at least every six months, or sooner, if comparison with check standards indicates a problem.

7.2.2 PCB Congener and Surrogate Stock Standards

7.2.2.1 PCB Congener Stock Mix 1: For the PCB Congeners listed in Table A-1, except BZ-205 (surrogate), a commercially prepared stock standard solution is obtained. The concentration of the PCB Congeners contained in Mix 1 is 4.0 ug/mL and the concentration of the TCMX (surrogate) is 6.6 ug/mL PCB Congener Stock Add-on Mix: This stock standard is prepared by adding 400 uL of 100 ug/mL of the following individual congener standards brought to a final volume of 10 mL with Hexane and resulting in a final concentration of 4 ug/mL: BZ-205 (surrogate).

7.2.2.2 TCMX (2000 ug/mL) and BZ-205 (100 ug/mL) Surrogate Stock Standards are commercially purchased standards.

7.2.2.3 PCB Congener Matrix Spike Stock Standard is a commercially purchased standard containing BZ-8, BZ-18, BZ-28, BZ-44, BZ-52, BZ-66, BZ-77, BZ-101, BZ-105, BZ-118, BZ-126, BZ-128, BZ-138, BZ-153, BZ-170, BZ-180, BZ-187, BZ-195 and BZ-206 at a concentration of 100 ug/mL. (NOTE: BZ-49, BZ-87, BZ-156, BZ-169 and BZ-187 are also used, however these are separate commercially purchased stock solutions also at a concentration of 100 ug/mL).

7.2.3 Calibration Curve Standard Solutions

7.2.3.1 The calibration standards are prepared as follows:

Calibration Level	Recipe	Stock Concentration (ug/mL)	Final Volume (ml of Hexane)
1	5 uL of PCB Congener Stock Mix 1 and 5 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
2	10 uL of PCB Congener Stock Mix 1 and 10 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
3	25 uL of PCB Congener Stock Mix 1 and 25 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
4	50 uL of PCB Congener Stock Mix 1 and 50 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
5	100 uL of PCB Congener Stock Mix 1 and 100 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
6	200 uL of PCB Congener Stock Mix 1 and 200 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40

7.2.3.2 Refer to Table A-3 for details of calibration standard concentrations.

7.2.4 Surrogate Standards

7.2.4.1 The Working Surrogate Solution is made as follows: 2.5 uL of the 2000 ug/mL commercially purchased TCMX stock and 50 uL of the 100 ug/mL commercially purchased BZ-205 stock brought to a final volume of 20 mL with Acetone (see section 7.2.2.3). The final concentration of the Working Surrogate Solution is 0.025 ug/mL. Refer to Table A-3 for details of surrogate standard concentrations in the calibration standards.

NOTE: The Working Surrogate Solution is prepared and used as part of the scope of the organic preparation SOP PT-OP-001. The preceding information is provided for reference only.

7.2.5 Laboratory Control Standard (LCS) and Matrix Spiking (MS) Solution

NOTE: The LCS/MS spiking solution is prepared and used as part of the scope of the organic preparation SOP PT-OP-001. The following information is provided for reference only.

7.2.6 The working LCS/MS spiking solution is prepared by combining 1mL of the PCB Congener Matrix Spike Stock Standard and 1 mL each of the BZ-49, BZ-87, BZ-156, BZ-169 and BZ-187 stock standards brought to a 100 mL final volume with Acetone. The final concentration of the working LCS/MS spiking solution is 1.0 ug/mL.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Refer to Section 8 of the main body of this SOP.

9.0 QUALITY CONTROL

9.1 Refer to Section 9 of the main body of this SOP.

10.0 PROCEDURE

10.1 Calibration and Standardization

10.1.1 Refer to Section 10 of the main body of this SOP for general calibration requirements.

10.2 Initial Calibration

10.2.1 Refer to Table A-5 for the initial calibration analytical sequence.

10.2.2 The response for each PCB congener will be calculated by the procedures described in the general method for 8082A analysis, with the following modifications.

10.2.3 A minimum five-point calibration of each of the individual congeners mixes is generated. At least 2 separate mixes are prepared to ensure that there is complete resolution of all congeners in the mixes. Calibration levels are listed in Table A-3. Refer to Table A-6 for preparation of calibration levels.

10.3 Initial Calibration Verification

10.3.1 The ICV will consist of second source standards of all congeners of interest. The ICV is prepared from a second source stock standard of a concentration of 4ppm for all congeners of interest. 25 uL of the ICV stock standard is added to Hexane and brought to a final volume of 40 mL for a concentration of 0.0025 ppm.

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 20% of the expected value.

Corrective Action: If this is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

10.3.2 12 hour Calibration

The 12-hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration.

The retention time windows for any analytes included in the daily calibration are updated.

For this method, samples must be bracketed with successful calibration verification runs. **Cannot use quadratic curve fit for South Carolina projects.**

10.3.3 Calibration Verification

A mid-level calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples or 12 hours, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

The daily CCV analysis at a concentration other than the mid level (to meet NELAC requirements) will consist of all of the congeners of interest. All other CCVs will be mid level calibration standards.

Calibration Controls	Sequence	Control Limit
Initial Calibration Standards	5 pt. Curve (see Table A-3) prior to samples	< 20% RSD (alternatively, if the correlation coefficient is >0.99, linear regression may be used).
Second Source Verification	After initial calibration	± 20% Difference of expected value
Cont. Calib. Verif. (CCV)	After initial calibration, Every 20 samples	± 20% Difference*
Retention Time Windows	After initial calibration, update daily	3 X Std. Deviation
Note: If the CCV is > ± 20 % for any one compound, data will be acceptable if the result is J flagged. The result must be less than the RL.		

10.3.4 Procedure

Refer to Section 10 of the main body of this SOP for general procedural requirements.

If one surrogate is out of control in a sample and all surrogates are in control for the method blank and LCS, then matrix effect has been demonstrated for the sample and repreparation is not necessary. The client may be contacted for input if the re-extraction is expected to take place after the sample holding time has been exceeded. Refer to section 9.14 for corrective action.

10.3.5 Extraction

The extraction procedure is described in SOP No. PT-OP-001.

10.3.6 Cleanup

Cleanup procedures are described in SOP No. PT-OP-001.

10.3.7 Suggested gas chromatographic conditions are given in Table A-2.

10.3.8 Allow extracts to warm to ambient temperature before injection.

10.3.9 The suggested analytical sequence is given in Table A-5.

11.0 CALCULATIONS / DATA REDUCTION

11.1 Identification of Congeners

11.2 Retention time windows are used for identification of PCB congeners. Second column confirmation must be performed.

11.3 Congeners 90 and 101 coelute on both of the GC columns. Currently the ICV mix includes congener 90 and 101 but the calibration standards do not include congener 90. This does not affect any sample results. Future ICV mix will be purchased without congener 90.

11.4 Surrogate recovery results are calculated and reported for TCMX and BZ-205. Corrective action is only necessary if BZ-205 and TCMX are both outside of acceptance limits.

12.0 METHOD PERFORMANCE

12.1 Refer to section 12.3 for initial demonstration of capability requirements under Section 12 of the main body of this SOP. The LCS spiking level used for IDOC.

12.2 Method detection limits (MDL) are determined for congeners as per SOP PT-QA-007.

13.0 POLLUTION CONTROL

13.1 Refer to Section 13 of the main body of this SOP.

14.0 WASTE MANAGEMENT

14.1 Refer to Section 14 of the main body of this SOP.

15.0 REFERENCES

15.1 Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatograph, Revision 1, February, 2007, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.2 Method 8000B, Determinative Chromatographic Separations, Revision 2, December, 1996, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.3 Department of Defense, Quality System manual (QSM) for Environmental Laboratories, Final Version 3.0, Jan 2006.

15.4 SOP PT-QA-001, Employee Orientation and Training, current version.

15.5 SOP CA-Q-S-002, Manual Integration, current version.

15.6 SOP PT-QA-007, Determination of Method Detection Limits (MDLs), current version.

15.7 SOP PT-QA-016, Nonconformance & Corrective Action System, current version.

15.8 SOP PT-QA-018, Technical Data Review Requirements, current version.

15.9 SOP PT-QA-021, Quality Control Program, current version.

15.10 PT-LQAM, Pittsburgh Laboratory Quality Assurance Manual, current version.

16.0 ATTACHMENTS

16.1 Table A-1: Standard Analyte List and Reporting Limits

16.2 Table A-2: Recommended GC Operating Conditions

16.3 Table A-3: Calibration Levels

16.4 Table A-4: LCS/Matrix Spike and Surrogate Spike Levels

16.5 Table A-5: Suggested Analytical Sequence

16.6 Table A-6: Surrogate Recovery Limits

17.0 REVISION HISTORY

17.1 Revision 2

17.1.1 Updated the CCV %D from 15 to 20% in the table in section 10.3.3 of this Appendix.

17.1.2 Removed Congeners from Table A-1 that are no longer supported in TA Pittsburgh. Also removed High Level Soil information from this Table as per Larry Matko, TA Pittsburgh’s Technical Director.

17.1.3 Cannot use quadratic curve for SC work.

17.1.4 Volumes were corrected in Table A-1 concerning assumed concentration factors used in calculating Reporting Limits. Also the information for the High Level Soil was removed as per Larry Matko, TA Pittsburgh’s Technical Director.

17.1.5 Removed Congeners from Table A-3 that are no longer supported in TA Pittsburgh, as per Larry Matko, TA Pittsburgh’s Technical Director.

17.1.6 Spike concentrations were corrected in Table A-4 for the LCS/ Matrix Spike and Surrogate spiking levels for Congener Analysis.

17.1.7 Table A-5 was corrected to reflect current practice for Congener Analysis.

17.1.8 The comments listed under Table A-6 were corrected to reflect current practice for Congener Analysis.

17.2 Revision 3

17.2.1 Added section 7.1, Reagents to list the solvents and gases used in Congener analysis.

17.2.2 Added section 7.2, Standards to explain how congener and surrogate standards, from stock to working solutions, are made (purchased, in the case of the stock standard).

17.2.3 In section 10.3.1, added Acceptance Criteria and Corrective Action for the second source ICV standard; also added text to explain how the ICV working standard is prepared.

17.2.4 Added to section 11.3: Congeners 90 and 101 coelute on both of the GC columns. Currently the ICV mix includes congener 90 and 101 but the calibration standards do not include congener 90. This does not affect any sample results. Future ICV mix will be purchased without congener 90.

17.2.5 Updated the Congener RL’s found in Table A-1 to current TALS reporting limits. Also combined soil/tissue/sediment into one group in Table A-1 and added wipe to the chart following Table A-1 and 1 wipe as the extraction volume.

17.2.6 In Table A-2 updated the GC operating conditions to reflect current settings.

17.2.7 In Table A-3 updated the surrogate concentrations in each of the calibration levels to the current levels used.

17.2.8 In Table A-4 combined soil/sediment/tissue into one column and added a column for wipes. Also corrected the surrogate spike level in Table A-4 to the correct concentration levels for each matrix.

17.2.9 Removed Table A-6 “Preparation of Calibration Standards” from the document and from section 16.6 since it was not needed and changed old Table A-7 “Surrogate Recovery Limits” to new Table A-6.

17.2.10 Removed DoD references from Appendix A, lab is not ceritifed for PCB Cogeners for DoD.

18.0 METHOD MODIFICATIONS

18.1 Refer to Section 18 of the main body of this SOP.

Table A-1
Standard Analyte List and Reporting Limits

		Reporting Limit			
Compound *	**CAS #**	**Water ng/L**	**Soil/Sediment/ Tissue ug/kg**	**Wipe ng/wipe**	**Waste ug/kg**
BZ-8	34883-43-7	1.0	1.0	1.0	10
BZ-18	37680-65-2	1.0	1.0	1.0	10
BZ-28	7012-37-5	1.0	1.0	1.0	10
BZ-44	41464-39-5	1.0	1.0	1.0	10
BZ-49	41464-40-8	1.0	1.0	1.0	10
BZ-52	35693-99-3	1.0	1.0	1.0	10
BZ-66	32598-10-0	1.0	1.0	1.0	10
BZ-77	32598-13-3	1.0	1.0	1.0	10
BZ-87	38380-02-8	1.0	1.0	1.0	10
BZ-101	37680-73-2	1.0	1.0	1.0	10
BZ-105	32598-14-4	1.0	1.0	1.0	10
BZ-118	31508-00-6	1.0	1.0	1.0	10
BZ-126	57465-28-8	1.0	1.0	1.0	10
BZ-128	38380-07-3	1.0	1.0	1.0	10
BZ-138	35065-28-2	1.0	1.0	1.0	10
BZ-153	35065-27-1	1.0	1.0	1.0	10
BZ-156	38380-08-4	1.0	1.0	1.0	10
BZ-169	32774-16-6	1.0	1.0	1.0	10
BZ-170	35065-30-6	1.0	1.0	1.0	10
BZ-180	35065-29-3	1.0	1.0	1.0	10
BZ-183	52663-69-1	1.0	1.0	1.0	10

Table A-1 Standard Analyte List and Reporting Limits					
		Reporting Limit			
Compound *	**CAS #**	**Water ng/L**	**Soil/Sediment/ Tissue ug/kg**	**Wipe ng/wipe**	**Waste ug/kg**
BZ-184	74472-48-3	1.0	1.0	1.0	10
BZ-187	52663-68-0	1.0	1.0	1.0	10
BZ-195	52663-78-2	1.0	1.0	1.0	10
BZ-206	40186-72-9	1.0	1.0	1.0	10
BZ-209	2051-24-3	1.0	1.0	1.0	10

* The congener identifications are consistent with the short-hand identifications recommended by Ballschmiter and Zell (1980).

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	**Final Volume**	**Dilution Factor**
Groundwater	1000 mL	2 mL	1
Soil/Tissue/Sediment	10 g	20 mL	1
Waste	1 g	20 mL	1
Wipe	1 wipe	2 mL	1

Table A-2

Parameter	Recommended Conditions
Injection port temp	225°C
Detector temp	325°C
Temperature program	80°C ramping 30°C/min to 190°C, then 20°C/min to 230°C, then 4°C/min to 270°C and finally 20°C/min to 300°C holding for 5 minutes (22 minute run-time)
Column 1	ZB 50, 30 m, 0.25 mm id, 0.25 µm FT
Column 2	ZB 1701, 30 m, 0.25 mm id, 0.25 µm FT
Injection	1-2µL
Carrier gas	Hydrogen
Make up gas	Nitrogen

Table A-3

Calibration Levels ug/mL

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
BZ-8	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-18	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-28	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-44	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-49	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-52	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-66	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-77	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-87	0.0005	0.001	0.0025	0.0050	0.010	0.020

Table A-3

Calibration Levels ug/mL

BZ-101	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-105	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-118	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-126	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-128	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-138	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-153	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-156	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-169	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-170	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-180	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-183	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-184	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-187	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-195	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-206	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-209	0.0005	0.001	0.0025	0.0050	0.010	0.020

SURROGATES

TCMX	0.000825	0.00165	0.004125	0.00825	0.0165	0.033
BZ-205	0.0005	0.001	0.0025	0.0050	0.010	0.020

Table A-4

LCS/Matrix Spike and Surrogate Spike Levels for Congener Analysis

ng/L, ng/wipe or ug/kg

Compound	Aqueous	Soil/Sediment/Tissue	Wipe	Waste
BZ-8	20	20	20	200
BZ-18	20	20	20	200
BZ-28	20	20	20	200
BZ-44	20	20	20	200
BZ-49	20	20	20	200
BZ-52	20	20	20	200
BZ-66	20	20	20	200
BZ-77	20	20	20	200
BZ-87	20	20	20	200
BZ-101	20	20	20	200
BZ-105	20	20	20	200
BZ-118	20	20	20	200
BZ-126	20	20	20	200
BZ-128	20	20	20	200
BZ-138	20	20	20	200
BZ-153	20	20	20	200
BZ-156	20	20	20	200
BZ-169	20	20	20	200
BZ-170	20	20	20	200
BZ-180	20	20	20	200
BZ-183	20	20	20	200
BZ-184	20	20	20	200

Table A-4				
LCS/Matrix Spike and Surrogate Spike Levels for Congener Analysis				
ng/L, ng/wipe or ug/kg				
BZ-187	20	20	20	200
BZ-195	20	20	20	200
BZ-206	20	20	20	200
BZ-209	20	20	20	200
Surrogates				
TCMX	5	5	5	50
BZ-205	5	5	5	50

Table A-5

Suggested Analytical Sequence

Initial Calibration	
Injection #	
Solvent blank (optional)	
PCB Congener Mix 1	6-point
ICV (second source standard(s) of all congeners of interest)	
Samples 1-20 (or 12 hours)	
Solvent blank (optional)	
PCB Congener Mix 1	Level 3
After 12 hours:	
PCB Congener Mix 1	Level 3
Samples 1-20 (or 12 hours)	
Solvent blank (optional)	
PCB Congener Mix 1	Level 3
12 hour Calibration	

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated.

Table A-6
Surrogate Recovery Limits

Surrogate	Water Recovery Limits	Soil Recovery Limits
PCB 205(BZ)	35-140	35-140
Tetrachloro-m-xylene	35-140	35-140

NOTE: The Surrogate Recovery Limits are subject to change as in-house control limits are evaluated and updated.

Title: Extractable Residue (Lipids) from Animal Tissue

SOP # PT-OP-011 (Extractable Residue – Lipids)

Approvals (Signature/Date):

Sharon Bacha Organics Department Manager	3/4/2014 Date	Steve Jackson Regional Safety Coordinator	11/8/2013 Date
Violet Fanning Quality Assurance Manager	3/2/2014 Date	Deborah L. Lowe Laboratory Director	11/26/2013 Date
Roseann Ruyechan Inorganics Department Manager	3/4/2014 Date		

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1.0 Scope and Application

1.1 This SOP describes the procedure for determining organic solvent extractable residue from fish tissue. Normally this residue is predominantly lipid material from the tissue, but it may include other non-polar material as well (e.g. petroleum hydrocarbons).

1.2 This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above.

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following as indicated PT-QA-M-001, Quality Assurance Manual.

2.0 Summary of Method

2.1 A 10-gram aliquot of homogenized tissue is extracted via soxtherm. The extract is dryed and evaporated to dryness. The residue remaining after evaporation is determined gravimetrically. TestAmerica Pittsburgh is certified to perform this procedure and it is referenced on the scope of accreditation as SOP (00416) OP-011.

3.0 Definitions

3.1 Refer to the glossary in the Laboratory Quality Assurance Manual (PT-QA-M-001), current version.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, 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 of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP and should not be used.

5.3 Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection.

5.4 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6 Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.7 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor and/or the EH&S coordinator.

6.0 Equipment and Supplies

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1 Syringe or positive displacement pipette: 1 mL

6.2 Analytical balance, capable of accurately weighing ± 0.0001 g

6.3 Toploader Balance: >100 g capacity, accurate ± 0.1 g

6.4 Soxtherm Model S 306A

6.5 Soxtherm thimbles

6.6 Horizon SpeedVap 9000II

6.7 70 ml Disposable Evaporation Pan.

7.0 Reagents and Standards

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard.

7.1 Reagents

7.1.1 Methylene chloride, pesticide grade or equivalent

7.1.2 Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.

7.1.3 Fish Oil (Sigma): purchased commercially.

7.2 Standards

7.2.1 Not applicable

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 The tissue samples are stored frozen and are to be extracted within 1 year of sample collection.

8.2 The extracts are stored at ambient temperature and analyzed within forty (40) days of extraction.

9.0 Quality Control

9.1 The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 per preparation batch[1]	< RL
Laboratory Control Sample (LCS)	1 per preparation batch[1]	30-150%
Laboratory Control Sample Duplicate (LCSD)	1 per preparation batch[1]	30-150%; ± 25% RPD
Sample Duplicate (SD)	1 per preparation batch[1]	± 25% D

[1] A batch is limited to 20 samples.

[2] Control Limits are maintained in TALS LIMS.

9.2 Method Blank

9.2.1 The method blank is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2 Corrective Action for Blanks

9.2.2.1 If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data must be addressed in the project narrative.

9.2.2.2 If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the project narrative.

9.3 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD): The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.1 Corrective Action for LCS

9.3.1.1 The LCS/LCSD recoveries must be evaluated against in-house control limits. If the results are outside established control limits, the system is out of control and corrective action must occur.

9.3.1.2 Corrective action will include repreparation and reanalysis of the batch unless the client agrees that another corrective action is acceptable. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data must be addressed in the project narrative.

9.4 Surrogates

9.4.1 Not applicable.

9.5 Duplicates

9.5.1 Sample duplicates are performed at a frequency of one per analytical batch of up to 20 samples.

9.6 Nonconformance and Corrective Action

9.7 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10.0 Procedure

10.1 Calibration and Standardization

10.1.1 Not Applicable

10.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.3 Lipid Extraction Procedure (Organic Prep Department)

10.3.1 Weigh 10.0 g of the homogenized tissue into a soxtherm thimble. Record the weight to the nearest 0. 1 g in the TALS worksheet. Add sodium sulfate until no free liquid is present in sample. **The method blank consists of 10.0 grams of sodium sulfate. The LCS/LCSD consists of 10.0 grams of sodium sulfate and spiked with 1.0 g of Fish Oil (Section 7.1.3).**

NOTE: If less than 10.0 g of homogenized tissue is used for the extraction, record the actual weight that is extracted and record the Final Volume as 10 mL in the TALS worksheet so that the prep factor will be properly applied to the RL.

10.3.2 Add 120 mL of methylene chloride.

10.3.3 Extract at 150°C for approximately 2 hours.

10.3.4 Remove from soxtherm and cool.

10.3.5 Transfer to a pre-weighed 40-mL VOA vial.

10.4 Lipid Analytical Procedure (Wet Chemistry Department)

10.4.1 Calibrate the analytical balance at 2 mg and 1000 mg using class “S” weights before and after each batch of samples.

10.4.2 Calibration shall be within ± 10% (i.e. ± 0.2 mg) at 2 mg and ± 0.5% (i.e. ± 5 mg) at 1000 mg. If values are not within these limits, recalibrate the balance.

10.4.3 Weigh Disposable Evaporation Pans using an analytical balance, record the weights, and put pans aside to be used later.

10.4.4 Place a phase separation filter paper into a filter funnel. Add approximately 5-10 grams of sodium sulfate on top of the filter paper. Rinse with methylene chloride and dispose of the rinsate.

10.4.5 Filter the extract through the sodium sulfate and phase separator filter paper into a pre-weighed 70 mL evaporation pan. Rinse the collection flask, sodium sulfate and filter paper twice with approximately 5 mL portions of methylene chloride.

10.4.6 Place pans into the SpeedVap and evaporate to dryness. The SpeedVap is set to 50° C.

10.4.7 Place the pans in a desiccator for one hour.

10.4.8 Weigh the pans on an analytical balance and record the weights to the nearest 0.1 mg.

10.4.9 Determine the Lipid concentration using the equation in section 11.

10.5 Analytical Documentation

10.5.1 Record all information in the TALS LIMS worksheet for Percent Lipid extraction and analysis. See example Percent Lipids Extraction and Analysis Worksheets in the Attachments at the end of the SOP.

11.0 Calculations / Data Reduction

$$Concentration(\%) = \frac{(A - B)}{W} \times 100$$

Where:

A = Weight of beaker + residue, g

B = Weight of the beaker, g

W = Weight of sample extracted, g

12.0 Method Performance

12.1 The supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use, and has the required experience.

12.2 **Demonstration of Capabilities** – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3 Method Detection Limit Study – MDLs are not performed for this procedure.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.2 This method does not contain any specific modifications that serve to minimize or prevent pollution.

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1 Extracted solid samples contaminated with methylene chloride. This waste is collected in waste container identified as "Lab Trash Waste", Waste #12.

14.1.2 Used sodium sulfate contaminated with methylene chloride from the extract drying step. This waste is collected in waste container identified as "Lab Trash Waste", Waste #12.

14.1.3 Assorted flammable solvent waste from various glassware rinses. This waste is collected in waste containers identified as "Mix Flammable Solvent Waste", Waste #3.

14.1.4 Methylene chloride waste from various glassware rinses. This waste is collected in waste containers identified as “Methylene Chloride Waste”, Waste #2.

14.1.5 Miscellaneous disposable glassware contaminated with solvents and sample residue. This waste is collected in waste container identified as “Lab Trash Waste”, Waste #12.

15.0 References / Cross-References

15.1 TestAmerica Laboratory Quality Assurance Manual (PT-QA-M-001), current version.

15.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Method 8290 Sections 6.7, 7.2.2, 7.3.3, and Method 3550.

15.1.2 United States Army Corps of Engineers Waterways Experiment Station. May 1995. A Comparison of Three Lipid Extraction Methods. Technical Note EEDP-01-35. 3909 Halls Ferry Road, Vicksburg, Mississippi 39180-6199.

15.1.3 TestAmerica SOP No. PT-OP-001, Extraction and Cleanup of Organic Compounds from Waters and Soils, SW846 3500-series and 3600-series, and EPA 600-series methods, current version.

15.1.4 SOP PT-OP-011, Extractable Residue (Lipids) from Animal Tissue, current version. Referenced as SOP (00416) OP-011 on our PADEP Scope of Accreditation.

