# Determination of Polychlorinated Biphenyls (PCBs) as Aroclors or Congeners By Gas Chromatography/Electron Capture Detection (GC/ECD)

**References:** Method 8082A, Rev. 1, February 2007 and Method 8000B Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final update III, December 1996 (USEPA, Office of Solid Waste and Emergency Response, Washington, DC).

Quality Assurance and Quality Control for SW-846 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC) for the Massachusetts Contingency Plan (MCP)

USEPA Contract Laboratory Program Statement of Work for Organic Analysis Multi-Media, Multi-Concentration" August 1991. Document Numbers OLM03.1 and OLM04.2.

Compendium Method TO10A – Determination of Pesticides and PCBs in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by GC/ECD Detection

## 1. Scope and Application

**Matrices:** Water, soil, sediment, sludge, product, PUF, and tissue (either animal or vegetable)

**Definitions:** Refer to Alpha Analytical Quality Manual.

This method is applicable to the analysis and quantification of sample extracts for Polychlorinated Biphenyls (PCBs) as Aroclors or Congeners by gas chromatography/electron capture detector. See Table I for the target compound list of PCB as Aroclors and PCB Congeners with CAS Registry Number included. The sensitivity of the method usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, the method may also be performed on samples that have undergone cleanup.

Quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment. Therefore, the 8082 method provides procedures for the determination of selected individual PCB Congeners and should be used depending on regulatory requirements and project needs. The PCB Congener approach potentially affords greater quantitative accuracy when PCBs are known to be present. The PCB Congener method is of particular value in determining weathered Aroclors. Caution should be used when using the PCB Congener method where regulatory requirements are based on Aroclor concentrations. The base list of components can be found in Table I. Additional congeners may be added to this base list of targets. Additional targets must be run under the same GC conditions to document retention times on both analytical columns. Congeners may be added without additional method proficiency demonstration.

The following extraction and cleanup methods may apply, prior to sample analysis:

- *Extraction of Water Samples by Separatory Funnel* (OP-001),
- *Microscale Solvent Extraction - 3570* (OP-016)
- *Soxhlet Extraction (OP-019)*
- *Soxhlet Extraction of PUFs (OP-020)*
- *Tissue Preparation and Homogenization* (OP-003),
- *Shaker Table Extraction* (OP-013),
- *Sulfur Cleanup* (OP-007),

- *Sulfuric Acid Cleanup* (OP-010),
- *Silica Gel cleanup* (OP-014)
- *GPC Cleanup by HPLC* (OP-006)

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC-ECD and in the interpretation of GC-ECD data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

# 2. Summary of Method

Sample extracts are analyzed on a gas chromatograph which is fitted with two capillary columns of differing polarities each employing separate detectors. The extracts of PCB Congeners are spiked with internal standard prior to analysis. The target analytes are resolved on each column and detected using an electron capture detector (ECD). Analytes are introduced into the GC/ECD by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC which is temperature and flow programmed to separate the analytes. Identification of target analytes is accomplished by confirming a target hit on two dissimilar columns using Retention Time (RT) and Pattern Recognition (PR). Concentrations are calculated from the ECD response using either external (for PCB Aroclors) or internal (PCB Congeners) standard techniques. For all PCB Aroclors, the concentrations are derived from a multi-level calibration curve, either linear using a least square regression, or non-linear.. For PCB Congeners, the concentrations are determined using mean Relative Response Factor derived from multi-level calibration curve. Alternatively, different calibration models may be employed for either Congeners or Aroclors.

## 2.1 Method Modifications from Reference

None.

# 3. Reporting Limits

Generally, the Reporting Limit for aqueous samples is 0.02 ug/L for individual Aroclors and 0.001 ug/L for individual Congeners. This value is based on a 1mL final volume and 1000 mL of the sample extracted. The Reporting Limit for solid/tissue samples is 4 ug/Kg for individual Aroclors and 0.2 ug/Kg or for individual Congeners, based on 1 mL final volume and 5 g of the sample extracted (wet weight) using extraction method 3570 (MSE). Utilizing extraction method 3540C (soxhlets) the achievable RL for client samples at a 30g extraction weight is 1.33 ug/Kg or for individual Congeners, based on 4 mL final volume. The Reporting Limit for Aroclors in PUF air samples is 20-100ng/PUF cartridge, based on the soxhlet extraction of one low volume PUF and a final volume of 1mL. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed.

# 4. Interferences

**4.1** Contaminants in solvents, reagents, glassware, syringes, and other sample processing hardware may cause inferences that lead to discrete artifacts, biases, and/or elevated baselines. Demonstrate that all of these materials are free from interferences under the conditions of the preparation and analysis by extracting and analyzing a laboratory method blank with each batch of up to 20 samples. Also new solvent lots are prescreened and approved by GC/ECD analyst prior to use. All glassware and equipment should be properly cleaned and solvent rinsed prior to use. Never use any glassware, or other implements such as syringes, on multiple samples within the batch unless there has been thorough cleaning and decontamination between samples.

**4.2** Contaminants co-extracted from the sample may cause matrix interferences. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the environment and matrix being investigated. In some cases depending on the severity of the observed interferences, a target hit on one column may be biased high when interfered with. Skilled analyst judgment is imperative in determining if a chromatographic peak appears interfered with. In these cases the non-interfered, non-biased hit can be forced for reporting purposes providing a more suitable value. In extreme cases of interference additional cleanups can be employed, or it may be necessary to analytically re-run the sample at a dilution.

**4.3** The ECD is theoretically a halogen specific detector. However, phthalate esters can be a major source of contamination in that they do respond to the ECD and can interfere with accurate determination of the analytes of interest. No forms of plastic or any material containing plasticizers (phthalates) should be used in conjunction with any part of this analysis.

**4.4** Elemental sulfur is readily extracted from soil samples and may cause chromatographic interferences in the determination of pesticides. Sulfur can be removed through the use of Method 3660B. In addition, all samples for PCB analysis are acid cleaned following Method 3665A. Associated SOPs are noted in Section 1.0, above.

**4.5** Care must be taken to ensure a complete hexane exchange is performed on the extract, because residual methylene chloride would be an interferent in the analysis. Residual methylene chloride can also damage the electron capture detectors.

**4.6** The injector system of the gas chromatograph can become contaminated with non-volatile components from samples. This can act to decrease the resolution and responsiveness of the system. Maintenance of the analytical system should be performed when system performance degrades (liner changes, injection port cleaning, etc.) to avoid prolonged down time.

**4.7** Aldol condensation products (4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) can arise during soil extraction when using methylene chloride:acetone. These products may interfere with detection of analytes and should be monitored.

**4.8** Raw GC data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

**4.9** Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. Therefore, exercise caution, and do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses, unless glassware has undergone meticulous cleaning.

**4.10** Per method 8082A each analytical column analyzing for PCB congeners must be evaluated for the presence of coelutions amongst PCB congeners with pesticides. An aliquot of PCB congeners/pesticide LCS solution is prepared and analyzed routinely on each analytical column

in the laboratory. The subsequent coelutions of these compounds are documented and stored internally in the "ECD Standard Verification Logbook" within the facility.

# 5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

# 6. Sample Collection, Preservation, Shipping and Handling

## 6.1 Sample Collection

Aqueous samples are collected in 1L or 2L amber glass bottles and stored without preservative at 4°C

1-5g of tissue sample are required.

Soil/sediment samples are collected in 4oz. glass soil jars,

Air PUF samples are collected following the guidance in reference method TO-10A Compendium.

## 6.2 Sample Preservation

Aqueous and Air PUF samples are stored without preservative at 4°C

Tissue Samples are stored at 4°C, or if desired, frozen.