16.0 Method Modifications:

16.1 Not applicable

17.0 Attachments

17.1 Example Percent Lipids TALS Extraction Worksheet.

17.2 Example Percent Lipids TALS Analytical Worksheet.

18.0 Revision History

18.1 Revision 4, 08/18/09

18.2 Revision 5, 3/4/2014

18.2.1 On the cover page replaced Brian Pino with Sharon Bacha, Organics Department Manager; replaced Nasreen DeRubeis with Violet Fanning as QAM; replaced Larry Matko with Deb Lowe as Lab Director; updated Steve Jackson’s title to Regional Safety Coordinator; added Roseann Ruyechan, Inorganics Department Manager to the cover page.

18.2.2 Changed the text in section 1.3 to match the text found in the SOP Checklist concerning client requested method modifications.

18.2.3 In sections 1.3 and 15.1 changed PT-LQAM to PT-QA-M-001 as the SOP number for the QA Manual.

18.2.4 In section 5.1 removed the Radiation Safety Manual since does not pertain to this facility; in section 5.4 changed MSDS to SDS to comply with industry standards.

18.2.5 Under sections 6 and 7 added the equivalency text from the SOP Checklist in regards to equipment, supplies, reagents and standards.

18.2.6 Added the following equipment and supplies: Horizon SpeedVap 9000II as section 6.6 and 70 ml disposable evaporation pan as section 6.7.

18.2.7 Added the Quality Control information into a table format and removed text in section 9 contained in this Table; removed sections 9.2 and 9.2.1 for batch definition since it can be found in the QA Manual.

18.2.8 Removed section 10.2 since method modifications are addressed in section 1.3.

18.2.9 Noted that section 10.3 is the Lipid Extraction Procedure performed by Organic Prep; added a NOTE: under section 10.3.1 to discuss how to properly record initial and final volumes so that prep factors will properly apply to the RL; noted in section 10.3.3 that the lipids are extracted for approximately 2 hours; added section 10.4 Lipid Analytical Procedure and sections 10.4.1 through 10.4.9 to discuss the steps in the lipid analysis.

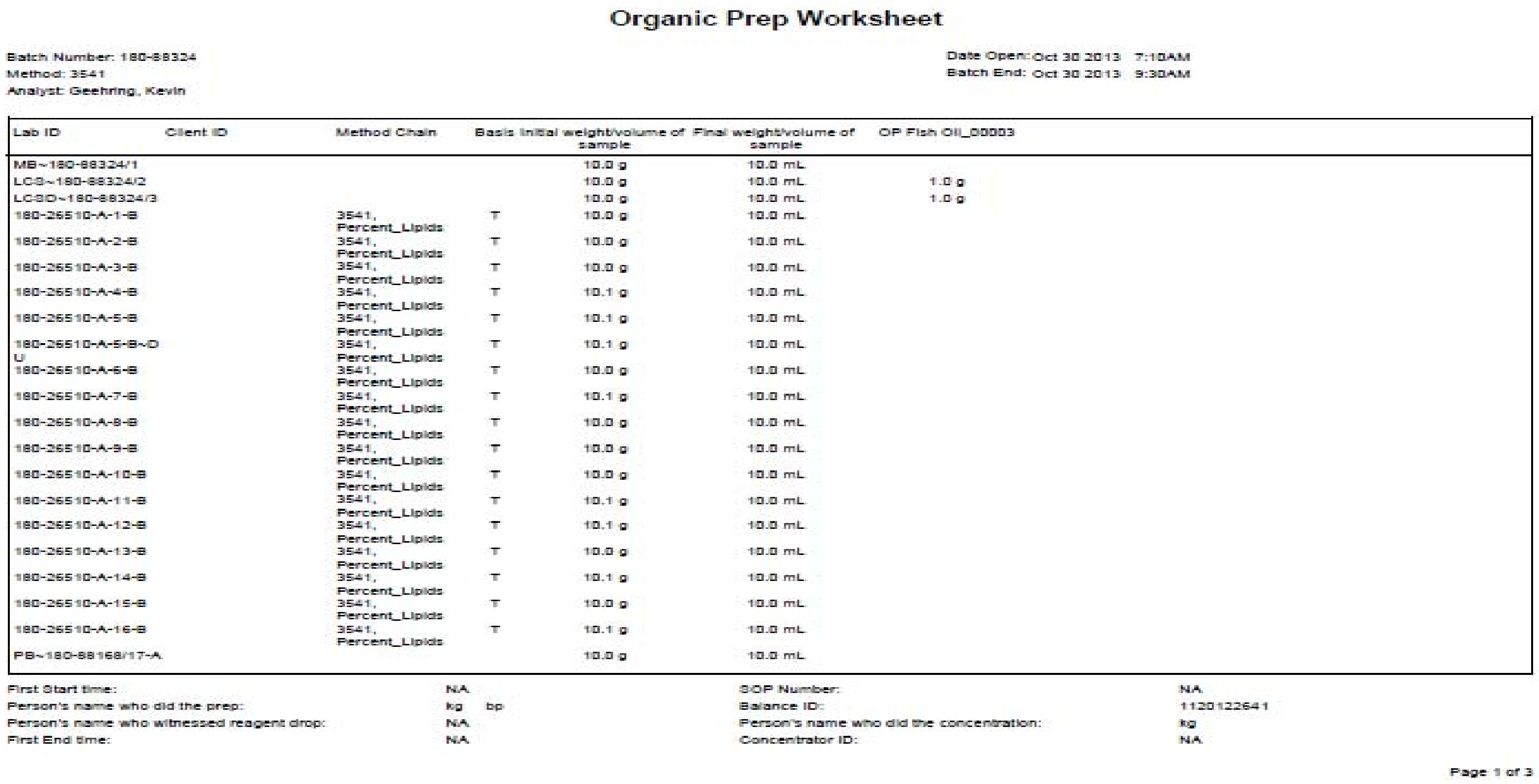
18.2.10 In section 10.5, noted that prep information is entered directly into TALS LIMS and referred to an example TALS Worksheet at the end of the SOP.

18.2.11 In section 12.1 changed group/team leader to supervisor; added section 12.2 for DOC’s and section 12.3 concerning MDL Study.

18.2.12 In section 17.1 updated the Attachment title to “Example Percent Lipids TALS Extraction Worksheet”; added section 17.2 Example Percent Lipids TALS Analytical Worksheet. And added tan example in the Attachments at the end of the SOP.

ATTACHMENTS

Example Percent Lipids TALS Extraction Worksheet



Organic Prep Worksheet

Batch Number: 180-88324
Method: 3541
Analyst: Geehring, Kevin

Date Open: Oct 30 2013 7:10AM
Batch End: Oct 30 2013 9:30AM

Lab ID	Client ID	Method Chain	Basis	Initial weight/volume of sample	Final weight/volume of sample	OP Fish Oil_00003
MB~180-88324/1				10.0 g	10.0 mL	
LCS~180-88324/2				10.0 g	10.0 mL	1.0 g
LCSD~180-88324/3				10.0 g	10.0 mL	1.0 g
180-26510-A-1-B		3541, Percent_Lipids	T	10.0 g	10.0 mL	
180-26510-A-2-B		3541, Percent_Lipids	T	10.0 g	10.0 mL	
180-26510-A-3-B		3541, Percent_Lipids	T	10.0 g	10.0 mL	
180-26510-A-4-B		3541, Percent_Lipids	T	10.1 g	10.0 mL	
180-26510-A-5-B		3541, Percent_Lipids	T	10.1 g	10.0 mL	
180-26510-A-5-B~DU		3541, Percent_Lipids	T	10.1 g	10.0 mL	
180-26510-A-6-B		3541, Percent_Lipids	T	10.0 g	10.0 mL	
180-26510-A-7-B		3541, Percent_Lipids	T	10.1 g	10.0 mL	
180-26510-A-8-B		3541, Percent_Lipids	T	10.0 g	10.0 mL	
180-26510-A-9-B		3541, Percent_Lipids	T	10.0 g	10.0 mL	
180-26510-A-10-B		3541, Percent_Lipids	T	10.0 g	10.0 mL	
180-26510-A-11-B		3541, Percent_Lipids	T	10.1 g	10.0 mL	
180-26510-A-12-B		3541, Percent_Lipids	T	10.1 g	10.0 mL	
180-26510-A-13-B		3541, Percent_Lipids	T	10.0 g	10.0 mL	
180-26510-A-14-B		3541, Percent_Lipids	T	10.1 g	10.0 mL	
180-26510-A-15-B		3541, Percent_Lipids	T	10.0 g	10.0 mL	
180-26510-A-16-B		3541, Percent_Lipids	T	10.1 g	10.0 mL	
PB~180-88168/17-A				10.0 g	10.0 mL	

First Start time:	NA	SOP Number:	NA
Person's name who did the prep:	kg bp	Balance ID:	1120122641
Person's name who witnessed reagent drop:	NA	Person's name who did the concentration:	kg
First End time:	NA	Concentrator ID:	NA

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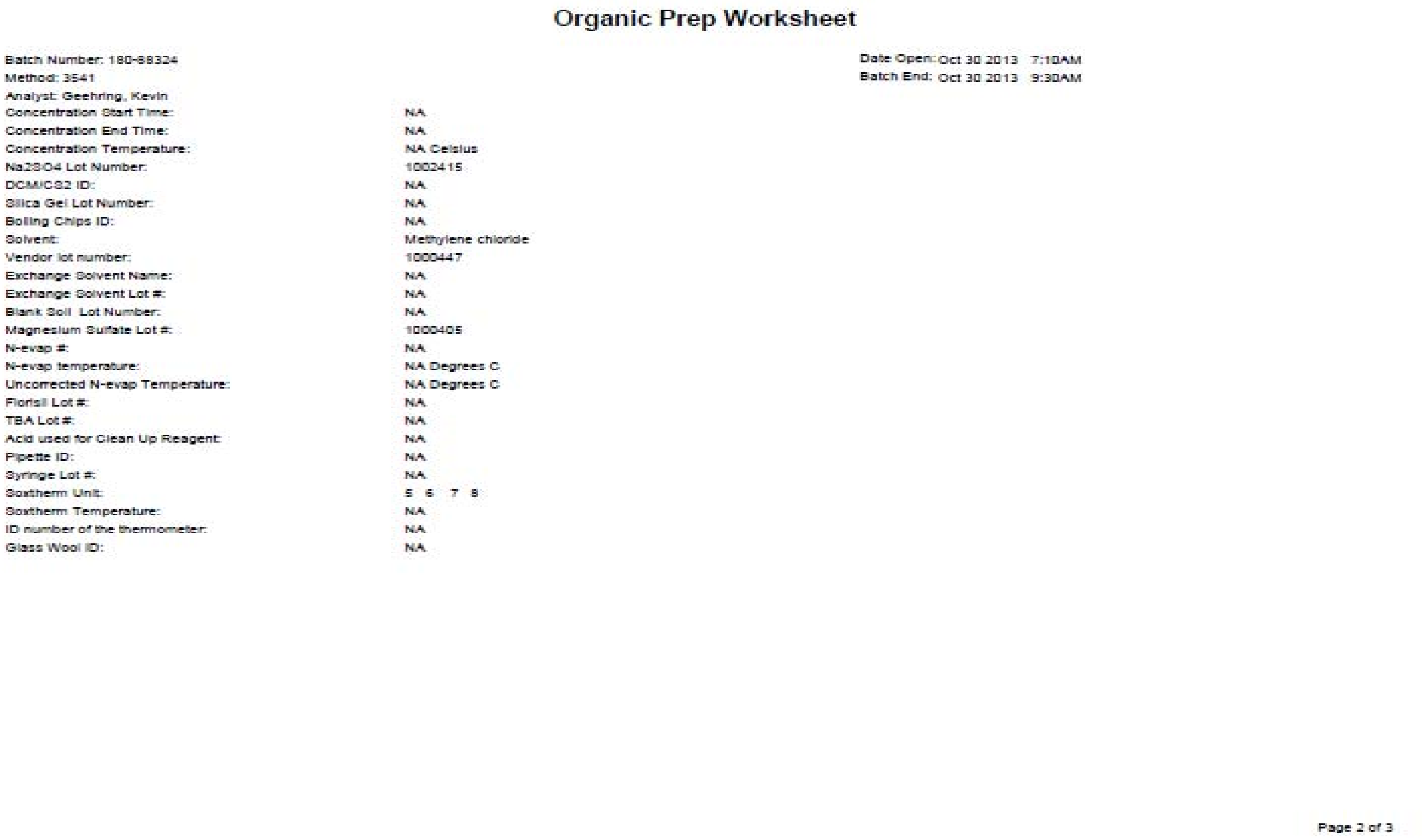

Organic Prep Worksheet

Batch Number: 180-88324
Method: 3541
Analyst: Geehring, Kevin

Date Open: Oct 30 2013 7:10AM
Batch End: Oct 30 2013 9:30AM

Concentration Start Time:	NA
Concentration End Time:	NA
Concentration Temperature:	NA Celsius
Na2SO4 Lot Number:	1002415
DCM/CS2 ID:	NA
Silica Gel Lot Number:	NA
Boiling Chips ID:	NA
Solvent:	Methylene chloride
Vendor lot number:	1000447
Exchange Solvent Name:	NA
Exchange Solvent Lot #:	NA
Blank Soil Lot Number:	NA
Magnesium Sulfate Lot #:	1000405
N-evap #:	NA
N-evap temperature:	NA Degrees C
Uncorrected N-evap Temperature:	NA Degrees C
Florisil Lot #:	NA
TBA Lot #:	NA
Acid used for Clean Up Reagent:	NA
Pipette ID:	NA
Syringe Lot #:	NA
Soxtherm Unit:	5 6 7 8
Soxtherm Temperature:	NA
ID number of the thermometer:	NA
Glass Wool ID:	NA

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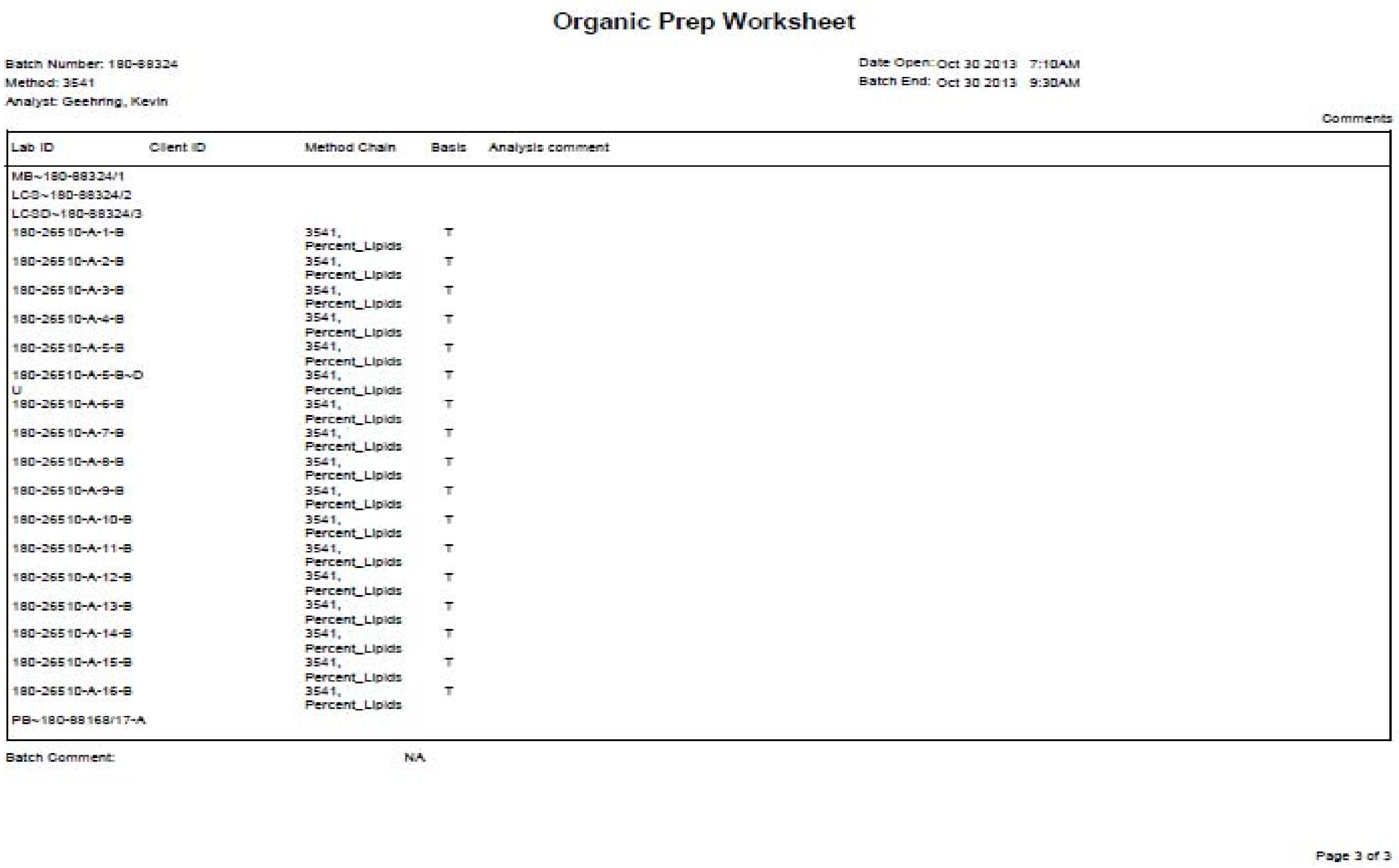


Organic Prep Worksheet

Batch Number: 180-88324
Method: 3541
Analyst: Geehring, Kevin

Date Open: Oct 30 2013 7:10AM
Batch End: Oct 30 2013 9:30AM

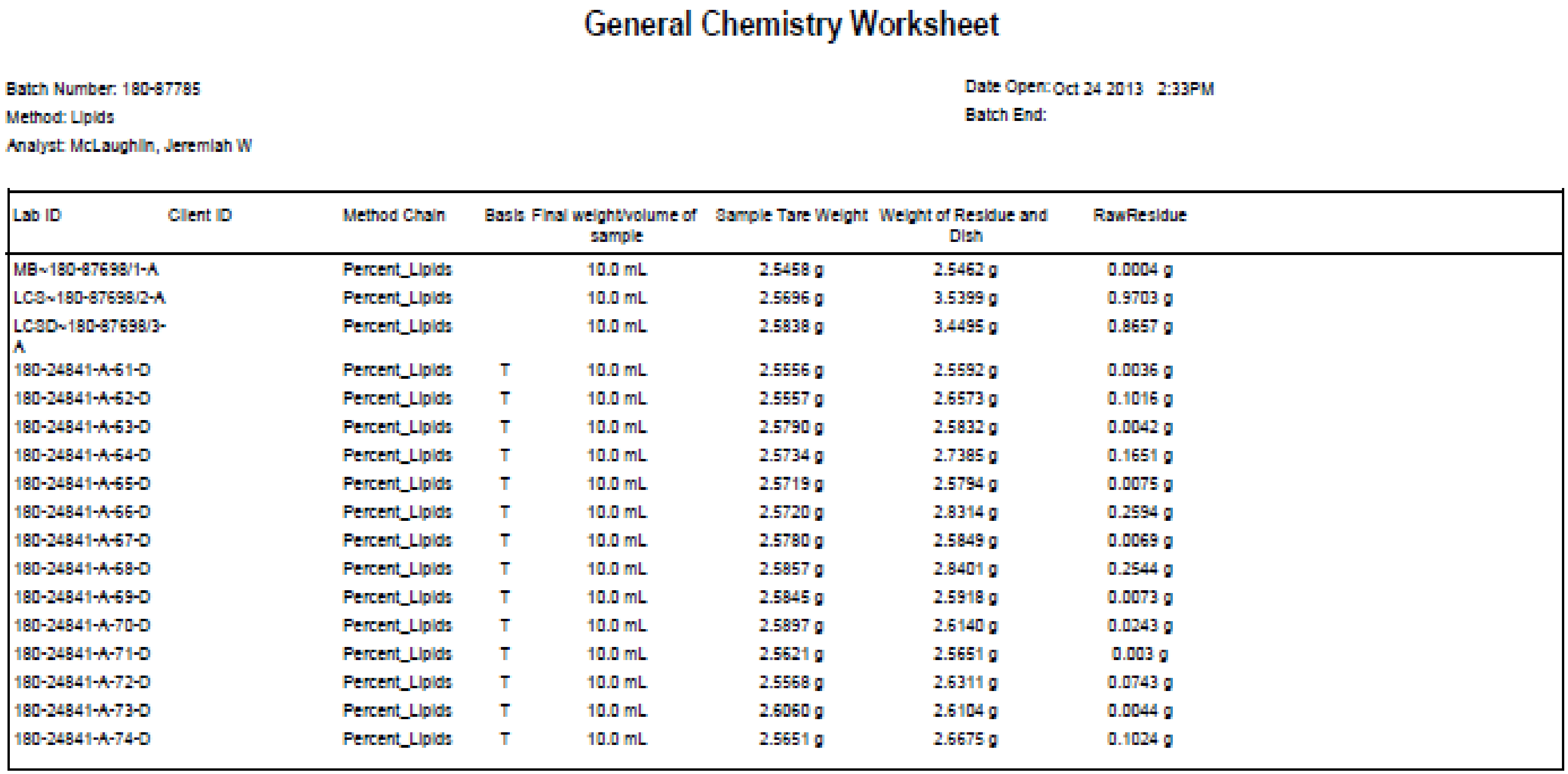
Comments

Lab ID	Client ID	Method Chain	Basis	Analysis comment
MB~180-88324/1				
LCS~180-88324/2				
LCSD~180-88324/3				
180-26510-A-1-B		3541, Percent_Lipids	T	
180-26510-A-2-B		3541, Percent_Lipids	T	
180-26510-A-3-B		3541, Percent_Lipids	T	
180-26510-A-4-B		3541, Percent_Lipids	T	
180-26510-A-5-B		3541, Percent_Lipids	T	
180-26510-A-5-B~DU		3541, Percent_Lipids	T	
180-26510-A-6-B		3541, Percent_Lipids	T	
180-26510-A-7-B		3541, Percent_Lipids	T	
180-26510-A-8-B		3541, Percent_Lipids	T	
180-26510-A-9-B		3541, Percent_Lipids	T	
180-26510-A-10-B		3541, Percent_Lipids	T	
180-26510-A-11-B		3541, Percent_Lipids	T	
180-26510-A-12-B		3541, Percent_Lipids	T	
180-26510-A-13-B		3541, Percent_Lipids	T	
180-26510-A-14-B		3541, Percent_Lipids	T	
180-26510-A-15-B		3541, Percent_Lipids	T	
180-26510-A-16-B		3541, Percent_Lipids	T	
PB~180-88168/17-A				

Batch Comment: NA

Page 3 of 3

Example Percent Lipids TALS Analytical Worksheet.



General Chemistry Worksheet

Batch Number: 180-87785
Method: Lipids
Analyst: McLaughlin, Jeremiah W

Date Open: Oct 24 2013 2:33PM
Batch End:

Lab ID	Client ID	Method Chain	Basis	Final weight/volume of sample	Sample Tare Weight	Weight of Residue and Dish	RawResidue
MB~180-87698/1-A		Percent_Lipids		10.0 mL	2.5458 g	2.5462 g	0.0004 g
LCS~180-87698/2-A		Percent_Lipids		10.0 mL	2.5696 g	3.5399 g	0.9703 g
LCSD~180-87698/3-A		Percent_Lipids		10.0 mL	2.5838 g	3.4495 g	0.8657 g
180-24841-A-61-D		Percent_Lipids	T	10.0 mL	2.5556 g	2.5592 g	0.0036 g
180-24841-A-62-D		Percent_Lipids	T	10.0 mL	2.5557 g	2.6573 g	0.1016 g
180-24841-A-63-D		Percent_Lipids	T	10.0 mL	2.5790 g	2.5832 g	0.0042 g
180-24841-A-64-D		Percent_Lipids	T	10.0 mL	2.5734 g	2.7385 g	0.1651 g
180-24841-A-65-D		Percent_Lipids	T	10.0 mL	2.5719 g	2.5794 g	0.0075 g
180-24841-A-66-D		Percent_Lipids	T	10.0 mL	2.5720 g	2.8314 g	0.2594 g
180-24841-A-67-D		Percent_Lipids	T	10.0 mL	2.5780 g	2.5849 g	0.0069 g
180-24841-A-68-D		Percent_Lipids	T	10.0 mL	2.5857 g	2.8401 g	0.2544 g
180-24841-A-69-D		Percent_Lipids	T	10.0 mL	2.5845 g	2.5918 g	0.0073 g
180-24841-A-70-D		Percent_Lipids	T	10.0 mL	2.5897 g	2.6140 g	0.0243 g
180-24841-A-71-D		Percent_Lipids	T	10.0 mL	2.5621 g	2.5651 g	0.003 g
180-24841-A-72-D		Percent_Lipids	T	10.0 mL	2.5568 g	2.6311 g	0.0743 g
180-24841-A-73-D		Percent_Lipids	T	10.0 mL	2.6060 g	2.6104 g	0.0044 g
180-24841-A-74-D		Percent_Lipids	T	10.0 mL	2.5651 g	2.6675 g	0.1024 g

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Pittsburgh
SOP Change Form
PT-QA-W-023 R0
Effective Date: 01/09/2013

SOP # with Revision PT-WC-020 R6
Title: Percent Moisture, Ash, Organic Matter and Total Solids in Soil Samples
Methods ; SM 2540G and ASTM D2974-07a

Effective Date of Amendment: 2-13-2013

CONTROLLED DISTRIBUTION

COPY # : 002

ISSUED TO : QA Webpage

Revision Number with Mod ID: _____

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the <u>front</u> of the SOP copy.***

1. Briefly explain the reason for the update.
Updated the SOP to reflect the analysis of duplicate samples 1in10 instead of 1in20 for a PADEP Audit Finding (RM 5,6. AND 7).