Soil/sediment samples are stored at 4°C, or if desired, frozen.

## 6.3 Sample Shipping

No special shipping requirements.

## 6.4 Sample Handling

The hold time for this method is 7 days for the extraction of aqueous and Air PUF samples, and 14 days for the extraction of solid and tissue samples. If a sediment or tissue samples are frozen, this suspends the holding time until removal from the freezer. All extracts must be analyzed within 40 days of the extraction date.

# 7. Equipment and Supplies

**7.1 Gas Chromatograph:** The instrumentation is a temperature-programmable gas chromatograph with heating range from 40°C to 325°C and splitless-type inlet system with electron capture detectors (Hewlett-Packard HP 5890 Series II GC, HP 6890 Plus, or similar).

**7.1.1** The injection port is designed for splitless injection onto a capillary column. The injection port utilizes a uniliner for direct injection, to decrease the potential for breakdown of compounds in the injection port and to increase sensitivity. These uniliners are used in place of the standard splitless liners. The 6890 model requires a drilled uniliner due to Electronic pressure control. The injection port will

require maintenance on an as needed basis if degradation or contamination is apparent.

**7.1.2** Columns and supplies:

**7.1.2.1** Restek's proprietary "RTX-5" Stationary Phase, 60m length x 0.25mm ID, 0.25µm film thickness - Restek Corporation, PN 10226, or equivalent.

**7.1.2.2** Restek's proprietary "RTX-CLPesticide II" Stationary Phase, 60m length x 0.25mm ID, 0.2µm film thickness - Restek Corporation, PN 11326, or equivalent.

**7.1.2.3** Guard column, deactivated fuse silica phenyl-methyl, 5 m x 0.32 mm ID. Restek PN 10027 or equivalent.

**7.1.2.4** The flow from the guard column can be split between the two analytical columns using a Siltek MXT-Y connecter Restek PN 20396

**7.1.3** The GC/ECD utilizes one injector with guard column sealed to the uniliner inside the injection port. Connector splits the guard column into the two dissimilar columns listed above. Each column is then attached to a separate detector. Rtx-5 is always the front detector column and the CLP II is the back detector column.

**7.2 Autosampler:** Adapted onto the Gas Chromatograph. Model is HP 6890 series autosampler with a GC autosampler controller and HP 5890 series autosampler with controller, or equivalent.

**7.3 Computer:** With Windows NT operating software utilizing HP Enviroquant G1701BA Version B.01.00 software.

**7.4 Gases:** Hydrogen, ultra high purity grade (99.9999% pure) and Nitrogen of equivalent purity. Hydrogen is generally supplied by a Proton Hogen GC 600 Hydrogen generator.

**7.5 Glassware:** Assorted Class-A volumetric flasks consisting of 5mL through 200mL flasks with ground-glass stoppers for preparing reagents, standards, and measuring sample volumes for sample requiring dilution beyond 1:100.

**7.6 Pipettes:** Disposable Pasteur brand, or equivalent.

**7.7 Cahn balance:** Capable of accurate measurement to the nearest 0.001 mg.

**7.8 Analytical balance:** Capable of accurate measurement to the nearest 0.0001 g.

**7.9 Top-loading analytical balance:** Capable of accurate measurement to the nearest 0.1g.

**7.10 Micro syringes:** 10, 25, 50, 100, 250, 500, and 1000 µL.

**7.11 VOA vials:** Non-preserved, certified, pre-cleaned VOA vials, 40 mL, with Teflon-lined screw caps.

**7.12 Vials:** 2 mL, with Teflon-lined crew caps for standard solutions (amber) and sample extracts (clear).

**7.13 Spatula:** Stainless steel.

# 8. Reagents and Standards

**8.1 Solvents:** All solvent expirations determined as indicated by manufacturer guidelines.

**8.1.1 Methylene Chloride,** ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (G-008) for additional details regarding solvent purity.

**8.1.2 Acetone,** ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (G-008) for additional details regarding solvent purity.

**8.1.3 Hexane,** ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (G-008) for additional details regarding solvent purity.

**8.1.4 Methanol,** Purge and Trap grade, see SOP *Reagent, Solvent, and Standard Control* (G-008) for additional details regarding solvent purity.

**8.2 Primary Standard Solutions:** All Primary standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated primary solution is opened, it is transferred to a labeled amber 2mL vial.

Primary Standards are stored as indicated by the vendor, away from light when not in use. Primary standards are discarded as indicated by the vendor expiration. If breakdown of a solution is observed, the solution will be discarded.

**8.2.1 Aroclor 1016 Solution:** Obtained from Accustandard at a concentration of 100ug/mL (catalog # C-216S-H-10X-PAK) – for the calibration curve and obtained from Restek at a concentration of 1000 ug/mL (catalog # 32006) – for LCS and ICV.

**8.2.2 Aroclor 1260 Solution:** Obtained from Accustandard at a concentration of 100ug/mL (catalog # C-260S-H-10X-PAK) – for the calibration curve and obtained from Restek at a concentration of 1000 ug/mL (catalog # C32012) – for LCS and ICV.

**8.2.3 Aroclor Solution:** Obtained from Ultra at a concentration of 100 ug/mL for one-point calibration.

| Compound | Part Number |
|---|---|
| Aroclor 1221 | PP-291 |
| Aroclor 1232 | PP-301 |
| Aroclor 1242 | PP-311 |
| Aroclor 1248 | PP-341 |
| Aroclor 1254 | PP-351 |
| Aroclor 1262 | PP-371 |
| Aroclor 1268 | PP-381 |

**8.2.4 Pesticides Surrogate Standard Spiking Solution:** Obtained from Ultra at a concentration of 200ug/mL (catalog # ISM-320) – for PCB Aroclors analysis.

**8.2.5 4,4'-Dibromooctafluorobiphenyl Solution-DBOB:** Obtained from Ultra at a concentration of 5000 ug/mL (catalog # PPS-172) – for PCB Congeners analysis.

**8.2.6 2,2',3,3',4,5,5',6-Octachlorobiphenyl Solution-BZ#198:** Obtained from Ultra at a concentration of 100 ug/mL (catalog # RPC-075S) – for PCB Congeners analysis.

**8.2.7 Internal Standard Solution:** 2,3,3',4,5,5',6 Heptachlorobiphenyl Solution-BZ#192 –obtained from Ultra at a concentration of 100 ug/mL (catalog # RPC-165S) – for PCB Congeners analysis.

**8.2.8 Custom PCB Standard:** Obtained from Accustandard at a concentration of 100 ug/mL (catalog # S-7911-2X) – for the calibration curve, for PCB Congeners analysis.

**8.2.9 EPA PCB Congener Calibration Check Solution:** Obtained from Ultra at a concentration of 0.2ug/mL (catalog # RPC-EPA) – for ICV, for PCB Congeners analysis.

**8.3 Stock Standard Solutions:** All Stock Standard Solutions are prepared volumetrically by diluting and mixing the appropriate primary standard solutions in hexane. Stock standards expire 1 year from the date of preparation or sooner if routine QC tests indicate a problem. All stock solutions are stored in the freezer (-20$^0$C÷ -10 $^0$C) in 10÷40 mL vials depending on the amount prepared. If breakdown of a solution is observed, the solution will be discarded.

**8.3.1 Aroclor1660 Calibration Stock Solution:** Consists of the Aroclor 1016 and 1260 from Ultra or equivalent, mixed with the extraction surrogates (TMX and DCB), at a stock concentration of 10 000ug/L for Ar1660 and 1000ug/L for surrogates, in hexane. To prepare calibration stock solution, add 250uL of *Aroclor 1016/1260 Primary Solution* at 1000ug/mL and 125uL of *Pesticides Surrogate Standard Spiking Solution* at 200ug/mL to a 25mL volumetric flask. Dilute to the volume with hexane.

**8.3.2 NOAA 22 Congeners Stock Solution:** Consists of the NOAA 22 Congeners from Accustandard or equivalent, at the concentration of 5000ug/L in hexane. To prepare congeners stock solution, add 1.25mL of *Custom PCB Standard* at 100ug/mL to a 25mL volumetric flask. Dilute to the volume with hexane.

**8.3.3 Pesticides Surrogate Stock Solution:** Consists of Pesticides Surrogates (TMX and DCB) from Ultra or equivalent, at a stock concentration of 5000ug/L. To prepare pest surrogate stock solution add 0.625 mL of *Pesticides Surrogate Standard Spiking Solution* at 200ug/L to a 25mL volumetric flask. Dilute to the volume with hexane.

**8.3.4 DBOB and BZ 198 Surrogate Stock Solution:** Consists of DBOB and BZ198 from Ultra or equivalent, at a stock concentration of 5000ug/L, in hexane. To prepare surrogate stock solution add 25uL of *DBOB Primary Solution* at 5000ug/mL and 1.25mL of *BZ#198 Primary Solution* at 100ug/mL to a 25mL volumetric flask. Dilute to the volume with hexane.

**8.4 Working/Spiking Standard Solutions:** All working/spiking standard solutions are prepared volumetrically by diluting and mixing appropriate primary or stock standard solutions in acetone, hexane or methylene chloride. Working/Spiking standards expire 6 months from the date of preparation or sooner if routine QC tests indicate the problem. All working/spiking solutions are stored in the freezer (-20$^0$C÷ -10 $^0$C) in 10÷40mL vials depending on the amount prepared. All spiking solutions must be assayed for use by analysis before release to the

preparation lab. All compounds must be within 20% of their true value. If breakdown of a solution is observed, the solution will be discarded.

**8.4.1 Aroclor 1660 calibration standard solution:** Consists of Aroclor 1016 and 1260 from our "Aroclor 1660 **calibration stock solution"** combined into one standard called Aroclor 1660 and the extraction surrogates (TMX and DCB) through dilution of Aroclor 1660 Calibration Stock solution. The extraction surrogates will only be combined with the 1660 calibration. An ICAL is run for each of the nine Aroclors before analysis. A minimum of 5 levels is analyzed for the curve although more levels may be analyzed. This Curve preparation will be followed for all nine Aroclors, minus the surrogates.

**7-Level Curve Preparation for Aroclors**

| Calibration Level | Aroclor Conc. | Surrogate Conc. | Volume of Std Added | Final Volume in Hexane |
|---|---|---|---|---|
| Stock | 10 00μg/L | 1000μg/L | 25uL of Ar1660-1μg/mL<br>250μL of surrogate-200μg/mL | 25mL |
| Level1 | 20μg/L | 2μg/mL | 20uL of Stock. | 10mL |
| Level 2 | 50μg/L | 5μg/L | 50uL of Stock | 10mL |
| Level 3 | 100μg/L | 10μg/mL | 100uL of Stock | 10mL |
| Level 4 | 250μg/L | 25μg/mL | 250uL of Stock | 10mL |
| Level 5** | 500μg/L | 50μg/mL | 1.25mL of Stock | 25mL |
| Level 6 | 1000μg/L | 100μg/mL | 1mL of Stock | 10mL |
| Level 7 | 2000μg/L | 200μg/mL | 2mL of stock | 10mL |

** CCV Level

**8.4.2 Congener Calibration standard solution:** Consist of 22 NOAA Congeners and the extraction surrogates (DBOB and BZ#198) through dilution of *NOAA 22 Congeners Stock Solution* listed in *8.3.2.* and *DBOB and BZ 198 Surrogate Stock Solution* listed in *8.3.4.* 8 levels are described, however a minimum 5 levels are required for initial calibration.

**Multi-Level Calibration Solution Preparation for Congeners and Surrogates**

| Calibration Level | Analyte Conc. ug/L | Surrogate Conc. ug/L | Volume of Std Added | Volume in Hexane |
|---|---|---|---|---|
| Level 1 | 1 | 1 | 0.05mL of Level 8 | 10mL |
| Level 2 | 5 | 5 | 0.25mL of Level 8 | 10mL |
| Level 3 | 10 | 10 | 0.50mL of Level 8 | 10mL |
| Level 4 | 20 | 20 | 0.04mL of Cong stock and 0.04mL of Surr. stock | 10mL |
| Level 5 | 50 | 50 | 0.1mL of Cong stock and 0.1mL of Surr. stock | 10mL |
| Level 6** | 100 | 100 | 0.5mL of Cong stock and 0.5mL of Surr. stock | 25mL |
| Level 7 | 150 | 150 | 0.3mL of Cong stock and 0.3mL of Surr. stock | 10mL |
| Level 8 | 200 | 200 | 0.4 mL of Cong stock and 0.4 mL of Surr. stock | 10 mL |

** CCV Level

**8.4.3 PEST/PCB Surrogate Spike Solution:** TMX and DCB obtained from Ultra Scientific or equivalent, at a concentration of 100ug/L ("ULOW") and/or 1000ug/L ("LOW") in Acetone. To prepare:

- "ULOW" surrogate spiking solutions add 5mL of *Pesticides Surrogate Stock Solution* (at 5000ug/L) into a 250mL volumetric flask and dilute to the volume with acetone.
- "LOW" surrogate spiking solutions add 1mL of *Pesticides Surrogate Standard Spiking Solution* (at 200ug/mL) into a 200mL volumetric flask and dilute to the volume with acetone.

All samples, blank and matrix spike are spiked with 1mL of the surrogate solution prior to extraction to monitor efficiency of sample extraction, chromatographic and calibration system. This amount may be adjusted to meet project specific concentrations, as needed.

**8.4.4 PEST/CONG Surrogate Spike Solution:** DBOB and BZ 198 obtained from Ultra or equivalent, at a concentration of 100ug/L in Acetone. To prepare surrogate spiking solutions add 5mL of DBOB and BZ 198 Surrogate Stock Solution (at 5000ug/L) into a 250mL volumetric flask and dilute to the volume with acetone. All samples, blank and matrix spike are spiked with 1mL of the surrogate solution prior to extraction to monitor efficiency of sample extraction, chromatographic and calibration system. This amount may be adjusted to meet project specific concentrations, as needed.

**8.4.5 PCB Aroclors LCS/LCSD/MS/MSD:** Aroclor 1660 commercially obtained from Accustandard or equivalent, at the concentration of 1000ug/L ("ULOW") and/or 10,000ug/L ("LOW") in Acetone. This is a source separate from the calibration curve. To prepare:

- "ULOW" spiking solution add 0.1mL of each primary solution at 1000µg/mL to a 100mL volumetric flask and bring it to volume with Acetone.
- "LOW" spiking solution add 1mL of each primary solution at 1000µg/mL to a 100mL volumetric flask and bring it to volume with Acetone.

1mL is spiked into each QC and field QC sample. This amount may be adjusted to meet project specific concentrations, as needed.

**8.4.6 NOAA 22 Cong LCS/LCSD/MS/MSD:** NOAA 22 Congeners obtained from Ultra or equivalent, at the concentration of 100ug/L in Acetone. To prepare spiking solution add 1mL of NOAA 22 Congeners Stock Solution at 5000µg/L to a 50mL volumetric flask and bring it to volume with Acetone. 1mL is spiked into each QC and field QC sample. This amount may be adjusted to meet project specific concentrations, as needed.