2. Category for SOP Change:

☐ Typographical Corrections (Non-Technical) – Retraining Not Required.

☐ Typographical Corrections (Technical- define below) – Retraining is required.

☒ Procedural Changes (Define Below) – Retraining Required.

3. Summary of Procedure Change (circle to indicate if there are attachments to this form: [✓ No] / Yes: # pages attached = ___)
UPDATED section 9.1 of SOP PT-WC-020 R6 to denote duplicate samples will be analyzed 1in10 per source method SM 2540G requirement.
Does this change result in a Method Modification? Yes [✓ No]
If yes, please explain below. Modification section of SOP must be corrected upon full revision.______

2-13-13
Approval Signature/Date: Technical

2-13-13
Approval Signature/Date: QA Manager or Designee

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Pittsburgh
SOP Change Form
PT-QA-W-023 R0
Effective Date: 01/09/2013

SOP # with Revision PT-WC-020 R6
Title: Percent Moisture, Ash, Organic Matter and Total Solids in Soil Samples
Methods ; SM 2540G and ASTM D2974-67a

Effective Date of Amendment:

CONTROLLED DISTRIBUTION

COPY # : 002

ISSUED TO : QA Webpage

Revision Number with Mod ID: _____

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Approval Signature/Date: Technical

2-13-13
Approval Signature/Date: QA Manager or Designee

Title: Percent Moisture, Ash, Organic Matter and Total Solids in Soil Samples

Methods: SM 2540G and ASTM D2974-07a

Approvals (Signature/Date):

Michael P. Wesoloski	1/19/ 2012		1/16/2012
Michael Wesoloski Technical Manager	Date	Steve Jackson Health & Safety Manager / Coordinator	Date
Nasreen K. DeRubeis	1/14/2012		1/21/2012
Nasreen DeRubeis Quality Assurance Manager	Date	Deborah L. Lowe Laboratory Director	Date

Copyright Information:

1.0 Scope and Application

1.1 This method is applicable to the determination of the moisture content of soil, rock and soil-aggregate mixtures by weight. This method is also used to determine the ash content in oil, liquid (non-aqueous, i.e. sludges) and petroleum samples.

2.0 Summary of Method

2.1 A homogenous sample is dried at 103°C to 105°C (to determine moisture), and 500 ± 50°C(to determine Ash). The difference in the weight loss of the sample represents the moisture content. The Ash content can also be determined by use of the muffle furnace.

2.2 Percent organic matter is equal to 100% - the % ash. Therefore the % organic matter is defined as the percentage of combustible material found in the sample based on dry weight.

2.3 For DoD QSM 4.1/4.2 Requirements, refer to SOP PT-QA-029.

3.0 Definitions

3.1 SD: Sample Duplicate is a replicate aliquot of an environmental sample taken from the same sample container, when possible, and processed with the first aliquot of the sample. The sample and SD result are compared to determine the effect of the sample matrix on the precision of the analytical process.

3.2 QC Batch: The QC batch is a set of 20 or fewer environmental samples plus associated laboratory QC samples that are similar in composition and that are processed within the same time period with the same reagent and standard lots. Refer to SOP PT-QA-021 for further definitions.

3.3 Percent organic matter is the percentage of combustible material found in the sample based on dry weight.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Non-homogeneous samples may give erratic results.

5.0 Safety

NOTE: The text pertaining to the Radiation Safety Manual only applies to operations that have rad licenses and should only be included for these labs. Other labs may delete the reference to this manual.

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, 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 of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 None

5.2 Primary Materials Used:

5.2.1 There are no materials used in this method that have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. 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.

{VITON gloves may be worn when halogenated solvents are used for extraction or sample preparation, nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.]}

5.3 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor and/or the EHSC.

6.0 Equipment and Supplies

6.1 Analytical balance: capable of accurately weighing ± 0.0001 g or 0.1 mg.

6.2 Drying Oven, 103°C ± 2°C

6.3 Desiccators

6.4 Evaporating dishes, various

6.5 Wooden Spatula

6.6 Top loading balance: capable of accurately weighing ± 0.01 g

6.7 Aluminum pans

6.8 Muffle furnace, 550°C ± 50°C

7.0 Reagents and Standards

7.1 Not Applicable.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples are not chemically preserved.

8.2 Samples are stored in plastic or glass containers at ≤ 6 °C (0-6 °C).

8.3 There is no recommended holding time.

9.0 Quality Control

9.1 A duplicate sample is analyzed with every set of 10 or fewer samples. The acceptance criteria is calculated as relative percent difference (RPD) between the original and duplicate samples analysis. The acceptable range between the sample and sample duplicate is RPD less than or equal to 20 percent. If the RPD is outside criteria, the analyst will check calculations and instrument performance (balance used), reanalyze the samples once, evaluate results, and, if necessary, narrate it when reporting the data. The duplicate samples are not counted as part of the samples in the QC batch. For further definitions refer to SOP PT-QA-021. For DoD requirements refer to SOP PT-QA-029.

10.0 Procedure

10.1 Calibration and Standardization

10.2 Since this method is based on gravimetric techniques, there is no calibration in the usual sense. Proper balance operation is verified daily or prior to sample analysis.

10.3 Oven temperature must be checked daily and recorded in the oven temperature log

10.4 Total Solids – 2540G

10.5 Preparation of evaporating dish: If volatile solids are to be measured, ignite a clean evaporating dish at 500± 50°C for one hour in a muffle furnace. Cool in desiccator and store in desiccator until ready for use.

10.5.1 Preparation of sample: Rocks, stones, twigs, leaves, or other foreign matter which interfere with homogenizing the sample must be carefully removed and the remaining sample mixed thoroughly so that a representative sample will be obtained. Where it has been necessary to remove artifacts, the action taken and artifacts removed from the sample must be adequately described by the analyst in a narrative provided with the sample data. If there is doubt concerning the proper handling of sample artifacts (due to the nature of the particular sample or project), the laboratory supervisor and project manager must determine the procedure to be followed, and the resulting actions must be documented in a narrative provided with the sample data. For volatile solid samples and samples analyzed for percent dry solids, supernatant liquids are mixed into the sample.

10.5.2 Place weighing dish plus 5 - 10g sample in a drying oven maintained at 103°C ± 2°C. Sample handling and drying must be conducted in a well ventilated area.

10.5.3 Dry the sample overnight (12 to 24 hours), but no longer than 24 hours. Remove the sample from the oven and cool in a dessicator. If dried less than 12 hours, it must be documented that constant weight was attained. In the event it is necessary to demonstrate the attainment of constant weight, data must be recorded for a minimum of two repetitive weight/dry/desiccator/weight cycles with a minimum of one hour drying time in each cycle. Constant weight would be defined as a loss in weight of no greater than 0.01g between start weight and final weight of last cycle.

10.6 Ash (Fixed) and Volatile Solids:

10.6.1 Transfer the dried residue from Section 10.5.3 to a muffle furnace, 500± 50°C and ignite for one hour. (If the residue contains large amounts of organic matter, first ignite it over a gas burner and under a fume hood in the presence of adequate air to lessen losses due to reducing conditions and to avoid odors in the laboratory.) Cool in desiccator to balance temperature and weigh.

10.7 Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

10.8 Organic matter is determined by subtracting percent ash content from one hundred.

11.0 Calculations / Data Reduction

11.1 Percent Total Solids:

$$\frac{(A - B)}{(C - B)} \times 100$$

Percent Volatile Solids:

$$\frac{(A - D)}{(A - B)} \times 100$$

Percent Ash (Fixed) Solids:

$$\frac{(D - B)}{(A - B)} \times 100$$

Where:

A = Weight of dried residue and dish, mg

B = Weight of dish

C = Weight of wet sample and dish, mg

D = Weight of residue and dish after ignition, mg

11.2 Percent Moisture

%Moisture = 100% - % Total Solids

11.2.1 Determine the amount of organic matter by difference as follows:

Organic matter, % = 100.0 - D

D = weight percentage of ash

11.2.2 Report all results to the nearest 0.1 percent. Report furnace temperature used for ash content determinations.

11.3 Duplicate Sample, Relative Percent Difference (RPD):

$$\text{RPD} = \frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2}\right)} \times 100$$

Where:

X_1 = Original Result

X_2 = Duplicate Result

12.0 Method Performance

12.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. The group/team leader must document the training and PE performance and submit the results to the QA Manager for inclusion in associate training files.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.2 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention.

13.3 This method does not contain any specific modifications that serve to minimize or prevent pollution.

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1 Used Soil Samples – This waste is collected in containers identified as “Lab Trash”, Waste #12.

15.0 References / Cross-References

15.1 Method 1311, Section 7.1.2.2, SW-846, September 1994.

15.2 CLP SOW for Inorganics Analysis ILMO4.0, Section IV, Part F.

15.3 Standard Methods for the Examination of Water and Waste Water, 2011 or Standard Methods Online; Methods: 2540G.

15.4 ASTM Standard D2974-07a, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat Materials. Volume 4.08.

15.5 ASTM Standard D2216-05, Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass.

15.6 PT-QA-001, Employee Orientation and Training.

15.7 PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability.

15.8 PT-QA-016, Nonconformance & Corrective Action System.

15.9 PT-QA-018, Technical Data Review Requirements.

15.10 PT-QA-021, Quality Assurance Program.

15.11 PT-QA-022, Equipment Maintenance.

15.12 PT-QA-024, Subsampling.

15.13 PT-QA-027, Sample Receiving and Chain of Custody.

15.14 PT-QA-029, QA/QC Requirements for DoD QSM Version 4.1.

15.15 PT-LQAM, Pittsburgh Laboratory Quality Assurance Manual, current version

16.0 Method Modifications:

16.1 N/A

17.0 Attachments

17.1 All sample preparation and analysis information will be documented electronically in TALS LIMS. All the documents associated with an analysis will be electronically available for inclusion in the final report.

17.2 Organic Matter Worksheet

18.0 Revision History

18.1 Revision 4, 11/7/08:

18.1.1 Updated to TA SOP format. Changed STL with TA. Updated Safety, Method Performance, Pollution Control and Waste Management sections. Added an example Organic Matter Worksheet.

18.2 Revision 5:

18.2.1 Annual Review performed. Added references to DoD QSM 4.1 requirements, SOP PT-QA-029. Section 8.2 changed sample storage requirements to ≤ 6 °C (0-6 °C). Added QA SOP references.

18.3 Revision 6

18.3.1 On the cover page, replaced Larry Matko with Deb Lowe as the Lab Director. Updated the ASTM Standard Method number from D2974-84 to D2974-07a on the cover page and in section 15.4.

18.3.2 In section 1.1, added an example of a non-aqueous liquid as sludge.

18.3.3 Added reference to QSM 4.2 in section 2.3.

18.3.4 Removed section 3.3 since Reagent Water is not used in this SOP.

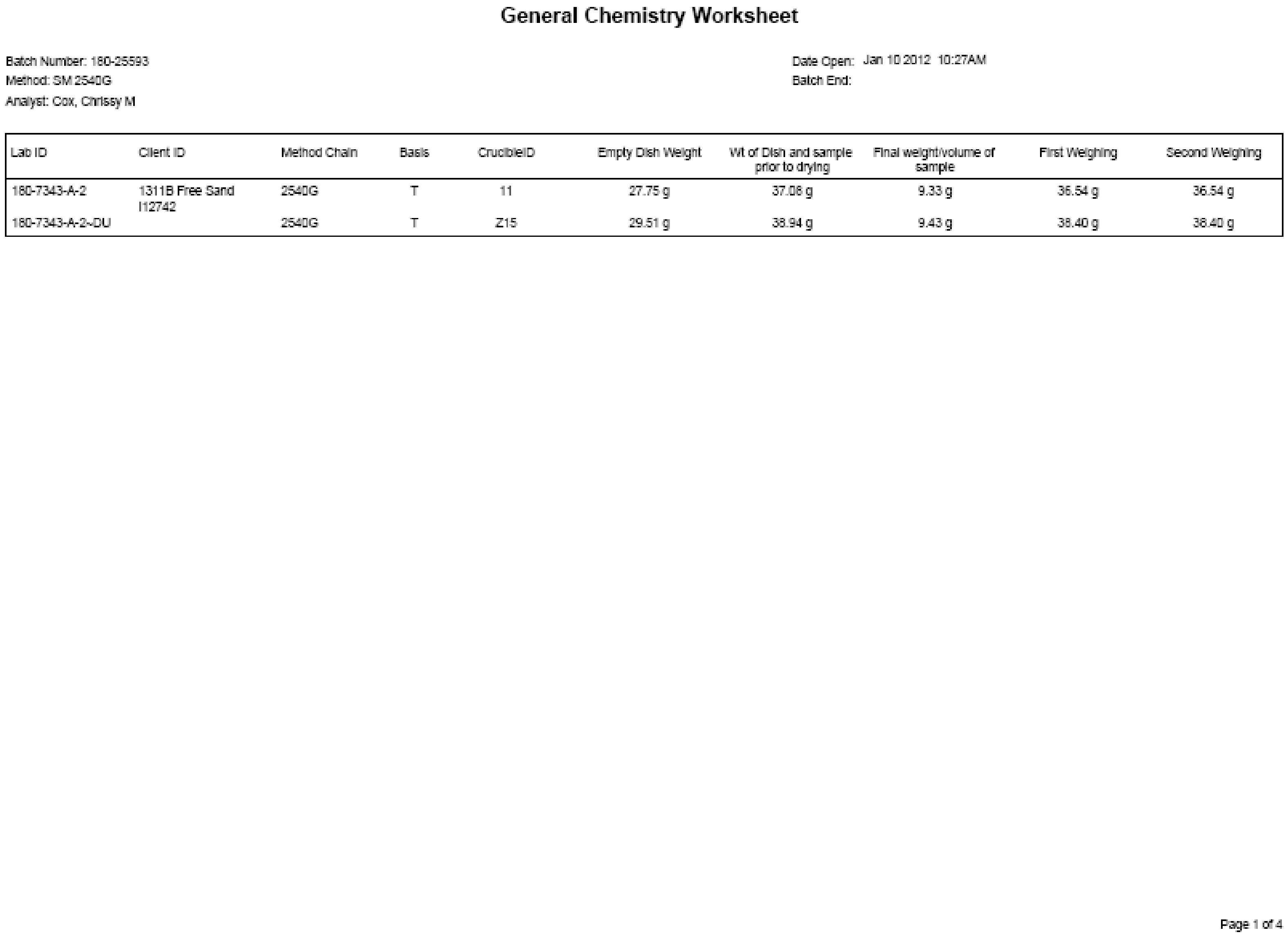
18.3.5 In section 6.5, replaced Tongue Blades with Wooden Spatulas.

18.3.6 Updated section 17.1 to show that all information for this SOP is now electronically stored and retrievable from TALS LIMS.

18.3.7 In section 17.2 replaced the old Organic Matter Worksheet with an example of the TALS Electronic Worksheet.

18.3.8 In section 15.3, the reference year of source method SM 2540G was updated from 1998 to 2011.

18.3.9 In section 9.1, corrected the 1 in 20 duplicate analysis to 1 in 10 as per SM2450G.



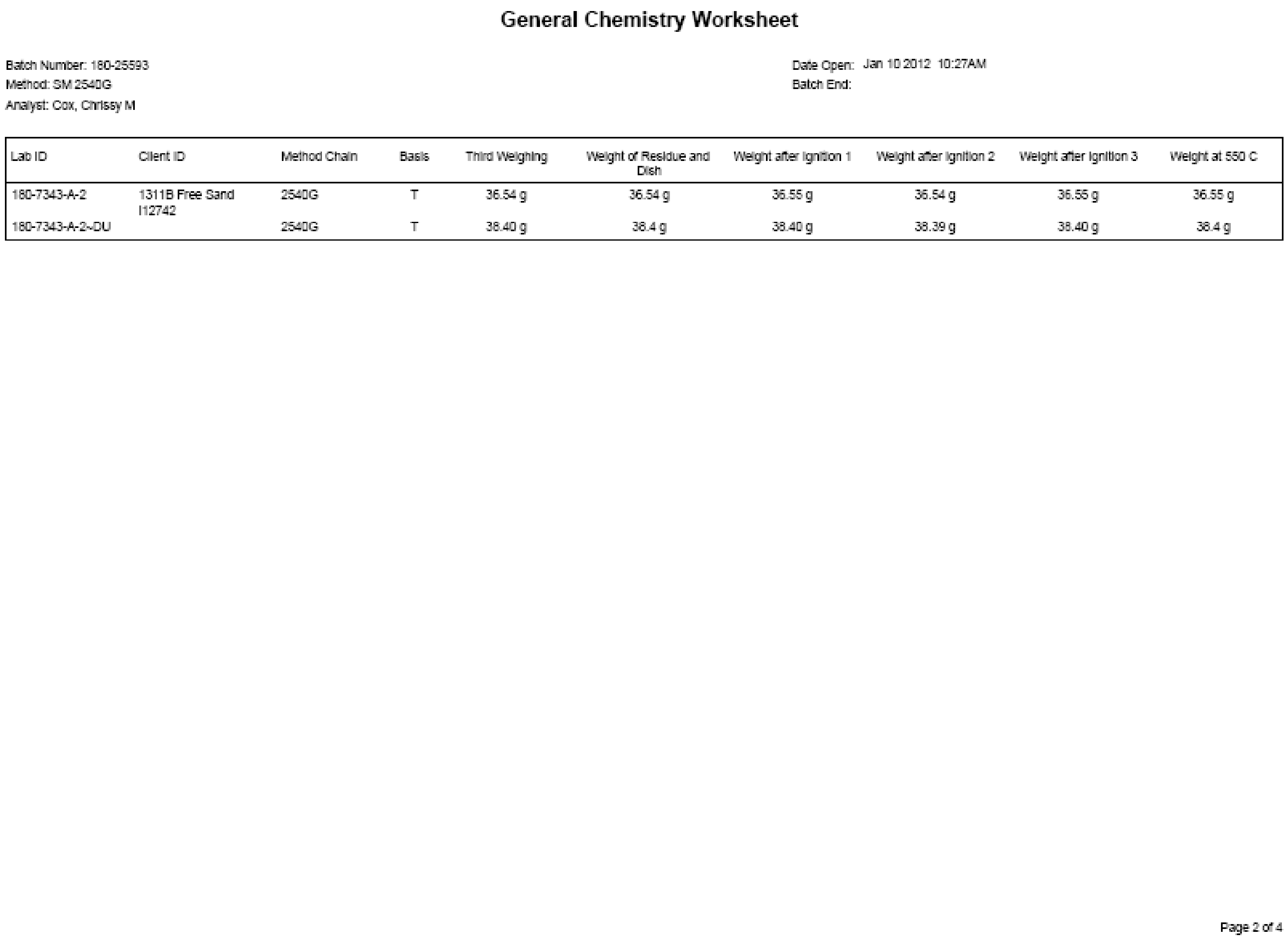
General Chemistry Worksheet

Batch Number: 180-25593
Method: SM 2540G
Analyst: Cox, Chrissy M

Date Open: Jan 10 2012 10:27AM
Batch End:

Lab ID	Client ID	Method Chain	Basis	CrucibleID	Empty Dish Weight	Wt of Dish and sample prior to drying	Final weight/volume of sample	First Weighing	Second Weighing
180-7343-A-2	1311B Free Sand I12742	2540G	T	11	27.75 g	37.08 g	9.33 g	36.54 g	36.54 g
180-7343-A-2-DU		2540G	T	Z15	29.51 g	38.94 g	9.43 g	38.40 g	38.40 g

Page 1 of 4



General Chemistry Worksheet

Batch Number: 180-25593
Method: SM 2540G
Analyst: Cox, Chrissy M

Date Open: Jan 10 2012 10:27AM
Batch End:

Lab ID	Client ID	Method Chain	Basis	Third Weighing	Weight of Residue and Dish	Weight after Ignition 1	Weight after Ignition 2	Weight after Ignition 3	Weight at 550 C
180-7343-A-2	1311B Free Sand I12742	2540G	T	36.54 g	36.54 g	36.55 g	36.54 g	36.55 g	36.55 g
180-7343-A-2-DU		2540G	T	38.40 g	38.4 g	38.40 g	38.39 g	38.40 g	38.4 g

Page 2 of 4

General Chemistry Worksheet

Batch Number: 180-25593
Method: SM 2540G
Analyst: Cox, Chrissy M

Date Open: Jan 10 2012 10:27AM
Batch End:

Lab ID	Client ID	Method Chain	Basis	Calculation Message
180-7343-A-2	1311B Free Sand I12742	2540G	T	OK
180-7343-A-2-DU		2540G	T	OK

Oven ID:	2/3
Oven, Bath or Block Temperature 1:	104/550 Celsius
Perform Calculation (0=No, 1=Yes):	1

General Chemistry Worksheet

Batch Number: 180-25593
Method: SM 2540G
Analyst: Cox, Chrissy M

Date Open: Jan 10 2012 10:27AM
Batch End:

Comments

Lab ID	Client ID	Method Chain	Basis	Analysis comment
180-7343-A-2	1311B Free Sand I12742	2540G	T	
180-7343-A-2-DU		2540G	T	

Batch Comment: oven 2- 10:32 1/10/12 - 08:44 1/11, oven 3- 08:55-09:55 0 1/11/12

ATTACHMENT D

INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT
STANDARD OPERATING PROCEDURES FOR FISH COLLECTIONS, USE OF SEINES, ELECTROFISHERS, AND SAMPLE PROCESSING

(43 Sheets)

STANDARD OPERATING PROCEDURES

FOR FISH COLLECTION, USE OF SEINES, ELECTROFISHERS, AND SAMPLE PROCESSING

BIOLOGICAL STUDIES SECTION
SURVEILLANCE AND STANDARDS BRANCH
OFFICE OF WATER MANAGEMENT
INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT

STANDARD OPERATING PROCEDURES FOR FISH COLLECTION, USE OF SEINES, ELECTROFISHERS, AND SAMPLE PROCESSING

I. Fish Collection Procedures

    A. Seining

    B. Procedures

II. Electrofishing

    A. Overview

    B. Safety in Electrofishing

    C. Field Check Lists

        1. Boat, Trailer, Boat Engine

        2. Generator/Variable Pulse Unit

        3. Ancillary Equipment

        4. Personnel/Crew Members

    D. Variable Voltage Pulsators Operation

        1. Coffelt Model VVP-2E

        2. Smith- Root Gas Powered Pulsator (GPP 2.5)

    E. Electrofishing Procedure

    F. Generator Maintenance

II. Preservation and Storage of Fish Samples

    A. Preservation/Storage

    B. Log Book/Field Sheets

    C. Data Entry

- D. Sample Shipment

III. Appendices

- A. Fish and Sediment Site Information Field Sheets
- B. Summary of Fish Collection Data Sheet
- C. Rapid Bioassessment Protocol V Fish Community Assessment Field Sheet
- D. Contract Laboratory Chain-of-Custody SMPLELOG

FISH COLLECTION PROCEDURES

Fish collection by Water Management personnel is usually done either by seining or by electrofishing. Seining is most often done in small, relatively shallow streams, while electrofishing is done in the larger streams, rivers, and lakes.

I. SEINING

The seine is a 1/4 -inch nylon mesh, flat, vertical net usually four feet high and of varying lengths. Sturdy vertical poles are attached to each end of the seine at the top and bottom as supports for the seine and as hand-holds. The seine has lead weights along the bottom (lead line) to keep that portion on the substrate and floats along the top (float line) to keep it floating on the surface.

Procedure

1. One person holds a pole at each end of the seine and they walk forward moving the net through the water to capture fish.

2. The seine should not be stretched tight, but kept slack so a "bag" develops in the net as it is moved through the water. Fish will be collected in this "bag."

3. Seining usually works best when the seine is moved in an upstream direction.

4. Another person, or the people holding the seine, may agitate the bottom ahead of the seine to scare fish into the net.

5. After pulling the seine a short distance (25 to 50 feet), it should be lifted and the fish collected.

6. Lifting the seine is accomplished in one of two ways:

   A) The lead line is moved ahead of the float line, being sure to keep the "bag" in the seine, and then by turning the poles to a horizontal position, the seine is lifted from the water in midstream taking care to not allow the top of the net to go underwater.

   B) The other method is to work the seine toward shore and again, keeping the "bag" in the seine, move the lead line of the seine ahead of the float line and slide the seine up onto the shore. This latter method usually works best, and results in the loss of fewer fish if the shoreline is suitable for it.

7. Fish are then removed from the net for processing and release. Fish that cannot be identified in the field are preserved in a jar with 10% formalin (See Preservation And Storage Of Fish Samples, SOP(*) page 496 solution and returned to the

Biological Studies Laboratory for identification.