**8.4.7 Independent Verification Standard (ICV) for Aroclors:** This is a source separate from the calibration curve containing one of the nine Aroclors,,, obtained from Ultra or Restek, at the concentration of 100ug/mL or 1000ug/mL. To prepare add 250uL to 25mL or add 250uL to 25mL volumetric flask and bring to volume with hexane. This will achieve the standard concentration of 1000µg/L. To get working STD (500ug/L) Add 5mL of the stock (1000ug/L) to a 10mL volumetric fask and bring up to volume with hexane.

**8.4.8 Independent Verification Standard (ICV) for Congeners:** This is a source separate from the calibration curve obtained from Ultra, at the concentration of

100ug/mL in hexane. This standard must be analyzed with the appropriate concentration of the internal standard.

**8.4.9** **Internal Standards (IS): BZ#192:** Prepared so that a 20-uL spiked into the sample extract prior to analysis, will produce a mid-calibration range response at 50ng/mL in final sample extract. To prepare add 0.625mL of primary solution at 100ug/mL to a 25mL volumetric flask and bring it to volume with hexane. Final concentration is 2.5ug/mL.

# 9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Quality Control (QC) samples are necessary to monitor both the sample extraction and instrument analysis procedures. The Quality Control samples described below are considered the method defaults, and are the minimum requirements, except where noted. Client and Project specific Data Quality Objectives (DQOs) supersede the requirements in this section where applicable. Client or Project specified DQOs shall be included, or referenced, in the final report to the client.

## 9.1 Blank(s)

**9.1.1** A method blank must be extracted (spiked with surrogates and/or internal standards) and analyzed once per every 20 samples or per extraction batch, whichever is more frequent. The analysis of the method blank extract will demonstrate the background contamination.

**9.1.2** An *acceptable* method blank should not contain any individual compound at the concentration of reporting limit, or above. All efforts must be made to identify and eliminate the source of contamination. The presence of analytes at concentrations at or above the reporting limit will warrant application of a “B” qualifier to that target compound(s) on all associated report forms, and perhaps re-extraction of all associated samples. The results are qualified with a “B” for any associated sample concentrations that are less then 5x the blank concentration for the analyte. Surrogate and internal standard recoveries must meet the QC limits for the method blank, see Sections 12.5 and 12.6. Re-extraction must be initiated immediately so that the minimum of time is wasted before re-extraction can occur - if at all possible, this re-extraction should take place within holding time. Re-extraction *corrective action* that would exceed the sample holding time criteria should be discussed with the client, Laboratory Director, QA Manager, and/or Section Supervisor prior to implementation. Exceptions may be made with approval of the Section Supervisor if the samples associated with an out of control method blank are non-detect for the affected compound(s) or if the concentrations of the affected compound(s) are greater than 5x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified, via the project narrative, of any method blank non-compliance associated with the sample results. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

## 9.2 Laboratory Control Sample / Laboratory Control Sample (LCS / LCSD)

**9.2.1** The LCS/ LCSD are extracted along with the samples as a measure of precision and accuracy. At a minimum, one LCS/LCSD sample set must be analyzed at a frequency of once per 20 samples, or per analytical batch, whichever is more frequent. The LCS contains Aroclor 1660 for PCS as Aroclors analysis and all 22 NOAA Congeners for PCB Congeners analysis.

**9.2.2** The acceptable recovery QC limits are found in Section 12 for an aqueous, solid, and tissue LCS.

**9.2.3** Acceptable relative percent difference (RPD) for LCS/LCSD analysis is ± 50 % for both aqueous and solid matrices. See section 12 for QA acceptance criteria. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

**9.2.4** Corrective Action: Repeat analysis or check to see if an analytical error has occurred. If the LCS recovery is still out of control, re-extract and re-analyze the LCS and all samples associated with that LCS/LCSD. If the LCS/LCSD do not meet quality control criteria for recovery of some of the spike and surrogate compounds, the results for the other samples and quality control samples within the batch must be evaluated to determine if this is an isolated problem for the LCS/LCSD and whether the data should be reported with the affected LCS/LCSD. If the recoveries for all components in the LCS/LCSD are outside of criteria, the entire analytical batch should be re-extracted and re-analyzed. Exceptions may be made with approval of the Department Manager if the samples associated with the out of control LCS/LCSD are also associated with a matrix spike and matrix spike duplicate that is in control which demonstrates an isolated problem pertaining to the LCS/LCSD only. An explanation of this out of control LCS/LCSD recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS/MSD results as batch QC. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

## 9.3 Initial Calibration Verification (ICV)

Refer to section 10.2

## 9.4 Continuing Calibration Verification (CCV)

Refer to section 10.4

## 9.5 Matrix Spike / Matrix Spike Duplicate (MS / MSD)

**9.5.1** MS/MSD analyses are performed once per 20 samples (5% frequency) only per client request. It is preferable to extract samples that have been selected specifically by the client. If none have been assigned then the laboratory analyst must choose a representative sample for each type of matrix prepared. The MS/MSD must be matrix specific. Aqueous and air PUF MS/MSD cannot be done unless client supplies sufficient sample. These samples are also spiked

with extraction surrogate. MS/MSD are spiked with the LCS spiking solution (Aroclor 1660 or NOAA 22 Congeners) at the same level as LCS/LCSD.

**9.5.2** The acceptable recovery and RPD QC limits are found in Section 12 for an aqueous, solid, tissue and product MS/MSDs.

**9.5.3** Acceptable relative percent difference (RPD) for matrix spike analysis is ± 50 % for both aqueous and solid matrices. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

**9.5.4** Corrective Action: The recovery of the matrix spikes are evaluated relative to the what was in the unspiked sample to indicate how well the methods worked on extraction of the analytes of interest from the sample matrix. The results for the unspiked and MS/MSD samples need to be compared carefully to determine how well the spiking study worked and if there was any indication of matrix-related problems that might affect the analytical batch. Re-extraction of the sample based on the MS results is rare. Repeat analysis or check to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the control limits and the associated LCS is within control, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the affected results as evidenced by the matrix spike and matrix spike duplicate exceedance. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

## 9.6 Laboratory Duplicate

**9.6.1** Laboratory matrix or sample duplicates are analyzed if requested by the client. The client must supply sufficient additional sample volume for this duplicate. Duplicates are used to evaluate the precision of the method. The QC limit is 50% RPD for target compounds found above 5 times the reporting limit.

**9.6.2** Corrective Action: If the %RPD exceeds the 50% control limit and the associated MS/MSD %RPD is within 50%, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results isolated to this sample, as evidenced by the matrix duplicate exceedance and the MS/MSD acceptance. If both the sample/duplicate and the MS/MSD exceed the control limits, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results as evidenced by the sample/duplicate and the MS/MSD exceedences. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

## 9.7 Method-specific Quality Control Samples

### 9.7.1 Surrogates

**9.7.1.1** Surrogates are monitored for recovery for all matrices. Two surrogates are used, one that elutes at the beginning and one at the end of GC run:

TMX/DCB for Aroclors and DBOB/BZ#198 for Congeners. Every sample, blank, and quality control sample (LCS, MS, MSD, Dup) must be spiked with the surrogates prior to extraction. The recovery limits are found in Section 12.