II. ELECTROFISHING

Electrofishing is the primary technique used by Water Management personnel to collect fish for quantitative and qualitative community studies, and for fish flesh contaminant analysis. The Biological Studies Section of the Office of Water Management has set up guidelines for safety of personnel in the field. However thorough understanding of an electrofishing setup, basic electricity principles, and potential environmental hazards is of paramount importance in maximizing the safety of all personnel involved in an electrofishing exercise. The following references must be thoroughly reviewed before a personnel participates in electrofishing exercises.

Cowx, I.G. and P. Lamarque. 1990. Fishing With Electricity. Applications In Freshwater Fisheries Management. Fishing News Books. Cambridge, MA 248pp.

Reynolds, J.B. 1983. Electrofishing. in: Fisheries Techniques. ed L.A. Nielsen and D.L. Johnson. American Fisheries Society Publ.

Novotny, D.W. and G.R.Priegel. 1974. Electrofishing Boats, Inproved Designs and Operational Guidelines To Increase The Effectiveness of Boom Shockers. Technical Bulletin #73. Wisconsin Department of Natural Resources. Madison, WI 49pp.

Goodchild, G.A. 1991. European Inland Fisheries Advisory Commission Code Of Practices And Guidelines For Safety With Electric Fishing. EIFAC Occasional Paper. No. 24 Rome. FAO 16pp.

These and Other references on electrofishing are available for review (see SOP(*) Biological Studies Section Literature and Taxonomic Source Literature Section page 1214). This literature is cited in the S.O.P. Literature Section. It is also important that the electrofishing personnel be aware of general physiologic affects of electricity on fish, differences in response to electricity by different fish species, habitat limitations as well as water quality limitations. All of these factors play an important role in affecting electrofishing efficiency and bias minimization when looking at fish communities.

The equipment used by IDEM-OWM-Biological Studies personnel for electrofishing utilizes pulsed-DC current. This is the most efficient current form for electrofishing and also elicits the least harm to fish when applied in a knowledgeable manner. Pulsed-DC is modified DC giving a unidirectional current with periodic interruption resulting in square waves (pulses) of voltage. Pulsed duty cycles (percent of time that power is on in a cycle) appear to have secondary effects. Settings at 25% and 50% give similar results hence 25% is preferable.

Conductivity and temperature of the water (as well as the fish) affects current in a straight line relationship. As the water temperature increases conductivity increases at an equivalent rate. When conductivity of the water gets too low (<100useimen/cm^3) there is a decrease in current at constant voltage. To increase the current to an affective level one must increase the applied voltage or increase the electrode size. Conversely high conductivity waters would require lower applied voltage and/or less electrode exposure (size) to achieve the desired current range. Conductivities greater than 500useimen/cm^3 can cause generator overloads unless electrode size is reduced. The electrofishing team must be aware of this when electrofishing in waters of extreme conductivities from normal range.

Fish responses are not the same for all species. However a voltage gradient of 0.1-1.0 volt/cm is most effective for stunning fish and this gradient range can be maintained in freshwater of normal conductivity (100-500useimen/cm^3) by adjusting the circuit voltage to produce an electrode current of 3-6 amperes.

The following are some practical suggestions that will aid in successful electrofishing. These are based on the principles of basic electricity and optimal system design.

1. Adjust the voltage to conform with the water absolute conductivity to insure maximum current density in the stun zone.

2. Operate at the highest current that produces maximum effectiveness.

3. Larger electrodes and a lower applied voltage is superior.

4. Personnel should be familiar with the waterbody and its morphology.

5. Check all equipment and safety checks before going out to electrofish.

6. Operate boats at very slow speed.

7. Concentrate efforts on available habitat cover and sample a variety of habitat types if possible.

8. Take care of your equipment as if it were your own. Your life could depend on it.

Safety In Electrofishing

Safety precautions necessary for safe electrofishing cannot be overemphasized. Even low currents passing through the body can cause serious injury. Death from electrocution can result from ventricular fibrillation (uncontrolled asynchronous contraction of the ventricular muscle fibers of the heart), respiratory arrest (resulting from current passing through the respiratory center), and asphyxia (caused by sustained contraction of the chest muscles). In addition other injuries can occur indirectly during electrofishing such as scrapes, bruises, falls, or drowning. Electrical shock can also cause burns at the point of electrode contact. In order to assure safe operation of the electrofishing boat or tote barge system, personnel must adhere strictly to safety precautions and procedures.

Operational safety procedures include:

1. All electrofishing personnel are required to wear a personal floatation device (type I, II, or III) while electrofishing, plus one approved type IV throwable P.F.D. should be on board the boat.

2. All electrofishing personnel are required to wear electrofishing safety gloves.

3. All electrofishing personnel are required to wear rubber boots. Hip or calf height rubber boots are usually worn while on board the boat, and chest waders are worn for wadable stream situations.

4. All electrofishing personnel are required to have Standard First Aid training and certification maintained.

5. All electrofishing personnel are required to have CPR (cardiopulmonary resuscitation) training and certification maintained.

6. All personnel operating a boat should be trained in boating safety and be familiar with Indiana Boating Regulations SOP(*) page 74.

7. One person will be in charge for each electrofishing effort (crew leader).

8. Dipnets used for electrofishing are made only of nonconducting material for the handle such as fiberglass or epoxiglass.

9. Keep and maintain a first-aid kit on board of the boat, or in the vehicle when tote barge electrofishing.

10. Always keep a cellular phone on-hand, either in the boat or in the truck on shore.

11. Keep a U.S. Coast Guard approved fire extinguisher (at least a B-1 type) on board of the boat and within reach of the boat operator. Inspect this each time before going out.

12. Stow all gear out of the way and keep the boat in a neat, orderly condition.

13. Never electrofish alone. At IDEM we fish in groups of three.

14. Never electrofish if any of the personnel feels exhausted.

15. All team members should constantly be aware of all of the other team members' positions.

16. Do not fish under inclement weather conditions.

17. Use proper lifting technique for carrying equipment to and from the shore or boat.

18. All personnel should be familiar with the fundamentals of basic electricity, and the correct procedures for operating electric fishing equipment.

19. Electrofishing should be done slowly and deliberately.

20. Make equipment and electrical line checks for condition, integrity, and grounding before <u>every</u> electrofishing effort.

21. Never bypass or use dummy loads on foot actuated switches.

22. Stop the generator before changing fuses, removing or inserting plugs, refueling, or making <u>any</u> changes in the electrical system or electrodes.

23. Twelve volt battery systems are also capable of producing fatal currents. Do not treat these circuits carelessly.

24. Do not operate any electrofishing system close to other boats or near a shoreline where people or animals are located.

25. Never place hands or conducting objects into the water near the boat while the <u>generator</u> is running.

26. Know where all power switches are located on the electrofishing system.

FIELD CHECK LIST:

Boat, Trailer, and Boat Engine

__Inspect trailer wheels for proper inflation and wheel bearing play. Don't forget the spare. Repack trailer wheel bearings at the beginning of each field season. A grease gun is used to maintain the grease in the bearing cups during the field season.

__Be sure to check the oil level in the lower housing unit of the outboard motor periodically.

__Make sure all components inside the boat and the boat itself is secured.

__Inspect trailer lights for breaking, turn signals and running lights.

__Watch for leaks in the boat engine fuel line.

__Have an adequate boat engine fuel supply prepared for the boat and generator for the start of the trip. All of the outboard boat engines require a fuel and oil mixture of 50:1.

__ Extra prop for engine being used.

Generator/Variable Pulse Unit

__Electrical connections secure and protected

__Mountings secure

__Exhaust directed away from operator

__Frame properly grounded

__Unit electrically bonded/connected to frame

__Oil topped up (Do Not overfill oil level)

__Fuel topped up (generator engines do not require a fuel to oil mixture). Use regular unleaded gasoline

__Controls and gauges operational

__hv (high voltage) checks done

__Adequate mechanical protection of wiring

__Adequate connectors and interlocking

__Audible tone generator working (if equipped)

__hv ON light working (if equipped)

__Operators foot switch(s) working (boats)

__Kill switch(s) working

__Anode switch(s) working

__Wiring to anode in good condition

__Anode in good condition-attached securely (boat)

__No screens or nets attached to anode hoop (tote barge)

__Anode handles of non-conductive material (tote barge)

__Cathode plate clean-connection secure

__Anode cables untangled-connections tight

__Watch for leaks in the generator engine fuel line

Ancillary Equipment

--Fire extinguisher-fully charged-correct type

--First aid kit present-fully stocked

--Fuel containers-regulation style

__Dip net handles made of non-conductive material

__Boat lights working

__Communication gear working (if applicable)

__Tool Box with tools, flashlight, extra spark plugs for boat engine, etc.

__Volt/Ohm meter

__Voltage gradient/absolute conductivity meter

Personnel/Crew Members

__Each crew member briefed on boat and system operation

__Minimum number of crew trained in CPR, first-aid and basic electronics present

__Each crew member wearing rubber safety gloves

__Each crew member wearing rubber boots, or waders

__Each crew member wearing P.F.D.

__Protective hearing gear is provided to each crew member

__Safety procedures covered

__In some situations a hard hat and safety goggles may be necessary

Variable Voltage Pulsators

1) Coffelt model VVP-2E

This variable pulsator is used on both the 16 foot jon boat ("Jaws") and the Waterman boat ("Coffelt boat"). We have 2 units of this pulsator model. Either unit may be used on either boat. However, there are two different harness sets (one for each unit) to be used on "Jaws." All wiring on the "Coffelt boat" is built in. The unit is placed under the Captain's console where the plugs are located. Wire harness bags consist of the power cord that goes from the generator to the vvp unit, the foot actuated safety switches, and the anode/cathode connecting wires. A wire harness set is needed when using "Jaws." Documentation is found in SOP(*) page__.

SPECIFICATIONS

CONDUCTIVITY RANGE:	10 to 2500useimen per cubic centimeter
INPUT VOLTAGE:	120 or 240 volts AC 60 hertz single phase
OUTPUT VOLTAGES:	AC: 0-720 volts RMS in 120 volt steps, DC: 0-1,020 volts peak in 170 volt steps
OUTPUT WAVE FORMS:	AC: sine wave, DC: fast rise, slow decay
DC PULSE FREQUENCY:	60, 120, or a variable slow channel from 2-40.
DC PULSE WIDTH:	Adjustable 1 to 7 milliseconds
INPUT-OUTPUT CONNECTIONS:	MS type environmentally sealed
OUTPUT POWER:	AC: 5000 watts max. at 720 volts and max pulse width. DC: 5000 watts max. at 1,020 volts and max. pulse width
OUTPUT CURRENT:	10 amperes maximum
MONITORS:	Electrode current meter, Digital timer registers actual shocking time in seconds, Input power pilot lamp
SAFETY INDICATORS:	Electrodes on and off lights
SAFETY SWITCHES:	Two remote foot switches (both must be pressed simultaneously), One panel mounted emergency shut down switch
ISOLATION:	Electrode circuits completely isolated from generator input
CIRCUIT PROTECTION:	Input: Magnetic hydraulic circuit breaker, Output: An automatic electronic circuit breaker
CONSTRUCTION:	Heavy-duty welded aluminum cabinet with splash proof lid and illuminated control panel
DIMENSIONS:	17" X 12" X 9"
ACCESSORIES:	Unit supplied with input power and electrode (anode and cathode) interconnecting cables and two remote safety switches

The electro-shocker model VVP-2E is designed to supply AC and pulsed DC voltages for use in electrofishing. The output voltage from the VVP-2E can be varied from 0 to 720 volts AC in 120 volt steps and from 0 to 1,020 volts DC pulsed in 170 volt steps. The AC output is a 60 hertz sine wave voltage. The DC pulsed output is a fast rise, slow decay voltage variable in pulse width from 1 to 7 milliseconds at a frequency of 60 or 120 pulses per second. An additional variable pulse frequency adjustment has been added to this unit. We call it the "catfish channel." It enables adjustment of the pulse frequency from 2 to 40 pulses per second.

The VVP-2E features an automatic electronic circuit breaker to protect it against overloads in-that when an overload occurs, the circuit breaker will automatically turn the output voltage off and, after a short delay, will turn it back on if the overload has been removed. The unit also contains a timer to indicate the actual time (in seconds) that voltage is being applied to the electrodes.

Front Panel Controls

1. ELECTRODE VOLTS Selector Switch: Selects the electrode voltage-- either AC or DC pulsed.

2. ELECTRODE CURRENT Meter: Indicates the actual current being drawn by the electrodes.

3. ELECTRODE ON TIME Digital Counter: Indicates the time in seconds that the voltage is applied to the electrodes.

4. ELECTRODES OFF Green Pilot Lamp: Indicates that no voltage is being applied to the electrodes.

5. PULSE WIDTH MILLISECONDS Selector Switch: Selects the DC pulse width from 1 to 7 milliseconds.

6. ELECTRODES ON Red Pilot Lamp: Indicates that voltage is being applied to the electrodes.

7. POWER ON-OFF Circuit Breaker: Is the main power on-off switch and circuit breaker.

8. SAFETY OPERATE-SAFE Toggle Switch: Is the master safety switch. It prevents operation of the system when in the SAFE position.

9. Red pilot lamp: Indicates that primary power is being applied to the unit.

10. <u>PULSE FREQUENCY 60 -120 Switch</u>: Selects the desired frequency of the DC pulsed voltage. Also a variable frequency adjuster has been added.

11. <u>ELECTRODES Connector</u>: For the positive (pin A), negative (pin B), and safety switch (pins C & D).

<u>Operating Instructions</u>

1. Review previously mentioned references on electrofishing.
2. Set the POWER ON-OFF circuit breaker to the OFF position.
3. Set the SAFETY OPERATE-SAFE switch to the SAFE position.
4. Set the ELECTRODE VOLTS switch to the OFF position.
5. Set the AC-DC PULSE switch to the desired position.
6. Set the PULSE FREQUENCY switch to the desired frequency (60).
7. Reset the ELECTRODES ON TIMER.
8. Connect the foot switches to the SAFETY SWITCH connectors.
9. Connect the electrodes to the ELECTRODES connectors.
10. Connect the INPUT 230 VOLT AC connector to a suitable 230 volt AC 60 hertz single phase 5000 watt power source (generator) and start generator (Honda EB or EG-5000).
11. Set the POWER ON-OFF circuit breaker to the ON position.
12. Set the ELECTRODE VOLTS switch to the desired electrode voltage. (Suggest 340 volts DC).
13. Set the PULSE WIDTH MILLISECONDS switch to the desired pulse width. (Suggest 2 - 3 milliseconds to start).
14. Set the SAFETY OPERATION - SAFE switch to the OPERATE position.
15. The unit is now ready for operation. The white pilot lamp should be lit and green ELECTRODES OFF pilot lamp should be lit.

16. Depressing both foot switches will now apply power to the electrodes and the ELECTRODE CURRENT meter will indicate the electrode current in amperes. The red ELECTRODES ON lamp should light and the green ELECTRODES OFF lamp should go off.

17. If an overload conditions exists, the green lamp will come back on momentarily and then the red lamp. They will quickly alternate on and off. THE UNIT SHOULD NOT BE OPERATED WHEN THIS CONDITION EXISTS. Immediately set the SAFETY OPERATE-SAFE switch to the SAFE position and then set the electrode voltage to the next lower position. The SAFETY OPERATE-SAFE switch can then be turned back to the OPERATE position. If the overload still exists, repeat the above until the ELECTRODES ON light remains on. The pulse frequency may also be turned down to avoid system overload.

Smith-Root Gas Powered Pulsator (GPP 2.5)

This is the control unit used for the SR-6 Tote Barge system. The Biological Studies Section has two identical units. The tote barge is used to electrofish in all wadable streams. Since the generator and pulsator are in a large floating barge (approx. 3 1/2' X 6'), the only thing that personnel must carry are the dip nets and the anode. This is much more desireable than a backpack setup.

The GPP Electrofisher is a rugged, portable field electrofisher. The output-control circuits are housed in durable weather resistant boxes. The power is supplied from specially manufactured generators. The generators are wound so that the output voltages are taken directly from the generator, eliminating the need for a transformer or the use of voltage doubler circuits.

Two output waveshapes are provided, pulsating AC or pulsating DC. The DC and AC outputs are fast rising, slow decay pulses. The DC pulse rate is selectable between 30, 60, and 120 pulses per second. The AC frequency is non-selectable and is set at 60 cycles per second. The output voltage is continuously adjustable using the PERCENT OF RANGE control.

For output voltages of 30 volts or more, an audio power-on indicator is provided. The unit also comes with a time counter to help evaluate fish capture effectiveness. The time counter operates only during actual shocking and at the rate of one count per second. The counter is resettable to zero with a front panel control button.

GPP 2.5 Specifications

PULSE SHAPE AC:	fast rise, slow decay
PULSE SHAPE DC:	fast rise, slow decay

PULSE FREQUENCY AC:	60 c.p.s.
PULSE FREQUENCY DC:	30, 60, or 120 p.p.s., selectable
TIMER RATE:	1 count per second while shocking
TIMER CAPACITY:	999999 seconds
TIMER ACCURACY:	+-2.5%
CONSTRUCTION:	splash-proof, aluminum case

GPP 2.5 Front Panel Controls And Plugs

1. OUTPUT MODE SELECTOR SWITCHES: Selects the output pulse rate of 30, 60, or 120 p.p.s.. The switches must be in the 120 p.p.s. position and the AC position to get AC output. All other positions will give a pulsed DC output.

2. VOLTAGE RANGE SWITCH: This switch selects the output voltage range or switches the output voltage to OFF.

3. PERCENT OF RANGE: Adjusts the peak voltage of the pulsed AC and the pulsed DC. The pulse duration is also being adjusted from 1 to 5 milliseconds.

4. HIGH VOLTAGE INDICATOR: Indicates when voltage is present on output power terminals.

5. ENUNCIATOR VOLUME: Provides volume control of audio alarm which is on when a voltage is present on output terminals. This should be turned up so that all personnel can hear it.

6. AMMETER: Indicates the current flowing through the water via the anode-cathode electrodes. The area of meter marked "low range" indicates that the voltage selector switch should be in the low range position. Always run the unit in the low range for normal conductivity waters to minimize fish mortality. The ammeter should read no higher than 1.5 amperes for this system.

7. SECONDS COUNTER: Records actual shocking time in seconds. (Actuated at on count per second only when high voltage is applied to the anode).

8. EMERGENCY SHUTDOWN: Provides a local override of remote safety switches.

9. OUTPUT CONTROL: Remote switch provided with GPP electrofisher is plugged in here and is used to actuate the circuits supplying high voltage to the anode.

10. OUTPUT POWER: This 7-pin connector supplies power to the anode and cathode, with pins 1 and 2 anode, and pins 6 and 7 cathode.

11. INPUT POWER: The unit receives power from the generator through this 7-pin connector.

12. 12VDC TERMINAL POSTS ON GENERATOR END BELL: Will provide up to 500 watts of 12 volts DC power. This may be used to run 12 volt lights, or with an external rectifier will charge batteries.

GPP 2.5 OPERATING PROCEDURE

Set-up

1. Check the oil in generator level and add as necessary S.A.E. 10W-30 detergent oil. Do not overfill.

2. Fill the generator gasoline tank with clean, fresh, regular grade unleaded gasoline. Do not mix oil with the gasoline.

3. Connect anode and cathode to their output plug.

4. Plug cable from generator into connector labeled INPUT PWR.

5. Fully choke the carburator of the generator engine to start and open the choke once the engine is running.

6. An exciter button is provided to flash the rotating field of the generator. If the rotating field should ever lose its residual magnetism press the button for a few seconds until the generator begins to operate (not on some generators).

Electrofishing Procedure

1. Set the OUTPUT MODE SELECTOR SWITCH to the desired mode. If you are not sure which mode you desire, it is suggested that you start with 60 p.p.s. DC. If you did your reading you will understand why.

2. Set the PERCENT OF RANGE to minimum.

3. Set the OUTPUT RANGE SELECTOR switch to LOW.

4. Set the EMERGENCY SHUTDOWN SWITCH to ON and ENUNCIATOR VOLUME to mid-range.

5. The operator then activates the REMOTE SWITCH; the H.V. indicator lamp and audio alarm should come on. A slight deflection of the AMMETER should be noted. You are now electrofishing.

6. Adjust the PERCENT OF RANGE and RANGE SELECTOR SWITCH as necessary to achieve optimum response in the fish being shocked. Experience has determined that the ammeter should not read any higher than 1.5 amperes. Under normal water conductivity conditions always select the LOW RANGE. Also, adjust the enunciator so that it is audible to all personnel. NOTE: Always deactivate the GPP unit before changing position of VOLTAGE RANGE SWITCH. (Damage to the voltage range selector switch may result from switching under load.)

7. If the AMMETER should be deflected into the area marked low range and the RANGE SELECTOR is in the high range position, switch to the low range position. (The meter is calibrated so that the rated power of the generator falls within the ranges.)

8. The low voltage windings on the generator are capable of delivering more power than the high voltage windings. If erratic operation occurs in the high range, switch to the low range. Do not operate the generator above the power ranges indicated on the meter face or possible engine and generator damage may occur.

9. A certain amount of experimentation will be required to learn what mode settings and voltage settings are best for the various water conditions and types of fish being shocked.

Generator Maintenance

1. Change oil after first five hours of operation. Thereafter change oil every 25 hours or operation (do not overfill).

2. SERVICE DUAL ELEMENT AIR CLEANER. Clean and re-oil pre-cleaner at three month intervals or every 25 hours, whichever occurs first. Yearly or every 100 hours, whichever occurs first, remove paper cartridge. Clean by tapping gently on flat surface replace if very dirty.

3. CLEAN COOLING SYSTEM (if equipped). Yearly or every 100 hours. whichever occurs first, remove the blower housing and clean the internal areas to avoid overspeeding, overheating and engine damage.

4. SPARK PLUG. Clean and reset gap at 0.030" every 100 hours of operation.

5. REMOVE COMBUSTION DEPOSITS from cylinder head and cylinder head shield every 100-300 hours of operation.

6. INSPECT SPARK ARRESTER screen assembly every 50 hours, replace if damaged.

7. INSPECT GENERATOR BRUSHES after each 100 hours of operation and replace when brushes are worn to 3/8" (1cm) or less. ALWAYS REPLACE BRUSHES IN SETS.

All electrofishing situations, whether boat or tote barge requires 3 personnel. With boat situations one person will captain the boat and will control the electrofishing pulsator. The other two personnel will stand on the bow of the boat and net the fish that are stunned. In wadable stream situations one person will push and control the tote barge, and operate and monitor the pulsator. The second person will do the fish shocking with the anode. The third person will be netting the fish that are shocked. It is important that the person controlling the tote barge maintain a little distance from the shocker and netter (4-6 meters). This helps to keep the anode wire from bunching up under the tote barge in very shallow water. Bunching causes abrasion on the anode wire which can damage its integrity. This is the reason that it is important to inspect the anode harness wire on a daily basis. Always bring a spare anode harness and anode on electrofishing trips to help avoid unnecessary down time.

In all electrofishing situations consider fish habitats for species selectivity. The object in fish tissue collection is to collect mature representative species of the waterbody. Individuals of bottom feeders, predators, and omnivores are selected which may show accumulation of toxic substances (if present) in their tissues. Selectivity for individuals would be the larger, older ones which are more commonly angled for by fishermen and which may have an accumulation of contaminants in the tissue. Results of the fish flesh contamination studies are used to determine if fish consumption advisories should be issued for the waterbody, and to determine the extent of the pollution conditions.

PRESERVATION AND STORAGE OF FISH SAMPLES

Fish collected as part of general biological surveys may be identified in the field, weighed, measured, and released or they may be preserved and brought back to the lab for identification. The fish are preserved in a 10% formalin solution (std. formaldehyde diluted from 37 to 10%). They should be left in the formalin for at least 48 hours and then rinsed thoroughly with distilled water. Specimens may then be stored in clean formalin or 70% isopropanol. Information on the location, habitat, date of collection, distance and/or time sampled are recorded on field data sheets. Samples returned to the lab will have all information written (in pencil) on cotton bond paper inside the jar.

Some fish are collected to be analyzed for pesticides, PCBs, metals, acid extractable and base/neutral extractable semivolatile organic compounds, and volatile organic compounds. Preferrable samples collected from a location would be of a representative bottom feeder, a predator, and an omnivore. Ideally a sample would then consist of 3-5 individuals for larger fish such as carp, or 8-10 individuals for smaller individuals such as sunfish, small bass, or suckers. Individuals in each sample are weighed and measured, then cut with a clean stainless steel filleting knife into skin-on scaleless fillets (skin-off fillets for catfish). Approximately 500 grams of fish tissue (minimum) is required per sample. A filleted sample approximates 30-50% of the fishes whole weight depending on the species. The individual fish fillets from a sample are then composited into one package. They are wrapped in non-animal fat greased aluminum foil (Reynolds brand #625), labeled with the date, site, county, location, species, number of individuals, sample preparation, and sample number, and sealed in plastic bast with labeling tape. The BSS sample number is written on the labeling tape with a dry marker. All samples are then placed on dry ice that is brought along on tissue collecting field trips. Dry ice is carried in a dry ice chest. Samples can be placed together with dry ice in coolers. When the field crew returns to the office the samples are transferred to the upright freezer located in the Water Quality Surveys and Standards Branch Equipment Room at the Bradbury office location.

Log Book/Field Sheets

Two log books maintained in which each and every fish (or other biological tissue) sample and sediment sample collected for contaminant analysis, whether eventually analyzed or not, is logged into at the time of collection. Book #1 starts at annual Biological Studies sample number 001-yr. Book #2 starts at annual sample number 500-yr (where yr is the sampling year). Samples are logged-in consecutively in a book regardless of the sample type or species. Two log books enable two separate field crews to be out simultaneously.

EXAMPLE:BSS sample #, site, county, location,
# of individuals, species, sample preparation
collectors' initials, date of collection, final sample weight in grams

A site information cover sheet is filled out for all sites where fish and/or sediment samples are collected. All information on the field data sheet is to be filled in completely with fish weights recorded in grams and lengths recorded in centimeters. Other fish species observations are checked off when doing tissue collections. Relative abundances may be indicated.

Average fish weight and average fish length is calculated and recorded on the field data sheet for each sample upon returning to the office. A file folder is created which will contain the field data sheets, habitat assessment scoring (if done), and a photocopy of the 7.5 minute topographic map location of the collection. The file is labeled:

site, county
location, collection year

Folders are filed by this index in the Rivers and Streams file drawer.

Data Entry

All biological tissue and sediment samples collected for toxic contaminant analysis are logged into a P.C. database called SMPLELOG. This is an initial sample log and sample tracking file set up in dBASE III+.

1. All site information is logged into this database.

2. After QC procedures for the site and sample information, a file called FISHSIZE (residing in the SMPLELOG subdirectory for that field season year) is appended to for all biological samples. Average length, length range, average weight, weight range, and sample preparation is entered for the samples (edit mode).

3. A retrieval is made for the Biological Studies Section Chief containing a list of the samples, their lengths, and weights for laboratory duplicate, analysis task, and analysis set assignment.

4. As laboratory duplicates, analysis tasks, and analysis sets are assigned they are logged into SMPLELOG for the respective samples. Don't forget to backup the files.

5. When the OK is given by the Biological Studies Section Chief to ship samples to the contract laboratory, a chain-of-custody sheet is printed out through R&R software for each analysis set (see example). The chain-of-custody sheet and any other paperwork to be sent to the laboratory with the shipment are put into a manila envelope and are placed in the cooler containing the samples for that analysis set that is prepared for shipment.

Sample Shipment

1. Biological samples being shipped to the contract laboratory are packed with blue ice and 5-10 pounds of dry ice. the chain-of-custody sheet is signed by the packer and placed inside the cooler which is then sealed with packing tape.

2. A strip of evidence tape is placed on the cooler lid/body. The integrity of this tape will be noted by the receiver of the shipment at the contract laboratory on the chain-of-custody sheet.

3. Each cooler is weighed. There is a package scale in The Biological Studies Laboratory. (Do not pack coolers to greater than 50 pounds.) Practice good lifting procedures when moving these filled coolers.

4. Appropriate information is filled in on the appropriate shipping bill for the courier

chosen by the contract laboratory. The person preparing the shipment is to follow the courier's instructions for this.

5. Each cooler to be shipped will be marked with an IDEM return address label.

6. Each cooler containing dry ice must have a dry ice label:
DRY ICE
UN 1845
ORM- A
#lb/container

Appropriate labels are now being provided by the contract laboratory.

7. The person doing the packing preparation will call the courier for a pick-up. Shipments are always done priority overnight. Do not ship on Friday to avoid Saturday delivery problems for the contract lab.

8. The contract laboratory contact is called to notify them of a next-day shipment coming.

THIS PAGE IS RESERVED FOR THE EXAMPLE OF THE FISH AND SEDIMENT SITE INFORMATION FIELD SHEET

INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT
OWM-BIOLOGICAL STUDIES
FISH AND SEDIMENT COLLECTIONS
SITE INFORMATION

COLLECTION DATE:____/____/____ BSS SAMPLE #:__________________ COLLECTORS (INITIALS):________

____

WATERBODY:____ LOCATION:____

COUNTY:____________________ ECOREGION:____________ SEGMENT:________ IAS NATURAL REGION CODE:__________

LATITUDE:____/____/____ LONGITUDE:____/____/____ HYDROLOGIC UNIT:________________

GRADIENT:________________FEET/MILE DRAINAGE AREA:__________SQUARE MILES TOPO MAP:______________

HABITAT ASSESSMENT (Y or N)?:________ # JARS PER SAMPLE:________ ANALYSIS TASKS:____________________

NOTES:

THIS PAGE IS RESERVED FOR THE EXAMPLE OF THE "SUMMARY OF FISH COLLECTION DATA" SHEET

THIS PAGE IS RESERVED FOR THE EXAMPLE OF THE "SUMMARY OF FISH COLLECTION DATA" SHEET CONTINUED

BIOLOGICAL STUDIES AND STANDARDS
Summary Of Fish Collection Data

Stream ______________________ Location ______________________

Date __________ Station Number ______________________ Sampling Time __________

Method of Sampling ______________________ Distance Sampled ______________________

Purpose of Survey ______________________ Collectors ______________________

Station Description ______________________

Fish Collected or Whole Fillet	Number	Length	Weight	Scale
Sample No. 1				
	1			
	2			
	3			
	4			
	5			
Sample No. 2				
	6			
	7			
	8			
	9			
	10			
Sample No. 3				
	11			
	12			
	13			
	14			
	15			

Fish Observed:

Family and Species	Number
CATOSTOMIDAE	
Blue Sucker	
Northern Hog Sucker	
White Sucker	
Buffalo	
River Carpsucker	
Quillback Carpsucker	
Highfin Carpsucker	
Silver Redhorse	
Golden Redhorse	

Family and Species	Number
Black Redhorse	
Shorthead Redhorse	
River Redhorse	
Spotted Sucker	
Chubsuckers	
Other	
CLUPEIDAE	
Gizzard Shad	
Threadfin Shad	
Skipjack Herring	

State Form 40133

Family and Species	Number
CENTRARCHIDAE	
Largemouth Bass	
Smallmouth Bass	
Spotted Bass	
Green Sunfish	
Longear Sunfish	
Bluegill Sunfish	
Redear Sunfish	
Rock Bass	
Black Crappie	
White Crappie	
Other	
CYPRINIDAE	
Carp	
Bluntnose Minnow	
Stoneroller	
Suckermouth Minnow	
Creek Chub	
Silverjaw Minnow	
Redfin Shiner	
Spotfin Shiner	
Emerald Shiner	
Striped (common) Shiner	
River Shiner	
Sand Shiner	
Other Shiners	
Other Cyprinidae	
ESOCIDAE	
Northern Pike	
Grass Pickerel	
Chain Pickerel	
ICTALURIDAE	
Channel Catfish	
Flathead Catfish	
Yellow Bullhead	

Family and Species	Number
Brown Bullhead	
Black Bullhead	
Madtoms	
LEPISOSTEIDAE	
Longnose Gar	
Shortnose Gar	
Spotted Gar	
PERCICHTHYIDAE	
White Bass	
Yellow Bass	
PERCIDAE	
Yellow Perch	
Walleye	
Sauger	
Johnny Darter	
Greenside Darter	
Fantail Darter	
Rainbow Darter	
Orangethroat Darter	
Other	
SALMONIDAE	
Brown Trout	
Chinook	
Coho Salmon	
Steelhead Trout	
Other	
SCIAENIDAE	
Freshwater Drum	
OTHER:	

THIS PAGE IS RESERVED FOR THE EXAMPLE OF THE RAPID BIOASSESSMENT PROTOCOL V "FISH COMMUNITY ASSESSMENT" FIELD SHEET

INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT
OWM-BIOLOGICAL STUDIES
RAPID BIOASSESSMENT PROTOCOL V
FISH COMMUNITY ASSESSMENT

PAGE___ OF ___

COLLECTION DATE:___/___/___ WATERBODY:____________ LOCATION:____________

COUNTY:____________ ECOREGION:________ SEGMENT:_____ IAS NATURAL REGION:______ HYDROLOGIC UNIT:________

LATITUDE:___/___/___ LONGITUDE:___/___/___ GRADIENT:______FT/MILE DRAINAGE AREA:______ TOPO MAP:_____

HABITAT ASSESSMENT?____ METHOD OF COLLECTION:________ DISTANCE SAMPLED:________ SAMPLE TIME:________ CREW:________

SPECIES	LENGTH (CM)	WEIGHT (GM)	ANOMOLIES?	SPECIES	LENGTH (CM)	WEIGHT (GM)	ANOMOLIES?

THIS PAGE IS RESERVED FOR THE EXAMPLE OF THE CURRENT CONTRACT LABORATORY CHAIN-OF-CUSTODY RETRIEVAL FROM SMPLELOG

PAGE # OF ______

COOLER _____ OF _____

CHAIN OF CUSTODY

INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT
OWM-BIOLOGICAL STUDIES

SHIPMENT PREPARED BY:

SHIPMENT DATE:mm/dd/yy

SAMPLE	DESCRIPTION	SAMPLE #	SITE	LOCATION	SPECIES	COLLECTION DATE	NUMBER OF PACKAGES	TASKS*
A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA
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A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA
A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA
A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA
A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA
A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA
A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA
A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA
A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA
A AAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAA	AA AAAAAAAAAAAAAAAAAAAAAA	MM/DD/YY	AAAAAAAAAAAAAAA	AAAAAAAAAAAAAAAAA

RELINQUISHED BY: (SIGNATURE)	RECEIVED BY: (SIGNATURE)	DATE: TIME: AM PM
RELINQUISHED BY: (SIGNATURE)	RECEIVED BY: (SIGNATURE)	DATE: TIME: AM PM
SHIPPED BY: (SIGNATURE) METHOD OF SHIPMENT:	RECEIVED FOR LABORATORY BY: (SIGNATURE AND NAME OF LAB, AND CONDITION OF EVIDENCE TAPE).	SPECIAL INSTRUCTIONS: * = THIS SAMPLE WILL BE THE LABORATORY DUPLICATE OF SAMPLE 010-91. = SAMPLES IN THIS COOLER

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REMEDIAL ACTION CONTRACT 2 FOR REMEDIAL, ENFORCEMENT OVERSIGHT, AND NON-TIME-CRITICAL REMOVAL ACTIVITIES IN REGION 5

APPENDIX B

QUALITY ASSURANCE PROJECT PLAN
REVISION 0
CBS MULTI-SITES
MONROE COUNTY, INDIANA

Prepared for
U.S. Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, IL 60604

Work Assignment No.	:	103-TATA-05ZZ
EPA Region	:	5
Date Prepared	:	March 14, 2014
Contract No.	:	EP-S5-06-02
Prepared by	:	SulTRAC
SulTRAC Project Manager	:	Jeffrey Lifka
Telephone No.	:	(312) 201-7491
EPA Work Assignment Manager	:	Mr. Thomas Alcamo
Telephone No.	:	(312) 886-7278

CONTENTS

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CONTENTS (CONTINUED)

Section **Page**

FIGURES

FIGURE

ABBREVIATIONS, ACRONYMS, AND SYMBOLS

%D	Percent difference
%R	Percent recovery
µg/kg	Micrograms per kilogram
µg/L	Micrograms per liter
CA	Corrective action
CAS	Chemical Abstract Services
CBS	CBS Corporation
CBS sites	CBS Multi-Sites
^{o}C	Degrees Celsius
CD	Consent decree
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	Chain of custody
DQI	Data quality indicator
EPA	U.S. Environmental Protection Agency
FSP	Field sampling plan
GC	Gas chromatography
HAZWOPER	Hazardous Waste Operations and Emergency Response Standard
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectroscopy
ID	Identification
IDEM	Indiana Department of Environmental Management
LCS	Laboratory control sample
LIMS	Laboratory information management system
MCL	Maximum contaminant level
mg/kg	Milligrams per kilogram
mg/L	Milligrams per liter
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not applicable
NFG	National Functional Guidelines
OSHA	Occupational Safety and Health Administration
OU	Operable unit
oz	Ounce
PCB	Polychlorinated biphenyl
ppm	Parts per million
PQO	Project quality objective
PRQL	Project-required quantitation limit
PTFE	Polytetrafluoroethylene
QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
QL	Quantitation limit
RAC	Remedial Action Contract
RCRA	Resource Conservation and Recovery Act
RL	Reporting limit
RPD	Relative percent difference

ABBREVIATIONS, ACRONYMS, AND SYMBOLS (CONTINUED)

RSD	Relative standard deviation
SAP	Sampling and analysis plan
SOP	Standard operating procedure
SOW	Statement of work
TBD	To be determined
UFP	Uniform Federal Policy for Implementing Environmental Quality Systems
WA	Work assignment
WAM	Work assignment manager
Westinghouse	Westinghouse Electric Corporation

1.0 INTRODUCTION

SulTRAC has prepared this quality assurance project plan (QAPP) as part of the sampling and analysis plan (SAP) for the CBS Multi-Sites (CBS sites) in Monroe County, Indiana, under the U.S. Environmental Protection Agency (EPA) Remedial Action Contract (RAC) 2 for Region 5, Contract No. EP-S5-06-02, and Work Assignment (WA) 103-TATA-05ZZ. Under WA 103-TATA-05ZZ, SulTRAC will provide technical assistance in sampling and analysis to measure the effectiveness of past remediation activities at the CBS sites. Sampling and analysis will focus on determining levels of contamination in fish tissue, surface water, groundwater, and sediment. Historical data from past sampling and analysis events will be used to analyze trends in contamination levels.

The SAP consists of the field sampling plan (FSP) (Attachment A) (SulTRAC 2014b) and the QAPP (Attachment B), which are among the site-specific plans to be prepared in accordance with Task 1 in the EPA statement of work (SOW) (EPA 2014).

This QAPP describes quality assurance (QA) and quality control (QC) protocols, as well as objectives, methods, and procedures that SulTRAC will follow during sampling and analysis support activities. SulTRAC will acquire data from its various field activities or combinations of other activities that will support assessment of remediation efficacy.

The primary goals of sampling activities are to (1) evaluate fish tissue contamination data, especially by comparison to historical fish tissue contamination data, (2) further define the nature and extent of groundwater contamination near the sites, (3) evaluate human health and ecological risks that may be posed by site contamination, and (4) evaluate contamination levels in local streams, including surface water and sediment.

Section 2.0 of this QAPP describes the site and its history, and Section 3.0 specifies the QAPP procedures. The QAPP worksheets appear after Section 3.0. References used to prepare this QAPP are listed after the worksheets, and figures appear after the list of references.

All information regarding laboratory analysis presented in this QAPP pertains to samples to be analyzed by the subcontracted laboratory, TestAmerica, Inc., in Pittsburgh, Pennsylvania, and Knoxville, Tennessee.

2.0 SITE DESCRIPTION AND HISTORY

The CBS sites are six associated sites located in and near Bloomington, Monroe County, Indiana. The technical assistance outlined in the SOW (EPA 2014) focuses on three of the six sites: Lemon Lane Landfill, Neal's Landfill, and Bennett's Dump. Lemon Lane Landfill and Neal's landfill were municipal landfills that accepted municipal and industrial waste. Bennett's Dump was originally a limestone quarry, but was used subsequently as a dumping ground for various types of waste. The area surrounding Bennett's Dump is currently undergoing industrial, commercial, and residential development. Figure 1 shows the site location.

The approximately 10-acre Lemon Lane Landfill isat the western edge of the City of Bloomington, surrounded by a residential community, a cemetery, railroad tracks, and vacant land. The landfill accepted both municipal and industrial waste during its operation from 1933 to 1964. From 1958 until 1964, disposal of a large number of electrical transformers containing polychlorinated biphenyls (PCB) occurred at the landfill, along with disposal of associated industrial byproducts contaminated with PCBs. It was determined that the PCB-contaminated waste at this landfill and the other associated CBS sites had been generated by Westinghouse Electric Corporation (Westinghouse). Westinghouse was purchased by Viacom Incorporated, which was then purchased by CBS Corporation (CBS). In 1985, Westinghouse, EPA, the City of Bloomington, Monroe County, and the Indiana State Board of Health signed a Consent Decree (CD) that required Westinghouse to implement interim control measures, construct an incinerator, and incinerate PCB-contaminated materials from six sites in and near Bloomington, including the Lemon Lane Landfill. During the early 1990s, the State of Indiana passed a number of laws that initially delayed and ultimately blocked the construction of the incinerator required by the 1985 CD. CBS is currently considered the sole responsible party for each of the CBS Multi-Sites, including Lemon Lane Landfill.

Groundwater and spring water at Lemon Lane Landfill are contaminated with PCBs. The spring water enters Clear Creek, and thus has contaminated the creek; this has induced issuance of an advisory not to eat fish from Clear Creek. The remedial action for the source control operable unit (OU) was completed in 2000, as was construction of a water treatment plant that treats 1,000 gallons per minute and has storage capacity of 1.2 million gallons. The Record of Decision for OU2 (groundwater), signed in September 2006 and also described in the 2009 Consent Decree Amendment, called for (1) installation of a new line to carry treated effluent, (2) sediment/soils cleanup at the swallow hole/ICS emergence/quarry springs area, and (3) installation of a storm water storage tank treatment system. All three of these activities have been completed, and Phase 2 will consist of evaluating both Quarry B spring and Rinker spring to determine if treatment of the springs must occur at the water treatment plant.

The approximately 18-acre Neal's Landfill is about 3 miles west of the City of Bloomington surrounded by farms and woodlands. The landfill accepted both industrial and municipal waste during its operation from 1949 to 1972. From 1962 to 1970, disposal of a large number of electrical transformers containing PCBs occurred at the landfill, along with disposal of associated industrial byproducts contaminated with PCBs. CBS is the sole responsible party for the contamination at Neal's Landfill. Groundwater and spring water are contaminated with PCBs at the site. During rain events, spring waters bypass the water treatment system and enter Conard's Branch and Richland Creek, introducing contaminants to both creeks. The remedial action for the source control OU was completed in 1999, and the groundwater and sediment OUs were described in the September 2007 Record of Decision Amendment and 2009 Consent Decree Amendment. The means to remediate groundwater and sediment include two collection systems to capture water from South Spring, North Spring, North Spring Bypass and Overflow Springs. Sediment contaminated with PCBs has been removed and disposed of at an off-site landfill. Sediment in-stream and within the banks was remediated to an average PCB concentration of 1 part per million (ppm) and floodplain sediment was remediated to an average PCB concentration of 5 ppm. The monitoring plan for Neal's Landfill includes a remedy reopener contingent on cleanup of fish tissue:if PCB concentrations in fish tissue do not decrease below specified levels, additional remedial measures may be implemented.

Bennett's Dump, approximately 2.5 miles northwest of the City of Bloomington, encompasses two parcels of land totaling approximately 4 acres. The site is surrounded by limestone quarry and cutting operations. Bennett's Dump was a limestone quarry before use as a dumping ground for various waste materials, including construction debris, household waste, and electrical components. A large number of electrical transformers containing PCBs were dumped at the site during the 1960s and 1970s. These transformers were traced to Westinghouse, rendering CBS the sole responsible party for contamination at this site. Groundwater, soils, and sediment were contaminated at the site. Groundwater emerging from the site enters and thus contaminates Stout's Creek. The remedial action for the source control operable unit (OU1) was completed in 1999, and the September 2006 Record of Decision and 2009 Consent Decree Amendment described in detail how implementation of the groundwater and sediment OUs was to occur. Two phases of work were scheduled. Phase 1, completed in 2010, included implementation of a passive quarry drain for the Wedge Quarry Complex. Phase 2 included a groundwater investigation to determine the effects of the passive quarry drain on groundwater flow and PCB concentrations in groundwater. At this time, the Phase 2 groundwater investigation is underway. Phase 2 also involves installation of a collection trench and water treatment plant with the size of the remedy components to depend on results of the passive quarry drain groundwater investigation. Completion of construction is expected in 2014. Additional sampling by EPA will be required to ensure effectiveness of the final remedy.

3.0 QUALITY ASSURANCE PROJECT PLAN PROCEDURES

This QAPP specifies procedures for attaining specific project objectives by acquisition of sampling and analytical data of known and adequate quality pertaining to the CBS sites. This QAPP was prepared in accordance with the EPA's "Uniform Federal Policy for Implementing Environmental Quality Systems" (UFP) (EPA 2005).

Initiation of field activities for sampling and analysis is anticipated in spring/summer 2014. The following is a list of expected field activities as discussed in SulTRAC's work plan (SulTRAC 2014a):

Conduct Fish Sampling Events (Fish Tissue)

SulTRAC will conduct two fish sampling events within streams on or near the CBS sites to determine levels of PCB contamination in fish tissue. Results will be compared to historical fish sampling data to determine the effectiveness of past remedial activities, and to assess need for additional remediation.

SulTRAC anticipates two fish sampling events, spaced 1 year apart. Sampling will occur at Clear Creek (associated with Lemon Lane Landfill), Conard's Branch and Richland Creek (associated with Neal's Landfill), and Stout's Creek (associated with Bennett's Dump). Fish will be collected at multiple locations within each stream, consistent with historical fish sampling locations. SulTRAC anticipates collecting a total of 264 fish samples, plus additional samples for laboratory QA/QC.

SulTRAC anticipates that all fish samples will be collected using a backpack electroshock unit. SulTRAC expects that the majority of sampling locations will be accessible using waders and the backpack electroshock unit. Possibly, stream conditions may render use of a boat necessary at certain locations. If so, the backpack electroshock unit (or deeper water electroshocking probes) will be utilized from the boat. Samples will be analyzed by TestAmerica.

Conduct Sampling for Remedial Activity Evaluation (Sediment, Groundwater, Surface Water)

SulTRAC will conduct a to-be-determined number of sampling events to evaluate the effectiveness of past remediation activities. SulTRAC anticipates collecting 33 sediment samples, 33 groundwater samples, and 33 surface water samples (plus associated laboratory QA/QC samples). Sampling events will proceed as directed by the EPA work assignment manager (WAM). Specific sampling locations will also be determined by the WAM.

SulTRAC anticipates collecting sediment samples using either a trowel or dedicated sediment sampling equipment based on site conditions and sample locations. SulTRAC anticipates collecting groundwater samples from existing monitoring wells by application of standard bailing, submerged, or peristaltic pumping techniques. SulTRAC expects to collect surface water samples by immersing sample containers in the body of water to be sampled, and filling directly. All samples collected will be analyzed for PCBs as Aroclors by TestAmerica.