**9.7.1.2** Corrective Action: If surrogate recoveries are not met, check to be sure there are no errors in peak integration, calculations, and surrogate solutions. In addition, if none of the above are a problem, check method and instrument performance by examining the LCS results. Re-analyze the sample. Check to see if an analytical or dilution error occurred and re-calculate. If surrogates are recovered below the limit, re-extract the sample and report the re-extract results, if re-extraction occurred beyond the holding time, and the re-extract surrogates are within the QC limits. If the surrogates are recovered below the limit in the re-extract, this confirms suspected matrix interference on the surrogates, and only the original analysis needs to be reported, unless specified otherwise by client or project data quality objectives (DQOs) or other specifications – both results of both analysis might need to be reported. If the chromatogram shows obvious matrix interference, no re-analysis or re-extraction is necessary. *This decision must be made with approval of the Department Manager.* Surrogate outliers and sample re-extracts must be noted in the case narrative to the client. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

**9.7.2 Internal Standards**

**9.7.2.1** Internal Standards will be evaluated for relative retention time drift and relative area counts compared to the associated opening CCV for PCB Congeners *only.* Internal standards are added to every field sample, QC sample, standard, and method blank. The internal standard retention times for associated samples should be ± 0.05 minutes relative to the retention times from the associated opening CCV. The area counts for the internal standards in the associated samples should be -50% to +200% of the area of the internal standards in the opening CCV.

**9.7.2.2** Corrective Action: Check to see if an analytical, dilution or spiking error occurred. If the chromatogram shows obvious matrix interference, no re-analysis is necessary. *This decision must be made with approval of the Department Manager.* Note the exceedance in the case narrative to the client. If no obvious interference is present, re-analyze the extract. If internal standards are now within the acceptance limits, report only the re-analysis, as long as the re-analysis occurred within the 40-day analytical hold time. If the re-analysis occurred outside of the 40-day analytical hold time, both the original and re-analysis must be reported. If the internal standards again are outside the acceptance limits, after re-analysis, either within or outside of the 40-day hold time, report only the original analysis, and include a narrative to the client that the suspected matrix interference on the internal standards was confirmed by sample re-analysis. If specify by the client or project data quality objectives (DQOs) or other specifications both results of both analysis might have to be reported.

#### 9.7.3 Standard Reference Materials (SRMs)

**9.7.3.1** Standard reference materials (SRMs) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project specific basis. For sediment and tissue sample batches, a certified SRM will be analyzed at the rate of one per batch of up to 20 samples per matrix for PCB Congeners *only.* These are not used as controls, but to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

**9.7.3.2** Measured results will be compared against certified values for those target analytes that are certified in these SRMs as follows. Acceptance criteria for SRM analysis will vary from project to project depending upon client data quality objectives (DQOs). Generally, ± 50% difference (%D) based on the true certified values of the target compounds of interest, or 40% - 140% recovery, serve as advisory acceptance criteria.

**9.7.3.3** Corrective Action: Repeat analysis and/or check to see if an analytical error has occurred. If the % recovery or %D still exceeds the control limits and the associated LCS/LCSD and/or MS/MSD are within control, include a project narrative with the results to client noting that the observed recovered of the SRM are isolated to this sample as evidenced by the LCS/LCSD and/or MS/MSD acceptance. Individual analyte exceedances will be narrated and may be used to assess potential interferences or system bias for individual analytes.

## 9.8 Method Sequence

An example sequence for 8082 Aroclor analysis, created 12/04 on ECD4. CCV identifiers and/or nomenclature can be associated according to date/instrument or a specifically generated WG (workgroup) reference number assigned by the company LIMS system.

**1.** Primer
**2.** Hexane
**3.** C4120401STD1242 AR1242 Level 5 @ 500ug/L *
**4.** C4120401STD1248 AR1248 Level 5 @ 500ug/L *
**5.** C4120401STD1254 AR1254 Level 5 @ 500ug/L *
**6.** C4120401STD1660 AR1660 Level 5 @ 500ug/L
**7.** C4120401STD2162 AR1262 Level 5 @ 500ug/L *
**8.** C4120401STD3268 AR1268 Level 5 @ 500ug/L *
**9.** Method Blank
**10.** Method LCS
**11.** Method LCSD
**12.** 7 client samples
**13.** C4120402STD1242 AR1242 Level 5 @ 500ug/L *
**14.** C4120402STD1248 AR1248 Level 5 @ 500ug/L *
**15.** C4120402STD1254 AR1254 Level 5 @ 500ug/L *
**16.** C4120402STD1660 AR1660 Level 5 @ 500ug/L
**17.** C4120402STD2162 AR1262 Level 5 @ 500ug/L *
**18.** C4120402STD3268 AR1268 Level 5 @ 500ug/L *
**19.** about 10 client samples
**20.** C4120403STD1242 AR1242 Level 5 @ 500ug/L *
**21.** C4120403STD1248 AR1248 Level 5 @ 500ug/L *
**22.** C4120403STD1254 AR1254 Level 5 @ 500ug/L *
**23.** C4120403STD1660 AR1660 Level 5 @ 500ug/L
**24.** C4120403STD2162 AR1262 Level 5 @ 500ug/L *
**25.** C4120403STD3268 AR1268 Level 5 @ 500ug/L *

*optional CCV levels may be employed to account for the presence of variable Aroclor patterns within client samples.

An example sequence for 8082 Congener analysis, created 12/04 on ECD3. CCV identifiers and/or nomenclature can be associated according to date/instrument or a specifically generated WG (workgroup) reference number assigned by the company LIMS system.

Primer
Hexane
C3120401STD50
Method Blank
Method LCS
Method LCSD
7 client samples
C3120402STD50
10 client samples
C3120403STD50

# 10. Procedure

## 10.1 Equipment Set-up

**10.1.1** Prior to performing GC/ECD analysis, the operator must read and become familiar with the operating procedure guidelines specified in the instrument operating manuals. The analyst must be trained and familiarized with the instrument software provided by the manufacturer. The instrument must be set up with the proper operating parameters (data acquisition and processing) and conditions described in

the operating manual. The criteria for the analytical calibration ranges and method and instrument detection limits must be established and documented prior to initial calibration. All analytical equipment is traceable to NIST via internal and external calibration checks and *Certificates of Analysis* that are received with the calibration standards.

**10.1.2 GC Instrumental Conditions**

**10.1.2.1** Inject an aliquot of 1uL into the capillary column of the gas chromatograph at the following conditions.

| Parameters | Settings for Aroclors Analysis | Settings for Congeners Analysis |
|---|---|---|
| Injector A Temp | 220°C | 220°C |
| Detector Temp | 300°C (HP) | 300°C (HP) |
| Oven Temp | 125°C | 100°C |
| Initial Value | 125°C | 100°C |
| Initial Time | 0 minute | 0 minute |
| Rate | 25°/minute to 197°C | 25°/minute to 197°C |
| Hold. Time | 0 minutes | 2.00 minutes |
| Rate | 5°/minute to 280°C | 1°/minute to 210°C |
| Hold. Time | 8.50 minutes | 0 minutes |
| Rate | - | 5°/minute to 210°C |
| Hold. Time | - | 7.12 minutes |
| Purge/Valve A ON | OFF | OFF |
| Run Time | 27.98 minutes | 40.00 minutes |
| Mode | Splitless | Splitless |

**10.1.2.2** Establish daily retention time windows as the retention time of the component in the opening verification standard ± 0.05. Retention time windows are calculated each time a new GC column is installed. It has been found that this window works well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results. The window listed above is used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms

## 10.2 Initial Calibration

**10.2.1** Before analysis of sample extracts, establish a multi-point calibration curve showing the linear range of the analysis for all nine Aroclors. If an individual Aroclor is out of calibration only that Aroclor will be re-calibrated. The ICAL for 1660 will have extraction surrogates added the solution. Each other Aroclor will have an individual curve. Exemption: 1221, 1232, 1262, and 1268 will be combined as follows 1221 and 1262, 1232 and 1268. The calibration curves are generated during initial calibration through the analysis of, at minimum, 5-level calibration standards, which define the working range of the method. See Section 8 for the preparation of the standard solutions for the initial calibration curve.

- The method of external standard calibration is used ***for Aroclors*** analysis. The response of each individual peak and the total response of the PCB in the sample are compared to a calibration curve to determine the analyte concentration in the sample.
- For each of the nine Aroclors, either a linear calibration model is employed, provided that the coefficient of determination (COD or $r^2$) is $\geq$0.99. Otherwise, nonlinear calibration of a second or third order equation can be employed. Statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approach. A quadratic model requires six standards, and a third order polynomial requires seven standards. In setting model parameters, do not force the line through the origin. The COD or $r^2$ must be $\geq$ 0.99. The analyst should select the regression order that introduces the least calibration error into the quantitation.

  *Alternatively, if average calibration factor used, %RSD must be less than 20%. If one or more peaks have %RSD greater than 20%, the calibration may still be acceptable if the average %RSD for all analytes in the calibration curve is less than 20%.

- Using the GC system software, the analyst must choose 3-5 peaks from the pattern which are characteristic of the Aroclor and are the least subject to degradation and weathering to obtain the response for the component of interest. Choose peaks that are at least 25% of the height of the largest Aroclor peak, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. Use at least 5 peaks for the AR1660, none of which should be found in both. Tabulate the peak area for each analyte against the mass injected of the analyte in the standard analyzed to obtain a calibration curve for each analyte across the working range of the method.

- Calibration Factors are determined for individual peaks. Choose 3 to 5 peaks in the standard to calculate calibration factors for each Aroclor. The calibration factors are then used to calculate the concentration of each corresponding peak in the sample. The 3 to 5 resulting concentrations are averaged to provide the final result for the Aroclor for the sample.

- The method of internal standard calibration is used ***for Congener*** analysis. The response of each individual peak relative to the internal standard respond is compared to a calibration curve to determine the analyte concentration in the sample.

- Use the initial multi-point calibration to determine Relative Response Factors (RRF) at each concentration level. Average the RRF, to generate mean RRF, for quantification of all target analytes and surrogate compounds. The RRF are based

on the internal standard compounds. Using average relative response factor, determine if the % RSD ≤ 20% over the working range of the curve for all analytes. If one or more peaks have %RSD greater than 20%, the calibration may still be acceptable if the average %RSD for all analytes in the calibration curve is less than 20%. If average %RSD > 20% a new initial calibration must be performed. Alternately, linear or non-linear regression (not forced through zero) can be used. For the calibration to be valid, the correlation coefficient must be at least 0.99.

**10.2.2** The following analytical sequence order should be followed for initial calibration of the system. The primer is used at the beginning of the analysis sequence to ensure all sites of activation in the system have been covered or deactivated prior to performing trace level analysis. The sequence below is a general sequence for determination of PCBs as Aroclors and uses a minimum of 5 levels for AR1660 and a single-point initial calibration for the remaining Aroclors near the mid-point of the expected calibration range. Construct an analytical sequence using the HP Enviroquant software:

- Click on the "Instrument" icon to open the Chem Station
- *Go into the "GC Top Environmental" screen*
- *Go into "Sequence"*
- Edit the "Sample Table Log" by entering the "Vial" number starting at position 1, the "Data File ID" same as the "Sample Name" (for the initial calibration curve the nomenclature is "I" for initial calibration "4" for instrument ID ECD4, the date, and the standard number "01, 02, etc." such as, I4120401, I4120402, etc.), the acquisition method (A412041660.m), which indicates the type of method and the date the method was created), and finally the "Miscellaneous Information" (such as, "PW120407A 20ug/L", for the first standard ID and concentration level, etc.). When complete, click "OK".
- Go back into "Sequence" and "Save" the sequence as the date, such as, C4120401.s. The ending "01" indicates the first sequence created on 12/04.
- Go back in to "Sequence" and "Print" the sequence that was just saved. This will become part of the instrument run log. See Section 15.0 for additional instrument run log details.
- Go back into "Sequence" and "Load and Run" the sequence that was just saved.

Example initial calibration sequence for Aroclor analysis, created on 11/16 on ECD4:

1. Primer
2. Hexane
3. I411611011660L1 AR1660 (PW111611O @ 20ug/L)
4. I411611011660L2 AR1660 (PW111611N @ 50ug/L) - optional
5. I411611011660L3 AR1660 (PW111611M @ 100ug/L)
6. I411611011660L4 AR1660 (PW111611L @ 250ug/L)
7. I411611011660L5 AR1660 (PW111611K @ 500ug/L)
8. I411611011660L6 AR1660 (PW111611J @ 1000ug/L)
9. I411611011660L7 AR1660 (PW11616I @ 2000ug/L)
10. HEXANE
11. I411611STD500 AR1660 (PW092711A @ 500ug/L)
12. HEXANE
13. I411611011242L1 AR1242 (PW111611X @ 20ug/L)

14. I4111611011242L2 AR1242 (PW111611W @50ug/L)
15. I4111611011242L3 AR1242 (PW111611V @100ug/L)
16. I4111611011242L4 AR1242 (PW111611U @250ug/L)
17. I4111611011242L5 AR1242 (PW111611T @500ug/L)
18. I4111611011242L6 AR1242 (PW111611S @1000ug/L)
19. I4111611011242L7 AR1242 (PW111611R @2000ug/L)
20. HEXANE
21. I4111611011242ICV AR1242 (PW052511A @500ug/L)
22. HEXANE

- Continue in this format For the rest of the six Aroclors.

Example initial calibration sequence for Congener analysis, created on 02/04 on ECD3:

**1.** Primer
**2.** Hexane
**3.** I3020401STD1 Level 1 (PW020407A @ 1ug/L)
**4.** I3020402STD5 Level 2 (PW020407B @ 5 ug/L)
**5.** I3020403STD10 Level 3 (PW020407C @ 10ug/L)
**6.** I3020404STD20 Level 4 (PW020407D @ 20ug/L)
**7.** I3020405STD50 Level 5 (PW020407E @ 50ug/L)
**8.** I3020406STD100 Level 6 (PW020407F @ 100ug/L)
**9.** I3020407STD150 Level 7 (PW020407G @ 150ug/L)
**10.** I3020408STD200 Level 8 (PW020407H @ 200ug/L)
**11.** HEXANE
**12.** I3020409STD100 ICV (PW120407J @ 100ug/L)

**10.2.3** When the sequence has finished running, the Enviroquant software will generate “Not Reviewed” quantitation reports. All reports must be “Quant Reviewed” before they can become part of the initial calibration acquisition method for sample analysis.

- Enter into the “Environmental Data Analysis” (off-line) screen.
- Go to “File” and under method select the method (A302041660.m) that the initial calibration standards were analyzed under, then select the first data file.
- Go into “Quant” and select “QEdit Quant Results” to process the data files. Refer to SOP\08-03 for details and processing of PCB Aroclor/Congener standards.
- When processing is complete for the first standard, “Save” the changes and “Exit.” Re-print the re-processed data file by “Generating Quant Report,” and save the hard copy for each level of the initial calibration.
- Repeat these steps for all initial calibration standards analyzed within the sequence.
- When all levels have been processed, go into “IntiCal,” and select “Update Levels,” and enter all levels for the initial calibration at the proper concentrations.
- After all responses are entered, “Save” the completed method and print the resulting response factor summary by selecting “Response Factors to Printer.”

**10.2.4** Acceptance Criteria:

- If average calibration factor or average relative response factor used, % RSD ≤ 20% over the working range of the curve for all analytes. If one or more peaks have %RSD greater than 20%, the calibration may still be acceptable if the average %RSD for all analytes in the calibration curve is less than 20%.
- If calibration curve used, the correlation coefficient must be ≥ 0.99 – for linear model or the COD or $r^2$ must be ≥ 0.99 – for non-linear model.