QAPP WORKSHEET #1
TITLE AND APPROVAL PAGE

Quality Assurance Project Plan for Technical Assistance, CBS Multi-sites, Monroe County, Indiana
Document Title

SulTRAC
Lead Organization

Adam Peterca, SulTRAC
Preparer's Name and Organizational Affiliation

1 South Wacker Drive, Suite 3700, Chicago, IL 60606 (312) 201-7768; adam.peterca@tetratech.com
Preparer's Address, Telephone Number, and E-mail Address

March 14, 2014

Preparation Date (Day/Month/Year)

Jeffrey Lifka
SulTRAC Project Manager — Signature/Date

John Dirgo
SulTRAC QA Officer — Signature/Date

Approval Signatures:

Signature/Date
Thomas Alcamo, Work Assignment Manager
Printed Name/Title

Approval Authority
Other Approval Signatures:

Signature/Date
QAPP Reviewer
Printed Name/Title

QAPP WORKSHEET #2
QAPP IDENTIFYING INFORMATION

1. Identify guidance used to prepare QAPP:

"Uniform Federal Policy for Implementing Environmental Quality Systems" (UFP) (EPA 2005) and "EPA Guidance for Quality Assurance Project Plans" (EPA 2002)

2. Identify regulatory program:

Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)

3. Identify approval entity: EPA Region 5

4. Indicate whether the QAPP is a generic or project-specific QAPP: Project-specific

5. List dates of scoping sessions that were held:

EPA SOW Revision No. 2, dated December 19, 2013 (EPA 2013)

EPA SOW Revision No. 2.1, dated January 16, 2014 (EPA 2014)

6. List dates and titles of QAPP documents written for previous work site, if applicable: Not applicable (NA)

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

EPA Region 5, SulTRAC, the City of Bloomington, Monroe County, State of Indiana, CBS Corporation

8. List data users: EPA Region 5, SulTRAC, the City of Bloomington, Monroe County, State of Indiana, CBS Corporation

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below: NA

QAPP WORKSHEET #2 (CONTINUED)
QAPP IDENTIFYING INFORMATION

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	QAPP Worksheet # or Crosswalk to Related Documents
Project Management and Objectives		
2.1 - Title and Approval Page	Title and Approval Page	1
2.2 - Document Format and Table of Contents	Table of Contents	
2.2.1 Document Control Format	QAPP Identifying Information	2
2.2.2 Document Control Numbering System		
2.2.3 Table of Contents		
2.2.4 QAPP Identifying Information		
2.3 - Distribution List and Project Personnel Sign-Off Sheet		
2.3.1 Distribution List	Distribution List	3
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QAPP WORKSHEET #2 (CONTINUED)
QAPP IDENTIFYING INFORMATION

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	QAPP Worksheet # or Crosswalk to Related Documents
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3.2.1 Analytical SOPs	Analytical SOPs	23
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3.3.3 Sample Custody	Example Chain-of-Custody Form and Seal	

QAPP WORKSHEET #2 (CONTINUED)
QAPP IDENTIFYING INFORMATION

Required QAPP Element(s) and Corresponding QAPP Section(s)	**Required Information**	**QAPP Worksheet # or Crosswalk to Related Documents**
3.4 - Quality Control (QC) Samples		
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5.3 - Streamlining Data Review	NA	NA
5.3.1 Data Review Steps to be Streamlined		
5.3.2 Criteria for Streamlining Data Review		
5.3.3 Amounts and Types of Data Appropriate for Streamlining		

QAPP WORKSHEET #3
DISTRIBUTION LIST

QAPP Recipient	Title	Organization	Telephone Number	E-mail Address
Thomas Alcamo	WAM	EPA Region 5	(312) 886-7278	alcamo.thomas@epa.gov
TBD	QAPP Reviewer	EPA Region 5	TBD	TBD@epa.gov
Jessica Fliss	Project Manager	IDEM[1]	(317) 233-2823	jfliss@idem.in.gov
Jeffrey Lifka	Project Manager	SulTRAC	(312) 201-7491	jeffrey.lifka@tetratech.com
Adam Peterca	Field Team Leader	SulTRAC	(312) 201-7768	adam.peterca@tetratech.com
Lance Summers	Project Scientist and Sample Custodian	SulTRAC	(312) 201-7767	lance.summers@tetratech.com
Mindy Gould	Project QA Reviewer	SulTRAC	(312) 201-7460	mindy.gould@tetratech.com
John Dirgo	QA Officer	SulTRAC	(312) 201-7765	john.dirgo@tetratech.com
Carrie Gamber	Laboratory Project Manager	TestAmerica	(412) 963-7058	carrie.gamber@testamericainc.com
John Bassett	Senior Hydrogeologist	AECOM	(812) 334-8301	john.bassett@aecom.com

Notes:

1	Indiana Department of Environmental Management (IDEM) may assist SulTRAC and AECOM with sampling efforts. If IDEM participates, then the IDEM project manager will receive a copy of the QAPP. IDEM management's decision to assist with sampling events is pending.
AECOM	AECOM Technical Services, Inc.
EPA	U.S. Environmental Protection Agency
QA	Quality assurance
TBD	To be determined
WAM	Work assignment manager

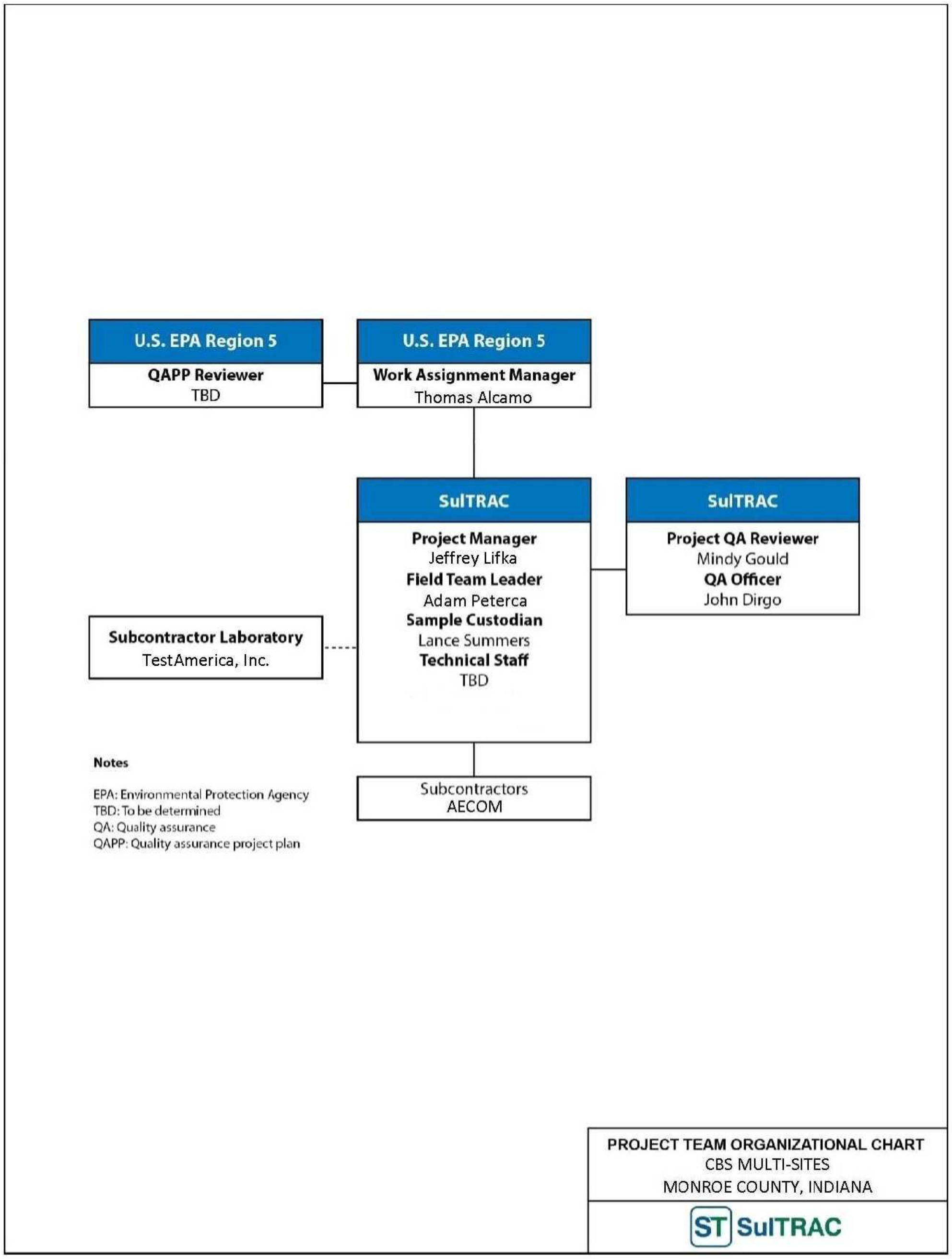
QAPP WORKSHEET #4
PROJECT PERSONNEL SIGN-OFF SHEET

Project Personnel	**Organization**	**Title**	**Telephone No.**	**Signature**	**Date QAPP Read**
Jeffrey Lifka	SulTRAC	Project Manager	(312) 201-7491		
Mindy Gould	SulTRAC	Project QA Reviewer	(312) 201-7460		
John Dirgo	SulTRAC	QA Officer	(312) 201-7765		
Lance Summers	SulTRAC	Project Scientist and Sample Custodian	(312) 201-7767		
Adam Peterca	SulTRAC	Field Team Leader	(312) 201-7768		
Carrie Gamber	TestAmerica	Laboratory Project Manager	(412) 963-7058		
John Bassett	AECOM	Project Manager	(812) 334-8301		
Jessica Fliss	IDEM	Project Manager	(317) 233-2823		

Notes:

TBD	To be determined
QA	Quality assurance

QAPP WORKSHEET #5
PROJECT ORGANIZATION CHART



QAPP WORKSHEET #6
COMMUNICATION PATHWAYS

Communication Drivers	Responsible Entity	Name	Telephone No.	Procedure (Timing, Pathways, etc.)
Point of contact with EPA WAM	Project Manager	Jeffrey Lifka	(312) 201-7491	Jeff Lifka will forward all materials and information about the project to Thomas Alcamo.
Manage all project phases	Project Manager	Jeffrey Lifka	(312) 201-7491	Communicate information to project team (including subcontractors) on a timely basis. Notify EPA WAM by telephone or e-mail of any significant issues. Direct field team and facilitate communication with subcontracted laboratory. Deliver all laboratory data packages to project QA reviewer for data validation.
Daily field progress report	Field Team Leader	Adam Peterca	(312) 201-7768	Conduct specific field investigation tasks, direct field activities of subcontractors, and provide daily communication with project manager and sample custodian.
Manage field sample organization and delivery to laboratories	Sample Custodian	Lance Summers	(312) 201-7767	Ensure field staff is collecting samples in proper containers, observing holding times, and properly packaging and preparing samples for shipment. Coordinate daily with subcontracted laboratory concerning sample quantities and delivery locations and dates. Communicate daily with field staff and project manager regarding any issues and developments.
Point of contact with TestAmerica	Sample Custodian	Lance Summers	(312) 201-7767	Contact the TestAmerica before each sampling event to schedule laboratory services. Notify project manager of any laboratory issues or developments. Track all laboratory data deliveries. Notify project manager and forward data to him.
Release of analytical data	SulTRAC Project QA Reviewer	Mindy Gould	(312) 201-7460	No analytical data can be released until validation is completed and the QA reviewer has reviewed and approved the release.
Report of laboratory data quality issues	Laboratory Project Manager	Carrie Gamber	(412) 963-7058	All QA/QC issues with project field samples will be reported to the sample custodian.

Notes:

EPA	U.S. Environmental Protection Agency
QA	Quality assurance
QC	Quality control
TBD	To be determined
WAM	Work assignment manager

QAPP WORKSHEET #7
PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS TABLE

Name	Title	Organization/ Affiliation	Responsibilities	Education and Experience Qualifications [1]
Jeffrey Lifka	Project Manager	SulTRAC	Manages project; coordinates between lead agency and subcontractor; coordinates laboratory data deliverables from sample custodian to project QA reviewer; manages field staff	B.S. Biology; Professional Master Environmental Engineering, 20 years of experience
Adam Peterca	Field Team Leader	SulTRAC	Supervises field sampling and coordinates all field activities; daily reporting to project manager while conducting field activities	B.S. Food, Agricultural, and Biological Engineering, 4 years of experience
Lance Summers	Project Scientist Sample Custodian	SulTRAC	Implements field plan; verifies sample processing, packaging, and shipping	B.S., Environmental Science/Management, 3 years of experience
Mindy Gould	Project QA Reviewer	SulTRAC	QA/QC oversight	M.S. Environmental Engineering, 30 years of experience
John Dirgo	QA Officer	SulTRAC	QA/QC oversight	B.S. Biology; M.S. and Sc.D. Environmental Health Sciences; 34 years of experience
John Bassett	Project Manager	AECOM	Coordinates field support of AECOM personnel with SulTRAC project manager and field team leader	M.A. Geology, 34 years of experience
TBD	Technical Staff	SulTRAC	Implements field plan	TBD
Carrie Gamber	Laboratory Project Manager	TestAmerica	Provides analytical services for the samples collected during the investigation.	B.S. Biology, 26 years of experience

Notes:

[1]	Staff resumes are located in SulTRAC files
QA	Quality assurance
QC	Quality control
TBD	To be determined

QAPP WORKSHEET #8
SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Field Staff	40-hour and 8-hour refresher OSHA HAZWOPER training	Various	Various	SulTRAC	SulTRAC	Corporate human resources office
Field Staff	CPR and First Aid training	Various	Various	SulTRAC	SulTRAC	Corporate human resources office

Notes:

HAZWOPER	Hazardous Waste Operations and Emergency Response Standard
OSHA	Occupational Safety and Health Administration
TBD	To be determined

QAPP WORKSHEET #9
PROJECT SCOPING SESSION PARTICIPANTS SHEET

Project Name	Technical Assistance for CBS Multi-sites		Site Name	CBS Multi-sites	
Projected Date(s) of Sampling	Spring 2014 – Spring 2016		Site Location	Monroe County, Indiana	
Project Manager	Jeffrey Lifka				
Date of Session	December 19, 2013				
Scoping Session Purpose:	Define scope of project				
Name	**Title**	**Affiliation**	**Phone #**	**E-Mail Address**	**Project Role**
Thomas Alcamo	WAM	EPA Region 5	(312) 353-4627	alcamo.thomas@epa.gov	WAM
Jeffrey Lifka	Project Manager	SulTRAC	(312) 201-7419	jeffrey.lifka@tetratech.com	Project Manager
Mindy Gould	Program Manager	SulTRAC	(312) 201-7460	mindy.gould@tetratech.com	Program Manager
Date of Session	January 16, 2014				
Scoping Session Purpose: Revise previously defined scope of project to include sediment, groundwater, and surface water sampling details					
Name	**Title**	**Affiliation**	**Phone #**	**E-Mail Address**	**Project Role**
Thomas Alcamo	WAM	EPA Region 5	(312) 353-4627	alcamo.thomas@epa.gov	WAM
Jeffrey Lifka	Project Manager	SulTRAC	(312) 201-7419	jeffrey.lifka@tetratech.com	Project Manager
Mindy Gould	Program Manager	SulTRAC	(312) 201-7460	mindy.gould@tetratech.com	Program Manager

Comments/Decisions: The initial SOW No. 2 released on December 19, 2013 outlined the scope of fish sampling activities to be conducted under WA Number 103-TATA-05ZZ. It provided details on the location of fish sampling, number of fish to be collected, and types of analyses to be completed. SOW No. 2.1 was released on January 16, 2014 to revise the previous SOW. The main purpose of this revision was to include details on sediment, groundwater, and surface water sampling that will be conducted to support EPA's efforts to evaluate the effectiveness of past remedial activities. The revision also included the number of samples to be collected, and types of analyses to be performed.

Notes:

EPA	U.S. Environmental Protection Agency
SOW	Statement of Work
WA	Work assignment
WAM	Work assignment manager

QAPP WORKSHEET #10
PROBLEM DEFINITION

The problem to be addressed by the project:

The CBS Multi-Sites consist of six locations in Monroe County, Indiana which were impacted by PCB contamination. This work assignment (WA) focuses on three of these sites: Lemon Lane Landfill, Neal's Landfill, and Bennett's Dump. PCB contamination at each of these sites was linked to the disposal of electrical transformers containing PCBs, as well as associated industrial waste which had been contaminated with PCBs. As a result, CBS is the sole responsible party for the sites.

Lemon Lane Landfill is a former municipal landfill which accepted municipal and industrial waste. The area of the site is approximately 10 acres. Source control remediation was conducted in 1987. Excavation of contaminated soil was completed in 2000. A water treatment plant for groundwater remediation was also installed in 2000. Additional work is nearly complete to install a new effluent line for the water treatment plant, and to evaluate further areas for both groundwater and sediment/soil remediation.

Neal's Landfill is a former municipal landfill which accepted municipal and industrial waste. The area of the site is approximately 18 acres. Excavation of contaminated soil was completed in 2002. Groundwater collection systems and a water treatment plant have been installed at the site, and are currently in use and being evaluated. Sediment in-stream and along stream banks within the site will be excavated in the future.

Bennett's Dump is a former limestone quarry that was used as a dumping ground for various types of waste, including construction debris, household waste, and electrical components, including a large amount of PCB-containing transformers. A passive quarry drain has been installed to aid in controlling groundwater flow. Further construction activities are being planned, including a collection trench and installation of a water treatment plant.

Springs originating on or near the CBS sites, coupled with storm water flow, have transported PCBs into several nearby streams, contaminating surface water, sediment, and organisms. Sediment, surface water, and fish tissue sampling have been conducted in the past to evaluate levels of contamination. The data produced by these sampling events have also been used to define trends in changing levels of contamination, and to evaluate the effectiveness of remedial activities conducted at the CBS sites. The sampling activities proposed in this QAPP will produce data to evaluate PCB contamination levels in fish tissue, sediment, groundwater, and surface water. Data will be used to define trends in contamination levels over time in each matrix sampled. Data will also be compared to applicable levels as follows:

- Fish tissue contaminant levels will be compared to historical levels and project-specific levels defined in a remedy reopener clause
- Sediment contaminant levels will be compared to historical levels and EPA Region 5 Ecological Screening Levels
- Surface water and groundwater contaminant levels will be compared to historical project action levels

Specific information on these analytical levels can be found in Worksheet #15.

QAPP WORKSHEET #10 (CONTINUED)
PROBLEM DEFINITION

The environmental questions being asked:

1. What are the current levels of PCB contamination in fish tissue?
2. Are there any statistically significant trends in PCB levels in fish tissue when compared to historical data?
3. Have PCB levels in sediment, groundwater, and surface water changed since past sampling events?
4. Are current remedial activities effective at reducing PCB contamination at and near the CBS sites?

Observations from any site reconnaissance reports:

EPA has conducted several site investigations over the course of the project. Previous investigations have focused on identifying contaminant sources, evaluating levels of contamination on and near the sites, determining the extent of contamination beyond the site boundaries, and evaluating the results of remediation activities.

A synopsis of secondary data or information from site reports: See Worksheet #13

The possible classes of contaminants and the affected matrices: Data obtained by EPA indicate that PCBs are the main contaminants of concern; affected matrices for this investigation include sediment, groundwater, surface water, and fish tissue.

Project decision conditions (“If…, then…” statements):

1. If the results of fish tissue sampling are above project-specific remedy-reopener levels, then the remedial actions previously taken at the Neal’s Landfill site will be reevaluated with a focus on additional actions to address contamination.
2. If the results of fish tissue, sediment, groundwater, and surface water sampling indicate that recent remedial actions have not adequately addressed contamination at the sites, then additional remedial actions will be considered.

QAPP WORKSHEET #11
PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

Who will use the data: EPA Region 5; County of Monroe, Indiana; City of Bloomington, Indiana; State of Indiana; CBS Corporation; and SulTRAC will use the data.

What will the data be used for?

The data collected from fish tissue sampling will be used to (1) analyze historical trends in PCB contamination levels in fish tissue and (2) evaluate the effectiveness of past remedial activities, including comparison to project-specific remedy-reopener contamination levels which could prompt additional remedial action. The data collected from sediment, groundwater, and surface water sampling will be used to (1) evaluate the effectiveness of past remedial activities and (2) compare current contamination levels to standard screening levels for each matrix sampled.

What type of data are needed (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?

SulTRAC will:

- Collect fish tissue samples to be analyzed for PCBs as congeners, PCBs as Aroclors, and lipid content. Samples will be collected using backpack electrofishing techniques.
- Collect sediment samples to be analyzed for PCBs as Aroclors. Samples will be collected using a trowel or dedicated sediment sampling devices dependent on site conditions.
- Collect groundwater samples to be analyzed for PCBs as Aroclors. Samples will be collected from existing monitoring wells using bailing, submerged, or peristaltic pumping techniques.
- Collect surface water samples to be analyzed for PCBs as Aroclors. Samples will be collected by immersing bottle directly in water.

All samples will be analyzed by the subcontracted laboratory, TestAmerica.

Standard procedures will be used for sample collection, handling, preparation, and analysis. See Worksheets #21 and #23.

How “good” do the data need to be in order to support the environmental decision?

The data from SulTRAC’s samples of fish tissue, sediment, surface water, and groundwater are needed to allow assessment of the nature and extent of contamination in these environmental media. The data will also be used to make comparisons to historical data, and to analyze any potential statistical trends in contaminant levels.

QAPP WORKSHEET #11 (CONTINUED)
PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

How much data are needed (number of samples for each analytical group, matrix, and concentration)?

SulTRAC will collect

(1) as many as 33 fish tissue samples from as many as 10 locations in 4 streams to be analyzed for PCBs as congeners and lipid content
(2) as many as 231 fish tissue samples from 10 locations in 4 streams to be analyzed for PCBs as Aroclors and lipid content
(3) as many as 33 sediment samples, locations to be determined by WAM, to be analyzed for PCBs as Aroclors
(4) as many as 33 groundwater samples, locations to be determined by WAM, to be analyzed for PCBs as Aroclors
(5) as many as 33 surface water samples, locations to be determined by WAM, to be analyzed for PCBs as Aroclors

In addition, laboratory quality control (QC) samples will be collected and analyzed, including MS/MSD samples.

Where, when, and how should the data be collected/generated? See above answers in Worksheet 10 and 11.

Who will collect and generate the data? SulTRAC (with assistance from AECOM and possibly IDEM) will collect all the samples discussed herein. All samples will be analyzed by TestAmerica.

How will the data be reported? Data will be reported by TestAmerica using standard data reporting techniques. Data will be reported in electronic and hard-copy formats.

How will the data be archived? Electronic and hard copies of analytical data will be archived by TestAmerica. Field data (notebooks, sampling sheets, etc.) will be maintained at SulTRAC's Chicago office. SulTRAC will also provide 10-year data storage.

QAPP WORKSHEET #12
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Fish Tissue
Analytical Group	PCBs (congeners)
Concentration Level[a]	NA

Sampling Procedure[b]	**Analytical Method SOP**[c]	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-8	A-2	Precision	RPD ≤ 50%	Field duplicate	S & A
S-8	A-2	Accuracy/Bias	35 to 135 % R	MS/MSD	S & A
S-8	A-2	Precision	RPD ≤ 50 %	MS/MSD	S & A
S-8	A-2	Accuracy	50 to 150% R	Surrogates	A
S-8	A-2	Accuracy	30 to 140 % R	LCS (called “OPR” in the method)	A
S-8	A-2	Accuracy/Bias, Contamination	< Reporting limit (RL)	Method blank	A
S-8	A-2	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Fish Tissue
Analytical Group	PCBs (Aroclors)
Concentration Level[a]	NA

Sampling Procedure[b]	Analytical Method SOP[c]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-8	A-1	Precision	RPD ≤ 50%	Field duplicate	S & A
S-8	A-1	Accuracy/Bias	50 to 140 % R	MS/MSD	S & A
S-8	A-1	Precision	≤ 20 % RPD	MS/MSD	S & A
S-8	A-1	Accuracy	50 to 140 %R	Surrogates	A
S-8	A-1	Accuracy	50 to 140 %R	LCS	A
S-8	A-1	Accuracy/Bias, Contamination	< RL	Method blank	A
S-8	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Fish Tissue
Analytical Group	Lipid Content
Concentration Level[a]	NA

Sampling Procedure[b]	Analytical Method SOP[c]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-8	A-3	Precision	RPD ≤ 50%	Field duplicate	S & A
S-8	A-3	Accuracy/Bias	30 to 150%R	LCS	A
S-8	A-3	Precision	RPD ≤ 25%	Sample Duplicate	S & A
S-8	A-3	Precision	RPD ≤ 25%	LCS Duplicate	A
S-8	A-3	Accuracy/Bias, Contamination	< RL	Method blank	A
S-8	A-3	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Sediment
Analytical Group	PCBs (Aroclors)
Concentration Level[a]	NA

Sampling Procedure[b]	Analytical Method SOP[c]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-2	A-1	Precision	RPD $\leq$ 50%	Field duplicate	S & A
S-2	A-1	Accuracy/Bias	50 to 140 % R	MS/MSD	S & A
S-2	A-1	Precision	≤ 20 % RPD	MS/MSD	S & A
S-2	A-1	Accuracy	20 to 150 %R	Surrogates	A
S-2	A-1	Accuracy	50 to 140 %R	LCS	A
S-2	A-1	Accuracy/Bias, Contamination	< RL	Method blank	A
S-2	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group	PCBs (Aroclors)
Concentration Level[a]	Low

Sampling Procedure[b]	Analytical Method SOP[c]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-3, S-4	A-1	Precision	RPD ≤ 50%	Field duplicate	S & A
S-3, S-4	A-1	Accuracy/Bias	55 to 120 % R	MS/MSD	S & A
S-3, S-4	A-1	Precision	≤ 25 % RPD	MS/MSD	S & A
S-3, S-4	A-1	Accuracy	25 to 150 %R	Surrogates	A
S-3, S-4	A-1	Accuracy	55 to 120 %R	LCS	A
S-3, S-4	A-1	Accuracy/Bias, Contamination	< RL	Method blank	A
S-3, S-4	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

Notes:

a	NA indicates regular concentration levels are acceptable for meeting project objectives.
b	Reference number from QAPP Worksheet #21
c	Reference number from QAPP Worksheet #23

DQI	Data quality indicator
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not Applicable
%R	Percent recovery
RL	Reporting limit
RPD	Relative percent difference
SOP	Standard operating procedure

QAPP WORKSHEET #13
SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Source (Originating Org, Data Types, data Generation/Collection Dates)	How data will be used	Limitation on Data Use
Fish Tissue	Tetra Tech EM Inc. "Statistical Analyses of Polychlorinated Biphenyl (PCB) Concentrations in Fish Samples Collected Near the Lemon Lane Landfill Site," Monroe County, Indiana. November 4, 2003.	Tetra Tech EM Inc.	These data will be used as historical data for comparisons of current levels of contamination in fish tissue. They will also be used for trend analysis of contamination levels, and to evaluate the effectiveness of past remedial actions. The document will also act as a guide for future statistical analyses.	None
Fish Tissue, Sediment, Groundwater, and Surface Water	CBS Corporation. Historical Sampling Results. CBS Multi-Sites: Lemon Land Landfill, Neal's Landfill, and Bennett's Dump	CBS Corporation	These data will be used as historical data for comparisons of current levels of contamination. They will also be used for trend analysis of contamination levels, and to evaluate the effectiveness of past remedial actions.	None
Fish Tissue, Sediment, Groundwater, and Surface Water	Indiana Department of Environmental Management (IDEM). Historical Sampling Results. CBS Multi-Sites: Lemon Land Landfill, Neal's Landfill, and Bennett's Dump	IDEM	These data will be used as historical data for comparisons of current levels of contamination. They will also be used for trend analysis of contamination levels, and to evaluate the effectiveness of past remedial actions.	None
Fish Tissue, Sediment, Groundwater, and Surface Water	U.S. Environmental Protection Agency Region 5. Historical Sampling Results. CBS Multi-Sites: Lemon Land Landfill, Neal's Landfill, and Bennett's Dump	EPA Region 5	These data will be used as historical data for comparisons of current levels of contamination. They will also be used for trend analysis of contamination levels, and to evaluate the effectiveness of past remedial actions.	None

Notes:

EPA	U.S. Environmental Protection Agency
IDEM	Indiana Department of Environmental Management
PCB	Polychlorinated biphenyl

QAPP WORKSHEET #14
SUMMARY OF PROJECT TASKS

Sampling Tasks:

1. Collect up to 264 fish tissue samples (plus quality assurance [QA]/quality control [QC] samples) from up to 10 sample locations in 4 creeks.
2. Collect up to 33 sediment samples (plus QA/QC samples), up to 33 groundwater samples (plus QA/QC samples), and up to 33 surface water samples (plus QA/QC samples). Locations and time of samples to be determined by WAM.