**10.2.5** The following *corrective actions* are recommended for failing initial calibrations:

- Check the calibration of the standard preparation that was performed. If the problem appears to be isolated to a single standard, the standard may be reanalyzed within twelve hours.
- Perform instrument maintenance and repeat the initial calibration.

The choice of corrective action must be made in consultation with the Department Manager, QA Manager, Project Manager, and/or the client.

**10.2.6** Complete the initial calibration by filling out the *Initial Calibration Checklist*. The initial calibration, along with any corresponding continuing calibration data and sample data, is then forwarded for secondary review.

**10.2.7 Initial Calibration Verification - ICV (separate source)**

The analysis of separate source standard must follow the initial calibration curve. All target analytes in this verification standard are at the mid-point concentration. Note: If this standard was analyzed immediately following the initial calibration curve, it must be re-"Calculated and Generated" in the "QEdit" field, against the completed initial calibration curve method and then re-processed, before it can be reviewed for acceptability.

Acceptance Criteria: ICV has to be analyzed from the separate source and % recoveries must be +/-15% for all targets.

## 10.3 Equipment Operation and Sample Processing

Samples are prioritized by analyst for analysis based on hold time and client due date. All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

**10.3.1** Samples are retrieved from the sample storage refrigerator, spiked with 20uL of the chosen internal standard solution per 1mL extract – for congeners only, sulfuric acid cleaned and loaded into the instrument autosampler trays following the generalized sequence below.

**10.3.2** Analyze extracts using the same experimental conditions used for the analysis of the calibration standards. Ensure that calibration verification standards are interspersed, at least, every 10-20 samples or every 12 hour period. Area measurements should all be to baseline unless an unresolved complex mixture (UCM) is observed, in which case, the areas measured should skim the top of the UCM.

**1.** Primer

**2.** Hexane

**3.** AR1660/ Congener CCV – C1060501 @ 500ug/L/50ug/L

**3*.** AR1242/1248/1254 1-point ICAL levels @ 500ug/L (optional)

**4.** 10 samples + QC's

**5.** AR1660/ Congener CCV – C1060501 @ 500ug/L/50ug/L

**6*.** AR1242/1248/1254 CCV @ 500ug/L (optional)

**7.** 10 samples

**8.** AR1660/ Congener CCV – C1060501 @ 500ug/L/50ug/L

**9*.** AR1242/1248/1254 CCV @ 500ug/L (optional)

**10.3.3** After sample analysis, "Not Reviewed" quantitation reports are generated by the software system. Samples are processed from "Not Reviewed" data files, to "Quant Reviewed" data files in a similar way the standards were previously processed. See Section 11 for details on sample processing. If a CCV fails the criteria outlined in Section 10.4.4, all samples since the last acceptable CCV must be re-analyzed (refer to Section 10.4.5).

**10.3.4** If the on-column concentration of any compound exceeds the calibration range of 2000ug/L – for Aroclors and 200ug/L – for Congeners, the sample must be diluted, re-spiked with the appropriate amount of internal standard and re-analyzed. Assuming all samples are at a 1mL final volume, the following example dilutions would apply. Adjust the volumes accordingly for other sample final volume amounts and other desired dilutions.

1:2 dilution = 500uL of sample : 500uL Hexane and 10uL of IS
1:4 dilution = 250uL of sample : 750uL Hexane and 15uL of IS
1:5 dilution = 200uL of sample : 800uL Hexane and 16uL of IS
1:10 dilution = 100uL of sample : 900uL Hexane and 18uL of IS, etc.

## 10.4 Continuing Calibration

The systems performance and calibration for each compound of interest must be checked and verified before sample analysis. A continuing calibration verification (CCV) standard, at the concentration of the mid-level of the initial calibration curve, must be analyzed at the beginning and end of every analytical sequence, and every 12 or at the minimum of every 20 samples whichever is more frequent. However, it is recommended that the CCV is run every ten samples to minimize the number of samples requiring reanalysis.

**10.4.1** Create the sequence: "Edit" the "Sample Table Log" to include the CCV standard (C1060501, where "C" is for CCV, "1" is for instrument number 1, followed by the date, and "01" for the first CCV analyzed this day), and acquire the CCV against the correct initial calibration method. "Save," then "Load and Run" the sequence.

**10.4.2** When the sequence has finished running, the Enviroquant software will generate a "Not Reviewed" quantitation report. All reports must be "Quant Reviewed" against the acquisition method for sample analysis.

- Enter into the "Environmental Data Analysis" (off-line) screen.
- Go to "File" and under method, select the method that the CCV was analyzed under, then select the CCV data file.
- Go into "Quant" and select "QEdit Quant Results" to process the CCV file. please refer to SOP/08-03 for manual integration details and processing of PCB standards
- When processing is complete, go into "ConCal," and select "Evaluate Data File as Continuing Calibration".

- Establish daily retention time windows as the retention time of the component in the opening verification standard ±0.05 min window.

**10.4.3** Acceptance Criteria: Compare the CCV resulting response against the average response for the initial calibration for each calibrated analyte and calculate the % difference (%D). See Section 11 for the calculations. If the %D ≤ ± 15%, sample analysis may take place following this verification standard. If the %D > ± 15% for Aroclors and individual congeners, the CCV may still be acceptable if ALL components (regardless of whether they have been targeted for a specific project) had an average %D of ≤ ± 15%. If a noncompliant analyte exhibits an increase in response in the noncompliant CCV and is not detected in the associated samples, the analyses may be reported and the CCV noncompliance narrated. In this case CCV data or a detailed narrative must be provided to the client. Otherwise, corrective action must take place.

**10.4.4** If the CCV does not meet the criteria for each calibrated analyte, the following *corrective actions* are recommended:

- Check the standard preparation. If the problem appears to be isolated to a single standard reanalyzed the standard
- If the verification is the beginning standard of the sequence, corrective action may consist of prepping the injection port (replace injection liner, clip column, gold seal, and septa) and re-analysis of the standard. If, after re-analyzing the standards, the acceptance criteria are still not met, a new initial calibration must be performed. The system must be in-control with all calibration criteria before sample analysis may proceed.
- If the verification is the ending standard, corrective action depends upon the severity of the verification results. The closing CCV can be re-analyze once to verify the results (e.g. if there are high responses for the CCV that could mean that re-concentration took place and fresh aliquot should be analyzed).
- If the CCV is a closing CCV, the data for the samples run before this CCV may be evaluated for hits. If there are no hits for the compounds that did not meet %D criteria, those samples do not need to be reanalyzed provided that the response of the analyte had increased and would have been detected in the sample if it were present.
- Both columns should meet QC criteria, however, they may be evaluated with the above corrective actions in mind.
- The choice of corrective action must be made in consultation with the Department Manager, QA Manager, Project Manager, and/or the client. The reasoning for choosing one of the above options must be documented in the project narrative to the client.

## 10.5 Preventive Maintenance

**10.5.1** Preventive maintenance may or may not include the following; replacing system uniliner, o-ring, merlin septum, MXTY connector or clipping a length of guard column.

**10.5.2** Additionally, preventive or routine maintenance for GC/ECD systems may involve baking out the ECD detectors, replacing guard, RTX-5 or CLPII columns.

# 11. Data Evaluation, Calculations and Reporting

**11.1** Qualitative identification of multicomponent analytes (Aroclors) requires pattern matching between the calibration standards and the response observed in the sample on both columns. Retention time windows should be used as a gauge; however, pattern recognition for the multicomponent analytes is most important. Qualitative identification for Congeners is made when a peak in a sample is observed within the retention time window for a calibrated analyte on both columns. In other words, an analyte is not considered present in a sample unless the analyte is detected on both columns within the retention time windows for that analyte established during calibration.