Analysis Tasks:

TestAmerica will analyze 33 fish samples for PCBs as congeners, and 231 fish samples for PCBs as Aroclors. TestAmerica will also analyze all fish samples for lipid content.

TestAmerica will analyze up to 33 sediment, up to 33 surface water, and up to 33 groundwater samples for PCBs as Aroclors.

QC Tasks: The following QC samples will be collected and analyzed during the sampling event: field duplicates, matrix spike (MS)/matrix spike duplicate (MSD) samples, and rinsate blanks.

Secondary Data: See Worksheet #13

Data Management Tasks: Analytical data will be archived in an electronic database after validation.

Documentation and Records: All samples collected will be documented in a logbook using a ballpoint pen. The time of collection, identification number, sampling location, field observations, sampler's name, and analyses will be recorded in the logbook for each sample. Each page of the logbook will be dated, numbered, and signed by SulTRAC personnel. Field data records will be maintained at SulTRAC's Chicago office. SulTRAC will follow custody procedures outlined in SulTRAC's program-level Quality Assurance Project Plan (QAPP) for the Remedial Action Contract (RAC) 2 contract. Further specifications are described in the Field Sampling Plan (FSP) (SulTRAC 2014b).

Assessment/Audit Tasks: No field or laboratory audits are currently planned.

Data Review Tasks: TestAmerica will review and evaluate all data generated, and will prepare a case narrative detailing any issues or inconsistencies discovered. The SulTRAC project manager and chemist will review the case narrative and will detail any analytical issues that may affect data quality in the data evaluation summary report.

QAPP WORKSHEET #15
REFERENCE LIMITS AND EVALUATION TABLE

Reference Limits Table – Fish Tissue

Analytical Group	Analyte	CAS Number	General Project Action Limit – Fish Tissue (mg/kg wet basis)	Neal's Landfill Remedy Reopener Level: Lower Reach of Conard's Branch (mg/kg wet basis; whole fish samples) [1]	Neal's Landfill Remedy Reopener Level: Richland Creek at Vernal Pike Bridge (mg/kg wet basis; whole fish samples) [1]	Neal's Landfill Remedy Reopener Level: Richland Creek at State Route 43 Bridge (mg/kg wet basis; fillet samples) [1]	PRQL – Fish Tissue (mg/kg unless otherwise noted)
PCBs	PCBs (Aroclors)	1336-36-3	Compare to historical levels for trend analysis	2.3	0.9	0.2	0.0167
PCBs	PCBs (congeners)	1336-36-3	Compare to historical levels for trend analysis	NA	NA	NA	0.01 to 0.02 ng/g
Lipids	NA	NA	Compare to historical levels for trend analysis	NA	NA	NA	0.1 %

Reference Limits Table – Sediment

Analytical Group	Analyte	CAS Number	Project Action Limit – Sediment (µg/kg)[2]	PRQL - Sediment (µg/kg)
PCBs	PCBs (Aroclors)	1336-36-3	59.8	16.67

Reference Limits Table – Water

Analytical Group	Analyte	CAS Number	Project Action Limit – Water (µg/L)[3]	PRQL - Water (µg/L)
PCBs	PCBs (Aroclors)	1336-36-3	0.3	0.01

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Notes:

1 Neal's Landfill Remedy Reopener Levels.

2 "U.S. EPA Region 5 RCRA Ecological Screening Levels." EPA 2003.

3 Historical Project Action Limit. Below Indiana State standards for PCB concentration in water (0.5 µg/L)

µg/L	Micrograms per liter
mg/kg	Milligrams per kilogram
CAS	Chemical Abstract Service
PRQL	Project-required quantitation limit

QAPP WORKSHEET #16
PROJECT SCHEDULE/TIMELINE TABLE

Activity	Organization	Date		Deliverable	Deliverable Due Date
		Anticipated Date of Initiation[1]	Anticipated Date of Completion[1]		
Fish Sampling Event No. 1	SulTRAC	Spring/Fall 2014	Spring/Fall 2014 (2 week event duration)	Data validation report	21 days after receipt of analytical results from laboratory
Fish Sampling Event No. 1 Data Evaluation	SulTRAC	Summer/Winter 2014	Summer/Winter 2014	Data Evaluation Summary Report	45 days after receipt of validated data
Fish Sampling Event No. 2	SulTRAC	Spring/Fall 2015	Spring/Fall 2015 (2 week event duration)	Data validation report	21 days after receipt of analytical results from laboratory
Fish Sampling Event No. 2 Data Evaluation	SulTRAC	Summer/Winter 2015	Summer/Winter 2015	Data Evaluation Summary Report	45 days after receipt of validated data
Sediment, Groundwater, and Surface Sampling Events (multiple)	SulTRAC	Spring/Summer 2014	Spring 2016	Data validation report	21 days after receipt of analytical results from laboratory
Sediment, Groundwater, and Surface Sampling Data Evaluation	SulTRAC	Summer 2014	Spring 2016	Data validation report (possibly including interim reports depending on sampling schedule)	45 days after receipt of validated data

Note:

[1] Initiation/Completion date based upon availability of subcontractors, sampling schedule determination by WAM, and SAP approval

QAPP WORKSHEET #17
SAMPLING DESIGN AND RATIONALE

The purpose of the proposed fish sampling activities is to (1) analyze historical trends in PCB contamination levels in fish tissue and (2) evaluate the effectiveness of past remedial activities, including comparison to project-specific remedy-reopener contamination levels which could prompt additional remedial action. The purpose of the proposed sediment, groundwater, and surface water sampling activities is to (1) evaluate the effectiveness of past remedial activities and (2) compare current contamination levels to standard screening levels for each matrix sampled.

Fish Sampling – SulTRAC will collect fish tissue samples from 10 locations in four streams located on or near the CBS sites. Specific locations and fish species were chosen to maintain continuity with past sampling activities. The following samples will be collected by SulTRAC:

- Up to 33 samples (plus associated laboratory QA/QC samples) to be analyzed for PCBs as congeners
- Up to 231 samples (plus associated laboratory QA/QC samples) to be analyzed for PCBs as Aroclors
- Up to 264 samples (to be obtained from same samples as above) (plus associated laboratory QA/QC samples) to be analyzed for lipid content

Sediment Sampling – SulTRAC will collect up to 33 sediment samples (plus associated laboratory QA/QC samples) from up to 30 sample locations. All samples will be analyzed for PCBs as Aroclors by the subcontracted laboratory. Sample location and timing will be determined by the EPA WAM. The purpose of these samples is to provide up-to-date data on sediment contamination levels to compare to historic levels, and to evaluate the effectiveness of past remedial actions.

Groundwater Sampling – SulTRAC will collect up to 33 groundwater samples (plus associated laboratory QA/QC samples) from up to 30 sample locations. All samples will be analyzed for PCBs as Aroclors by the subcontracted laboratory. Samples will be obtained from existing monitoring wells. Sample locations and timing will be determined by the WAM. The purpose of these samples is to provide up-to-date data on groundwater contamination levels to compare to historic levels, evaluate the effectiveness of past remedial actions, and determine the necessity for additional groundwater treatment.

Surface Water Sampling – SulTRAC will collect up to 33 surface water samples (plus associated laboratory QA/QC samples) from up to 30 sample locations. All samples will be analyzed for PCBs as Aroclors by the subcontracted laboratory. Sample locations and timing will be determined by the WAM. The purpose of these samples is to provide up-to-date data on sediment contamination levels to compare to historic levels, and to evaluate the effectiveness of past remedial actions.

Notes:

EPA	U.S. Environmental Protection Agency
PCB	Polychlorinated biphenyl
QA/QC	Quality Assessment/Quality Control
WAM	Work Assignment Manager

QAPP WORKSHEET #18
SAMPLING LOCATIONS/IDS, SAMPLE DEPTHS, SAMPLE ANALYSES AND SAMPLING PROCEDURES TABLE

Investigation Area	Sample Location	Matrix	Sequential Sample Number	Example Identification
*Neal's Landfill – NL	B – Lower reach of Conard's Branch above Vernal Pike bridge D – 1.0 mile downstream in Richland Creek at Vernal Pike bridge F – 3.0 to 3.5 miles downstream in Richland Creek at State Route 43 bridge	Sediment – SD Surface Water – SW Groundwater – MW or PZ Fish Tissue Fillet – FTF Fish Whole Body – FWB	01	NL-1-SD-01-MMYY NL-1-SW-01-MMYY NL-1-GW-01-MMYY NL-1-FTF-01-MMYY NL-1-FWB-01-MMYY
*Lemon Lane Landfill – CC (for Clear Creek)	1 – Allen Street 2 – Country Club Rd 3 – Fluck Mill Rd 4 – Gore Rd	Sediment – SD Surface Water – SW Groundwater – MW or PZ Fish Tissue Fillet – FTF Fish Whole Body – FWB	01	CC-1-SD-01-MMYY CC-1-SW-01-MMYY CC-1-GW-01-MMYY CC-1-FTF-01-MMYY CC-1-FWB-01-MMYY
*Bennett's Dump - BD	1 – Hunter Valley Rd 2 – Acuff Rd 3 – Maple Grove Rd	Sediment – SD Surface Water – SW Groundwater – MW or PZ Fish Tissue Fillet – FTF Fish Whole Body – FWB	01	BD-1-SD-01-MMYY BD-1-SW-01-MMYY BD-1-GW-01-MMYY BD-1-FTF-01-MMYY BD-1-FWB-01-MMYY

Notes:

*	See Table 4 in FSP (SulTRAC 2014b) for details on sampling locations, targeted fish species, types of samples, required weight, and sample preparation
MMYY	Two-digit month; two-digit year formatting

QAPP WORKSHEET #19
ANALYTICAL METHODS, CONTAINERS, PRESERVATIVES, AND HOLDING TIMES TABLE

Matrix	Analytical Group	Analytical and Preparation Method	Containers (number, size, type)	Preservation Requirements (chemical, temperature, etc.)	Maximum Holding Time (preparation/analysis)[1]
Fish Tissue	PCBs as Aroclors	U.S. EPA Method SW-846 8082	Wrap each fish in extra-heavy duty aluminum foil, place in waterproof plastic bag	Cool with ice (maximum shipping time 24 hours) OR Freeze with dry ice (maximum shipping time 48 hours)	Extract within 14 days (up to 1 year if frozen), and analyze within 40 days after extraction
Fish Tissue	PCBs as congeners	U.S. EPA Method 1668A	Wrap each fish in extra-heavy duty aluminum foil, place in waterproof plastic bag	Cool with ice (maximum shipping time 24 hours) OR Freeze with dry ice (maximum shipping time 48 hours)	Extract within 14 days (up to 1 year if frozen), and analyze within 40 days after extraction
Fish Tissue	Percent lipids	Subcontracted Laboratory internal SOP	Wrap each fish in extra-heavy duty aluminum foil, place in waterproof plastic bag	Cool with ice (maximum shipping time 24 hours) OR Freeze with dry ice (maximum shipping time 48 hours)	Extract within 14 days (up to 1 year if frozen), and analyze within 40 days after extraction
Groundwater	PCBs as Aroclors	U.S. EPA Method SW-846 8082	Two 1-L amber glass containers with PTFE-lined lid	Cool to ≤ 4 (±2) ºC	Extract within 7 days and analyze within 40 days after extraction
Surface Water	PCBs as Aroclors	U.S. EPA Method SW-846 8082	Two 1-L amber glass containers with PTFE-lined lid	Cool to ≤ 4 (±2) ºC	Extract within 7 days and analyze within 40 days after extraction
Sediment	PCBs as Aroclors	U.S. EPA Method SW-846 8082	One 250-mL wide-mouth glass container with PTFE-lined lid	Cool to ≤ 4 (±2) ºC	Extract within 7 days and analyze within 40 days after extraction

Notes:

1	Holding time is applicable from validated time of sample receipt and is measured to time of sample extraction and analysis.
°C	Degrees Celsius
L	Liter
mL	Milliliter
PCBs	Polychlorinated biphenyls
PTFE	Polytetrafluoroethylene
SOP	Standard Operating Procedure

QAPP WORKSHEET #20
FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

Matrix	Analytical Group	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Samples	No. of Field Duplicates[2]	No. of MS/MSDs[3]	No. of Equipment Rinsates[4]	Total No. of Samples to Laboratory
Fish Tissue	PCBs as Aroclors	A-1	10	210	21	11	1 per day of sampling if necessary	242
Fish Tissue	PCBs as congeners	A-2	10	30	3	2	NA	35
Fish Tissue	Lipid content	A-3	10	240	0	0	NA	240
Surface Water	PCBs as Aroclors	A-1	Up to 30	30	3	Up to 7	0	Up to 40
Groundwater	PCBs as Aroclors	A-1	Up to 30	30	3	Up to 7	1 per day of sampling if necessary	Up to 40
Sediment	PCBs as Aroclors	A-1	Up to 30	30	3	Up to 7	1 per day of sampling if necessary	Up to 40

Notes:

[1] Analytical and preparation SOPs are listed in Worksheet #23.

[2] Field duplicates are collected at a rate of 1 per 10 investigative samples of the same matrix.

[3] MS/MSD samples are collected at a rate of 1 per 20 investigative samples of the same matrix. MS/MSDS consist of extra sample volume and are included in the total number of samples.

[4] Rinsate samples will be collected if any sampling equipment is not dedicated or disposable. When this is the case, 1 equipment rinsate sample will be taken per day to verify quality of decontamination procedures.

MS/MSD	Matrix spike/Matrix spike duplicate
PCB	Polychlorinated biphenyl
SOP	Standard operating procedure

QAPP WORKSHEET #21
PROJECT SAMPLING SOP REFERENCES TABLE

Reference Number	Title, Revision, Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
S-1	General Equipment Decontamination, SOP 002-3	SulTRAC	Scrub brushes, large wash tubs or buckets, Alconox, distilled water	N	None
S-2	Sediment and Sludge Sampling, SOP 006-4	SulTRAC	Stainless-steel scoop or trowel, waders/rubber boots, boat, bottom dredge sampler, nylon rope, stainless steel bowls/trays, sample containers and labels.	N	None
S-3	Surface Water Sampling, SOP 009-4	SulTRAC	Sample bottles and labels	N	None
S-4	Groundwater Sampling, SOP 010-4	SulTRAC	Socket wrench, hammer, graduated cylinder/bucket, Teflon/stainless-steel bailer, submerged/peristaltic pump, sample containers and labels, wastewater containers	N	None
S-5	Static Water Level, Total Well Depth, and Immiscible Layer Measurement, SOP 014-01	SulTRAC	Water level indicator	N	None
S-6	Recording of Notes in Field Logbook, SOP 024-1	SulTRAC	Not applicable	N	None
S-7	Field Measurement of Groundwater Indicator Parameters, SOP 061-2	SulTRAC	Single or multiple parameter water quality measuring system, calibration solutions, distilled water, rinse bottle, waste container	N	None
S-8	IDEM. 1992, Revision 1. Section 4, Standard Operating Procedures for Fish Collections, Use of Seines, Electrofishers, and Sample Processing.	IDEM	Electrofishing unit, generator, nets, waders, small boat, buckets	N	None

Notes:
SOPs can be found in Attachments B and D of FSP (SulTRAC 2014b)

IDEM	Indiana Department of Environmental Management
SOP	Standard operating procedure

QAPP WORKSHEET #22
FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

Field Equipment	Calibration Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference	Comments
Multiparameter Water Quality Meter[1,2]	Oxidation-reduction potential: 2 standard solutions pH: 2 standard solutions Conductivity: 1 standard solution Temperature: no standard solution Turbidity: 2 standard solutions Dissolved oxygen: 2 standard solutions	Daily before first field measurement and after final field measurement	± 10 millivolts ± 0.01 pH unit ± 3% ± 0.1 °C ± 10% ± 10%	Repeat calibration; correct measurements for drift if necessary	Field team leader or field team members	Field Measurement of Groundwater Indicator Parameters, SOP 061-2	See note below
Water level meter	None required	NA	NA	NA	Field team leader or field team members	Static Water Level, Total Well Depth, and Immiscible Layer Measurement, SOP 014-1	NA

Notes:

SulTRAC will measure water temperature, pH, turbidity, dissolved oxygen, and specific conductance in purged groundwater until these parameters have stabilized within the identified tolerance.

[1] The field equipment will be calibrated per manufacturer's instructions.
[2] Standard solutions will be provided by the vendor to calibrate this instrument.

°C	Degrees Celsius
NA	Not applicable
SOP	Standard operating procedure

QAPP WORKSHEET #23
ANALYTICAL SOP REFERENCES TABLE

Reference Number	Title, Revision, Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
A-1	U.S. EPA SW-846 8082A (EPA 2007) and TestAmerica SOP PT-GC-005, Rev. 4	Definitive	PCBs as Aroclors (fish tissue, groundwater, surface water, sediment)	GC	Subcontracted Laboratory	No
A-2	U.S. EPA Method 1668B (EPA 2008) and TestAmerica SOP KNOX-ID-0013	Definitive	PCBs as congeners (fish tissue)	HRGC/HRMS	Subcontracted Laboratory	No
A-3	TestAmerica Laboratory SOP # PT-OP-011	Definitive	Lipid content (fish tissue)	Analytical balance	Subcontracted Laboratory	No

Notes:
Laboratory SOPs can be found in Attachment C of FSP (SulTRAC 2014b)

EPA	U.S. Environmental Protection Agency
GC	Gas chromatograph
HRGC/HRMS	High resolution gas chromatograph/high resolution mass spectrometer
PCBs	Polychlorinated biphenyls
SOM	Superfund Organic Methods
SOW	Statement of work

QAPP WORKSHEET #24
ANALYTICAL INSTRUMENT CALIBRATION TABLE

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
Gas Chromatograph/Electron Capture Detector (GC/ECD)	See A-1	After initial calibration, a check standard of Aroclor 1016/1260 is analyzed after every 10 samples and at the end of the sequence	Initial calibration, RSD for 1016/1260 ≤ 20 % or linear regression > 99 percent Continuing calibration standard ≤ 15 %D	Inspect system, correct problem, and rerun calibration and affected samples.	Analyst	See A-1
High Resolution Gas Chromatograph /High Resolution Mass Spectroscopy (HR GC/MS)	See A-2	Analyze calibration verification standard at start of every 12-hour sequence	Ion abundance ratios, relative retention time within method criteria; signal/noise ratios > 10; relative response factors 65 to 135 % of initial calibration	Inspect system, correct problem, and rerun calibration	Analyst	See A-2
Laboratory Balance	Professional service contract	At least once per year	Measurements within limits for standard class of weights used	Professional service	Contractor	See A-3

Notes:

[1] See Worksheet #23 for identification of analytical methods.

%D	Percent difference
ECD	Electron Capture Detector
GC	Gas chromatograph
MS	Mass Spectroscopy
RSD	Relative standard deviation
SOP	Standard operating procedure

QAPP WORKSHEET #25
ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE TESTING, AND INSPECTION TABLE

Instrument/ Equipment	Maintenance Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person for Corrective Action	SOP Reference[1]
GC/ECD	Daily instrument performance check	Inspect the system for leaks, correct settings, and so on.	Daily before use	As detailed in laboratory SOP	Correct problems and, if necessary, re-calibrate instrument.	Laboratory Analyst	See A-1
HR GC/MS	Daily instrument performance check	Inspect the system for leaks, correct settings, and so on.	Daily before use	As detailed in laboratory SOP	Correct problems and, if necessary, re-calibrate instrument.	Laboratory Analyst	See A-2
Laboratory Balance	Daily instrument performance check	Inspect for cleanliness; check performance with standard check weight in range to be used	Daily before use	Check weight within standard limits	Correct problems and, if necessary, call for outside service	Laboratory analyst	See A-3

Note:

ECD	Electron Capture Detector
GC	Gas chromatograph
HR	High resolution
MS	Mass Spectroscopy
SOP	Standard operating procedure

QAPP WORKSHEET #26
SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field sampling personnel/SulTRAC
Sample Packaging (Personnel/Organization): Field sampling personnel/SulTRAC
Coordination of Shipment (Personnel/Organization): Field sampling personnel, sample custodian/SulTRAC
Type of Shipment/Carrier: Cooler packed with ice and packing material such as bubble wrap/FedEx or other overnight courier (tissue, liquid, and solid samples)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Subcontracted laboratory personnel
Sample Custody and Storage (Personnel/Organization): Subcontracted laboratory personnel
Sample Preparation (Personnel/Organization): Subcontracted laboratory personnel
Sample Determinative Analysis (Personnel/Organization): Subcontracted laboratory personnel
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): See Worksheet # 27
SAMPLE DISPOSAL
Personnel/Organization: Subcontracted laboratory personnel
Number of Days from Analysis: TBD (or in accordance with subcontracted laboratory SOP)

Notes:

SOP	Standard operating procedure
TBD	To be determined

QAPP WORKSHEET #27
SAMPLE CUSTODY REQUIREMENTS

Sample packaging and shipment procedures

1- Immediately after sample collection, sample containers will be labeled with the appropriate identifiers. Clear tape will be placed over the sample containers' labels to prevent smearing.
2- The samples will be placed in Ziploc plastic bags and then in a cooler. The cooler will be packed with sufficient ice to maintain an internal temperature of 4+2 °C. The cooler will remain in a secured area or in view of the sampler until it is properly sealed for shipment to the laboratory.
3- Prior to shipping, the chain-of-custody (COC) forms, airbills, and all other relevant documents will be completed. COC forms will be sealed in plastic bags and taped to the inside of the cooler lid. Cushioning material, such as bubble-wrap, will be placed in the cooler.
4- The shipping cooler will then be sealed with tape and custody seals in a manner that will indicate whether the cooler was opened. The preferred procedure includes placement of custody seals at diagonally opposite corners of the cooler. The custody seals will be covered with clear plastic tape or strapping tape.

The field sampler is personally responsible for the care and custody of the samples until they are transferred to other personnel or properly dispatched to an overnight carrier or directly to a laboratory. When transferring possession of the samples, the individuals relinquishing and receiving the samples sign, date, and note the time of transfer on the COC form. Commercial carriers are not required to sign off on the COC form as long as the form is sealed inside the sample cooler and the custody seals remain intact.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal)

The laboratory sample custodian will receive all incoming samples and indicate receipt by signing the accompanying custody forms and retaining copies of the signed forms as permanent records. The laboratory sample custodian will record all pertinent information concerning the sample, including the persons delivering and receiving the sample, the date and time received, the method by which the sample was transmitted to the laboratory, sample condition at the time of receipt (sealed, unsealed, or broken container; temperature; or other relevant remarks), the sample identification number, and any unique laboratory identification number associated with the sample. This information should be entered into a computerized LIMS.

The laboratory will provide a secure storage area, restricted to authorized personnel, for all samples. Only the custodian can distribute samples to laboratory personnel authorized to conduct the required analyses. Laboratory analytical personnel are responsible for the care and custody of the sample upon receipt.

At the completion of sample analysis, any unused portion of the sample, together with all identifying labels, will be returned to the custodian. The returned tagged sample will be retained in secure storage until the custodian receives permission to dispose of the sample. Sample disposal will occur only on the order of the laboratory project manager in consultation with EPA or SulTRAC, or when it is certain that the information is no longer required or the samples have deteriorated. Likewise, laboratory records will be maintained until the information is no longer required and final disposition is ordered by the laboratory project manager in consultation with EPA or SulTRAC.

<u>Sample Identification Procedures</u>

Sample identification will be as described in Section 8.2 of the FSP and Worksheet 18. Each sample will also be assigned an identifying number by subcontracted laboratory software.

When the laboratory receives a sample shipment, its LIMS will generate the in-house identification numbers in accordance with its sample receipt and COC SOPs.

Notes:

COC	Chain of custody
EPA	U.S. Environmental Protection Agency
FSP	Field Sampling Plan
LIMS	Laboratory Information Management System
SOP	Standard operating procedure

QAPP WORKSHEET #28
QC SAMPLES TABLE

Matrix	Fish Tissue
Analytical Group	PCBs (congeners)
Concentration Level[a]	NA
Sampling SOP[b]	S-8
Analytical Method/ SOP Reference[c]	A-2
Sampler's Name/ Organization	SulTRAC
Analytical Organization	Subcontracted Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	**Frequency/ Number**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**DQI**	**Measurement Performance Criteria**
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias, Contamination	No target compounds > QL
LCS	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch.	Laboratory Analyst	Accuracy	%R as presented in Worksheet #12
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Surrogates	All samples	Reanalyze sample. If uponreanalysis, the monitoring compound meets criteria, report reanalysis results. If upon reanalysis, the monitoring compound does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	%R as presented in Worksheet #12

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Fish Tissue
Analytical Group	PCBs (Aroclors)
Concentration Level[a]	NA
Sampling SOP[b]	S-8
Analytical Method/ SOP Reference[c]	A-1
Sampler's Name/ Organization	TBD/SulTRAC
Analytical Organization	Subcontracted Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	**Frequency/ Number**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**DQI**	**Measurement Performance Criteria**
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias Contamination	No target compounds > QL
LCS	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch.	Laboratory Analyst	Accuracy	%R as presented in Worksheet #12
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Surrogates	All samples	Reanalyze sample. If upon reanalysis, the monitoring compound meets criteria, report reanalysis results. If upon reanalysis, the monitoring compound does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	%R as presented in Worksheet 12

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Fish Tissue
Analytical Group	Lipids
Concentration Level[a]	NA
Sampling SOP[b]	S-8
Analytical Method/ SOP Reference[c]	A-3
Sampler's Name/ Organization	TBD/SulTRAC
Analytical Organization	Subcontracted Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	**Frequency/ Number**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**DQI**	**Measurement Performance Criteria**
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias Contamination	No target compounds > QL
LCS	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias	%R as presented in Worksheet #12
LCS Duplicate	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Precision	RPD $\leq$ 25%
Sample Duplicate	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Precision	RPD $\leq$ 25%

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Sediment
Analytical Group	PCBs (Aroclors)
Concentration Level[a]	NA
Sampling SOP[b]	S-2
Analytical Method/ SOP Reference[c]	A-1
Sampler's Name/ Organization	TBD/SulTRAC
Analytical Organization	Subcontracted Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	**Frequency/ Number**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**DQI**	**Measurement Performance Criteria**
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias Contamination	No target compounds > QL
LCS	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch.	Laboratory Analyst	Accuracy	%R as presented in Worksheet #12
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Surrogates	All samples	Reanalyze sample. If upon reanalysis, the monitoring compound meets criteria, report reanalysis results. If upon reanalysis, the monitoring compound does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	%R as presented in Worksheet 12

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Water
Analytical Group	PCBs (Aroclors)
Concentration Level[a]	Low
Sampling SOP[b]	S-3, S-4
Analytical Method/ SOP Reference[c]	A-1
Sampler's Name/ Organization	TBD/SulTRAC
Analytical Organization	Subcontracted Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	**Frequency/ Number**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**DQI**	**Measurement Performance Criteria**
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias Contamination	No target compounds > QL
LCS	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch.	Laboratory Analyst	Accuracy	%R as presented in Worksheet #12
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Surrogates	All samples	Reanalyze sample. If upon reanalysis, the monitoring compound meets criteria, report reanalysis results. If upon reanalysis, the monitoring compound does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	%R as presented in Worksheet 12

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Notes:

a	NA indicates regular concentration levels are acceptable for meeting project objectives.
b	Reference number from QAPP Worksheet #21
c	Reference number from QAPP Worksheet #23

DQI	Data quality indicator
EPA	U.S. Environmental Protection Agency
LCS	Laboratory Control Sample
NA	Not applicable
QAPP	Quality assurance project plan
QC	Quality control
QL	Quantitation limit
%R	Percent recovery
RPD	Relative percent difference
TBD	To be determined

QAPP WORKSHEET #29

PROJECT DOCUMENTS AND RECORDS TABLE

Document	**Where Maintained**
Field notes/logbook (includes sampling notes and drilling logs)	Project file (field data), SulTRAC offices
Communication logs	Project file (field data), SulTRAC offices
Corrective action reports	Project file (field data), SulTRAC offices
Documentation of corrective action results	Project file (field data), SulTRAC offices
Documentation of deviation from methods	Project file (field data), SulTRAC offices
Chain of custody forms	Project file (laboratory data), SulTRAC offices
Laboratory raw data package	Project file, TestAmerica
Laboratory equipment calibration logs	Project file, TestAmerica
Validated data	Project file (laboratory data), SulTRAC offices

QAPP WORKSHEET #30
ANALYTICAL SERVICES TABLE

Matrix	Analytical Group	Concentration Level[1]	Sampling Location/ID Number	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name, Address, Contact Person, Telephone Number)	Backup Laboratory/Organization (Name, Address, Contact Person, Telephone Number)
Fish Tissue	PCBs (congeners)	NA	See Figures 2-4	A-2	20 business days	TestAmerica, Inc. 301 Alpha Drive RIDC Park Pittsburgh, PA 15238 Carrie Gamber (412) 963-7058	NA
Fish Tissue	PCBs (Aroclors)	NA	See Figures 2-4	A-1	20 business days		
Fish Tissue	Lipid Content	NA	See Figures 2-4	A-3	20 business days		
Sediment	PCBs (Aroclors)	NA	TBD by WAM	A-1	20 business days		
Groundwater	PCBs (Aroclors)	Low	TBD by WAM	A-1	20 business days		
Surface Water	PCBs (Aroclors)	Low	TBD by WAM	A-1	20 business days		

Notes:

1 NA indicates regular concentration levels are acceptable for meeting project objectives.

ID	Identification
NA	Not Applicable
PCB	Polychlorinated biphenyl
SOP	Standard operating procedure
WAM	Work Assignment Manager

QAPP WORKSHEET #31
PLANNED PROJECT ASSESSMENTS TABLE

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organization)	Person(s) Responsible for Responding to Assessment Findings (Title and Organization)	Person(s) Responsible for Identifying and Implementing CAs (Title and Organization)	Person(s) Responsible for Monitoring Effectiveness of CAs (Title and Organization)
No assessments planned	NA	NA	NA	NA	NA	NA	NA

Note:

CA	Corrective action
NA	Not applicable

QAPP WORKSHEET #32
ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES

Assessment Type	Nature of Deficiencies Documentation	Individuals Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Correct Action Response Documentation	Individuals Receiving Corrective Action Response (Name, Title, Organization)	Timeframe for Response
No assessments planned	NA	NA	NA	NA	NA	NA

Note:

NA	Not applicable

QAPP WORKSHEET #33
QA MANAGEMENT REPORTS TABLE

Type of Report	Frequency (daily, weekly, monthly, quarterly, annually, etc.)	Projected Delivery Dates	Persons Responsible for Report Preparation (Name, Title, Organization)	Report Recipients (Title and Organization)
Data Validation Letter Report	Once for each field sampling event	21 days after receipt of final laboratory data package	Jeffrey Lifka, SulTRAC, Project Manager	Thomas Alcamo, EPA Region 5 WAM
Data Evaluation Summary Report	Once for each field sampling event	45 days after receipt of all validated data	Jeffrey Lifka, SulTRAC, Project Manager	Thomas Alcamo, EPA Region 5 WAM

Note:

EPA	U.S. Environmental Protection Agency
WAM	Work assignment manager

QAPP WORKSHEET #34
VERIFICATION (STEP I) PROCESS TABLE

Verification Input	**Description**	**Internal/ External**	**Responsible for Verification (Name, Organization)**
Chain-of-custody forms	Chain-of-custody forms will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the chain-of-custody form should be initialed by the reviewer, a copy of the chain-of-custody form should be retained in the project file, and the original and remaining copies should be taped inside the cooler for shipment.	Internal	Field team leader, SulTRAC
Field notes/ logbook	Field notes will be reviewed internally and placed in the project file. A copy of the field notes may be attached to the final report.	Internal	Jeffrey Lifka, SulTRAC
Laboratory data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	Internal	Carrie Gamber, TestAmerica
	All received data packages will be verified externally in accordance with the data validation procedures specified in Worksheet #35.	External	Chemist, SulTRAC

QAPP WORKSHEET #35
VALIDATION (STEPS IIA AND IIB) PROCESS TABLE

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Chain of custody	Examine traceability of samples from sample collection to sample analysis	Chemist, SulTRAC
IIa	Holding time	Confirm that holding time requirements are met	Chemist, SulTRAC
IIa	Instrument calibration	Confirm that instrument calibration requirements are met	Chemist, SulTRAC
IIa	Analytical method	Confirm that analytical methods used are those specified in QAPP	Chemist, SulTRAC
IIb	Performance criteria	Confirm that QC samples meet specified performance criteria; document any deviations in data evaluation summary report	Chemist, SulTRAC

QAPP WORKSHEET #36
VALIDATION (STEPS IIA AND IIB) SUMMARY TABLE

Step IIa/IIb	Matrix	Analytical Group	Concentration Level[1]	Validation Criteria	Data Validator (Title and Organization)
IIa	Fish Tissue	PCBs (congeners)	NA	NFG	Chemist, SulTRAC
		PCBs (Aroclors)	NA	NFG	Chemist, SulTRAC
		Lipid Content	NA	NFG	Chemist, SulTRAC
IIa	Sediment	PCBs (Aroclors)	NA	NFG	Chemist, SulTRAC
IIa	Groundwater	PCBs (Aroclors)	Low	NFG	Chemist, SulTRAC
IIa	Surface Water	PCBs (Aroclors)	Low	NFG	Chemist, SulTRAC

Notes:

1 NA indicates regular concentration levels are acceptable for meeting project objectives.

NFG	National Functional Guidelines
PCB	Polychlorinated biphenyl

QAPP WORKSHEET #37
USABILITY ASSESSMENT

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: A team of SulTRAC personnel will perform the data usability assessment. SulTRAC's project manager will be responsible for information in the usability assessment. The project manager will also be responsible for assigning work to the individuals who will be supporting the data usability assessment. Note that the data usability assessment will be conducted on validated data. The results of the data usability assessment will be presented in the final project report.

Precision – Results of laboratory duplicates will be presented separately in tabular format. For each duplicate pair, the RPD will be calculated for each analyte whose original and duplicate values are both greater than or equal to the QL. The RPDs will be checked against the measurement performance criteria presented in Worksheet #12. The RPDs exceeding criteria will be identified in the tables. A discussion will follow summarizing the laboratory precision results. Any conclusions about the precision of the analyses will be drawn, and any limitations on the use of the data will be described.

Accuracy/Bias – Results for laboratory method blanks and instrument blanks will be presented separately in tabular format for each analysis. Similarly, the recovery results for spiked analytes in each analysis will be evaluated. The results for each analyte will be checked against the measurement performance criteria presented in Worksheet #12. Results for analytes that exceed criteria will be identified in the tables. A discussion will follow summarizing the laboratory accuracy/bias results. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn, and any limitations on the use of the data will be described.

Sensitivity – Results for all laboratory-fortified blanks will be presented separately in tabular format for each analysis. The results for each analyte will be checked against the measurement performance criteria presented in Worksheet #12 and cross-checked against the QLs presented in Worksheet #15. Results for analytes that exceed criteria will be identified in the tables. A discussion will follow summarizing the laboratory sensitivity results. Any conclusions about the sensitivity of the analyses will be drawn, and any limitations on the use of the data will be described.

Representativeness – The large numbers of samples collected are considered representative of site conditions, as long as completeness criteria in Worksheet 12 are met.

Comparability – The results of this study will be compared with PCB concentrations in fish tissue, sediment, surface water, and groundwater samples collected during previous investigations at Neal's Landfill, Lemon Lane Landfill, and Bennett's Dump. Sampling and analytical methods selected for this study are identical to methods used in previous investigations to ensure that data will be comparable.

QAPP WORKSHEET #37 (CONTINUED)
USABILITY ASSESSMENT

Completeness – A completeness check will be performed on all data generated by the laboratories. Completeness criteria are presented in Worksheet #12. Completeness will be calculated for each analyte and individual matrix as the number of data points that meet the measurement performance criteria for precision, accuracy/bias, and sensitivity, divided by the total number of planned data points. A discussion will follow summarizing the calculation of data completeness. This discussion will note any differences between the planned sample collection (number and location) and the actual sample collection. Any conclusions about the completeness of the data for each analyte will be drawn, and any limitations on the use of the data will be described.
Describe the evaluative procedures used to assess overall measurement error associated with the project: NA
Identify the personnel responsible for performing the usability assessment: SulTRAC's chemist will review analytical data and the case narrative to assess usability of the data. SulTRAC's project manager will review QC results for samples and assess the overall usability of the data set in close consultation with the EPA WAM.
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 usability assessment will be documented in the final data evaluation summary report, which will be generated 45 days after SulTRAC receives validated data from the subcontracted laboratory.

Notes:

EPA	U.S. Environmental Protection Agency
QC	Quality control
QL	Quantification limit
RPD	Relative percent difference
WAM	Work assignment manager

REFERENCES

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SulTRAC. 2014a. "Revised Workplan, Technical Assistance for CBS Multi-Sites, Monroe County, Indiana." January 17.

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EPA. 2003. "U.S. EPA Region 5 RCRA Ecological Screening Levels." August 22. On-line address: http://www.epa.gov/reg5rcra/ca/ESL.pdf

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EPA. 2008. "Method 1668B. Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS." November. On-line address: http://water.epa.gov/scitech/methods/cwa/bioindicators/upload/2009_01_07_methods_method_1668.pdf

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FIGURES

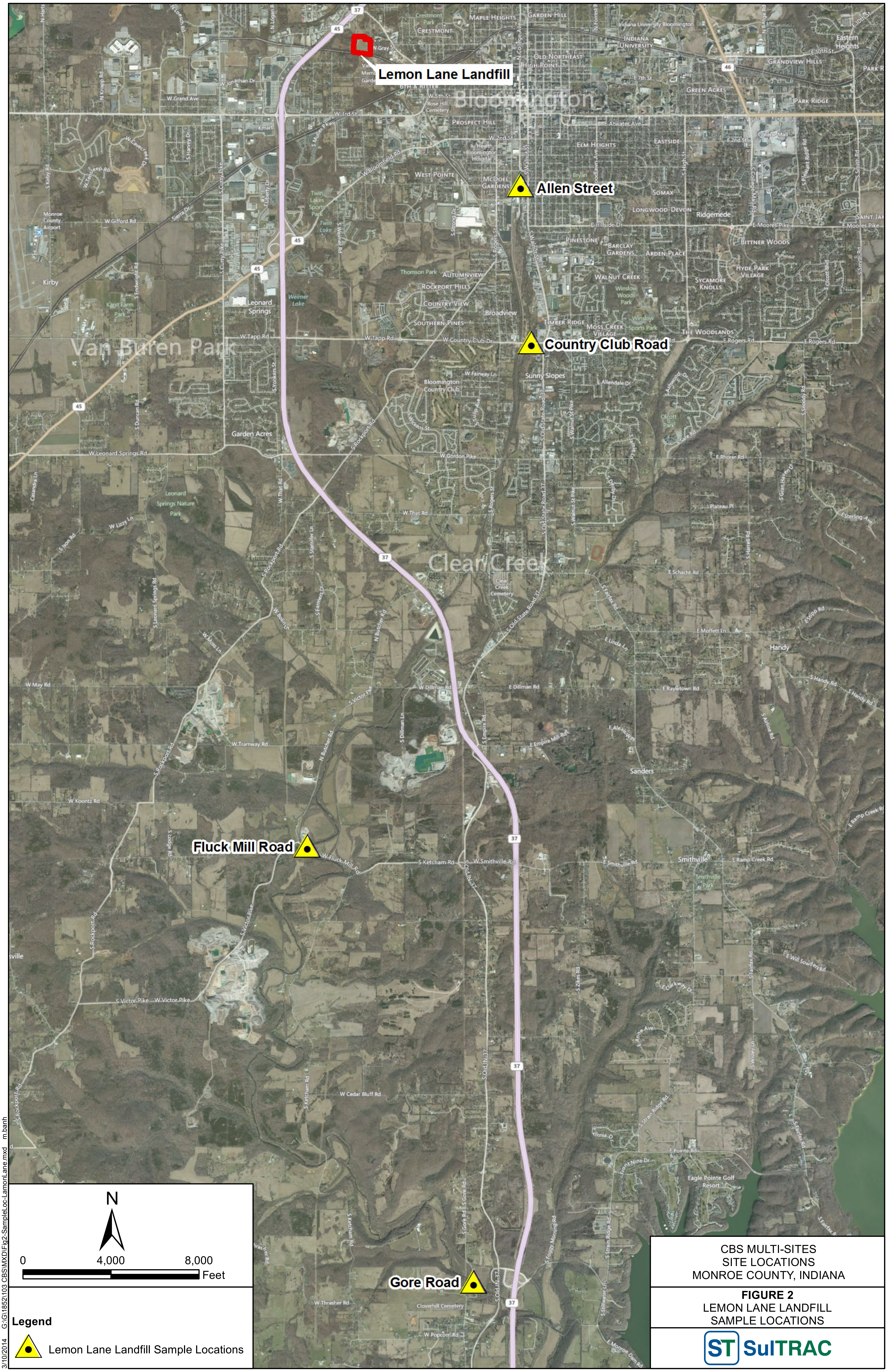
(Four Sheets)

CBS MULTI-SITES
SITE LOCATIONS
MONROE COUNTY, INDIANA

FIGURE 1
SITE LOCATIONS

ST SulTRAC

3/10/2014 G:\G\18521\03 CBS\MXD\Fig1-SiteLocations.mxd m.banh



CBS MULTI-SITES
SITE LOCATIONS
MONROE COUNTY, INDIANA

FIGURE 2
LEMON LANE LANDFILL
SAMPLE LOCATIONS

SulTRAC

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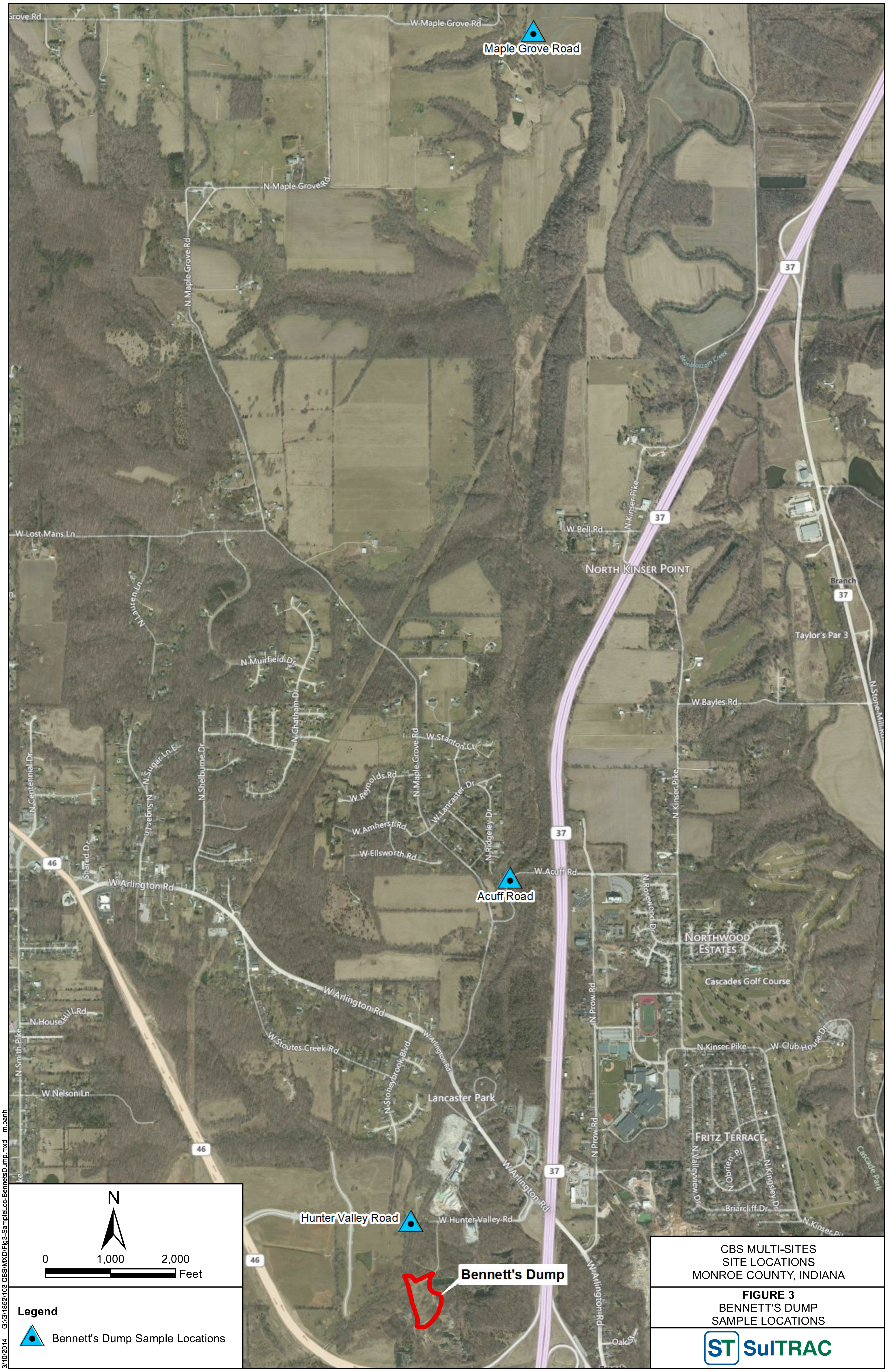

CBS MULTI-SITES
SITE LOCATIONS
MONROE COUNTY, INDIANA

FIGURE 3
BENNETT'S DUMP
SAMPLE LOCATIONS

ST SulTRAC

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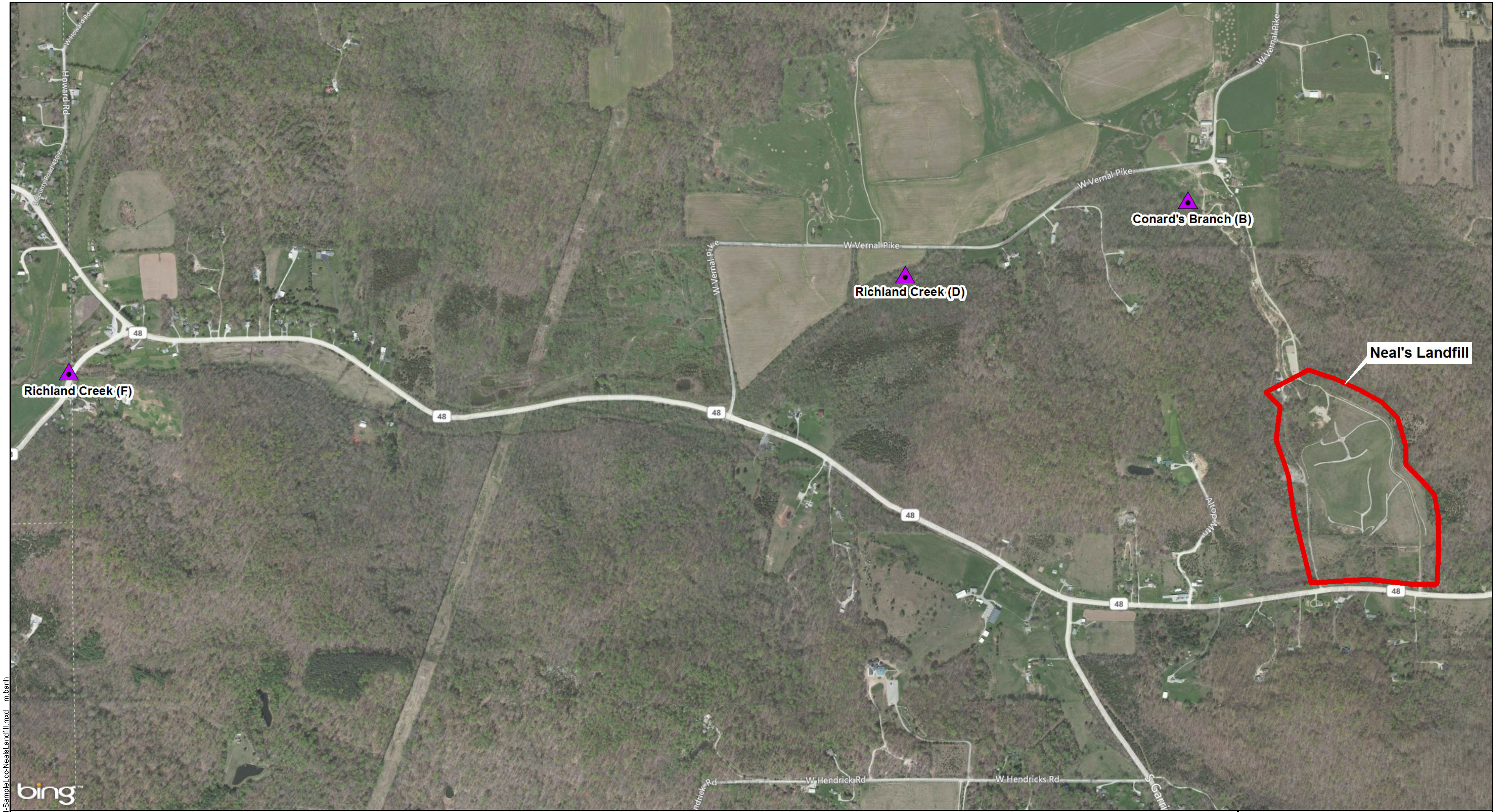

Legend

▲ Neal's Landfill Sample Locations

CBS MULTI-SITES
SITE LOCATIONS
MONROE COUNTY, INDIANA

FIGURE 4
NEAL'S LANDFILL
SAMPLE LOCATIONS

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