**11.2** For samples with PCB Aroclors positively identified on both columns, compare the responses of the 3 to 5 major peaks in the single point calibration standard for that Aroclor with the responses of the peaks observed in the sample extract. For Aroclor1660 use calibration curve to determine concentration of the detected Aroclor. The relative peaks and number of peaks in the sample should be similar to that observed in the standard; however, degradation, weathering and interferences may cause the sample pattern to differ from that observed from the standard. The peaks chosen for quantitation must be free from interferences. Calculate the concentration of each corresponding peak in the sample chromatogram and the 3 to 5 resulting concentrations are averaged to provide the final result for the sample.

**11.3** Calculate the relative percent difference (RPD) between compound concentrations on both columns. If the calculated RPD is >40%, check the chromatograms to see if an overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine baseline parameters established by the data system (or operator) during peak integration.

- *If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is conservative relative to protection of the environment. The disparity between the two columns should be noted by a P qualifier.

- *If the high RPD is clearly the result of an interferent on one of the columns, the lower result may be reported – I flag used.

**11.4** Calculate the recovery for the surrogates TMX and DCB for Aroclors, and DBOB and BZ 198 for Congeners, in each field, blank, and quality control sample extract on both columns. Calculate the recovery of the matrix spike and LCS compounds in the appropriate samples on both columns.

**11.5** Evaluate whether or not a dilution of an extract is needed by ensuring that all target PCBs areas measured fall within the calibration range of the instrument. If the range is exceeded, a dilution analysis is required. Dilute the extract to bring the out-of-range component(s) to within 50-100% of the full calibration range. Also evaluate the sample chromatogram for possible saturation of the detector.

**11.6** If a dilution analysis is made, compare this run to the initial analysis to ensure that the chromatography is similar, that compound detection is similar, and that the reported values for detected compounds make sense (i.e., that the final value for any compound detected in both analyses is about the same value). If the dilution run is not comparable to the initial run, corrective action should take place.

**11.6.1** *Corrective Action:* Re-evaluate both analyses (un-diluted and diluted) ensuring that integration of all peaks was performed properly and that the correct calibration curves were used for quantitation. Determine whether or not the results are off by a systematic bias which might be indicative of a poor injection or of an inaccurate dilution. If the initial run's injection is determined to have

been poor and if this analysis does not need to be reported to the client (based no project specific requirements) then the results of the dilution analysis only should be reported to the client (unless there are special project requirements to report data at specific reporting limits). If an inaccurate dilution is suspected, a new dilution of the extract should be made and this second dilution analyzed. The results from all three runs should be compared to verify the corrective action and to determine which result should be reported to the client.

**11.6.2** Reporting of the results for a sample where two different dilution analyses are conducted, two data sheets should be prepared for reporting the sample with all components reported from the initial run with the "E" flag for the over-range component, which should be reported from the dilution. The project narrative must accurately explain how the data are reported whether one or two data sheets are used to report one sample.

## 11.7 Calculations

**11.7.1** To calculate the ***Relative Standard Deviation*** (RSD) of all target compounds for the initial calibration, use the formula below. See Section 10.2.4 for initial calibration acceptance criteria. Additionally, use the initial multi-point calibration to determine ***Relative Response Factors*** ($RRF_I$s) – *Internal Standard Calibration* or for or ***Calibration Factor (CF$_I$)*** – for *ExternalStandard Calibration Model* at each concentration level, for each analyte. Average the $RRF_I$s from the initial multi-point calibration, to generate mean $RRF_I$s, for quantification of each analyte. Follow the same calculations for each surrogate compound.

The $RRF_I$s are based on the internal standard compounds and are calculated using the formula below.

$$\mathbf{RSD = SD \ / \ mean\ RRF_I \times 100}$$

where:

SD = Standard deviation between the five points, for that target analyte.

$$\mathbf{RRF_I = (A_c \times C_{IS}) / (A_{IS} \times C_c)}$$
$$\mathbf{CF_I = A_a / C_c}$$

where:

$A_c$ = Area of the standard compound to be measured.
$A_{IS}$ = Area of the representative internal standard compound.
$C_{IS}$ = Concentration of the representative internal standard compound (ug/L).
$C_c$ = Concentration of the standard compound to be measured (ug/L).

**11.7.2** Based on the mean $RRF_I$s, calculate the raw concentration for each target analyte and surrogate in the sample extracts using the following formula:

$$C_a = (A_a \times C_{IS}) / (A_{IS} \times RRF_I)$$

where:

$C_a$ = Calculated concentration of the target analyte, from quantitation report (ug/L).
$A_a$ = Area of the target analyte.
$A_{IS}$ = Area of the representative internal standard compound.
$C_{IS}$ = Concentration of the internal standard compound added to each extract (ug/L).

**11.7.3** Calculate the ***Sample Concentration*** (C) for each target compound by the following formula:

$$C = (C_a / V_s) \times FV \times DF$$

where:

C = Concentration in the sample (ug/L liquid, ug/Kg solid, ng/PUF).

$V_s$ = Original volume or weight of sample extracted, corrected for % solids, if applicable. (To correct for % solids, multiply the sample weight by the % solid as expressed as a decimal. For example: 15.24g x 0.843 = 12.85, for a sample size of 15.24g at 84.3% solid).

DF = Dilution factor or fraction of the original extract to which internal standard is added.

FV = Sample Final Volume.

If the response of any analyte in a sample exceeds the linear response range, as defined by the initial calibration standards in Section 13.0, dilute the extract so that the concentrations of all target compounds fall within the range of the calibration curve.

**11.7.4** Calculate the ***Surrogate Recoveries*** relative to the internal standards by the following formula:

$$\%R = \frac{C_S \times DF}{C_{Strue}} \times 100$$

where:

$C_{Strue}$ = Actual concentration of the surrogate in the extract (ug/L).

$C_S$ = Calculated concentration of the surrogate, from quantitation report (ug/L).

DF = Dilution factor or fraction of the original extract.

**11.7.5** Compare results for each analyte in the ***Continuing Calibration Verification*** (CCV), to the true value by determining the percent difference.

**Percent Difference (%D) = ([$C_{Ctrue}$ - $C_C$] / $C_{true}$) x 100**

where:

$C_{Ctrue}$ = Actual concentration of the compound in the CCV (ug/L).
$C_C$ = Calculated concentration of the compound in the CCV, from quantitation report.

**11.8** All solids including soils, sediments, and sludges must be reported on a dry-weight basis. Tissue results may be reported in wet-weight, or "as received," depending upon client request. Air PUF samples are reported on an "as received" basis.

**11.9** The primary analyst does data entry, or upload of the data, into the LIMS system. The LIMS is "linked" to the instrument, so the analyst must choose the sample(s) to be reported from that instrument's analytical sequence. All associated preparation and instrumental QC samples and dilutions are also chosen. Once the data/samples have been selected and "associated" with the proper QC samples, the batched data set is sent to the LIMS.

**11.10** Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP, SOP/08-01. All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

**11.11** Notebooks: Laboratory notebooks are designed to accommodate the specific analysis. Instrument printouts are used to document run sequences, and each daily sequence printout is filed in a three-ring notebook. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry on the sequence log. Requests for re-extraction are further documented in the "Request for Re-extraction" logbook. After one year of sample analysis, the sequence run log is permanently bound, assigned an internal ID number, and filed accordingly. Such files shall be archived so as to remain available for at least 10 years. All laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instructions,WI-108-01, and all record keeping and document control practices.

**11.12** Electronic records: All data files from computers, attached to instruments, shall be backed up daily onto the proper directory on the server. The backups shall be stored so as to be accessible for 10 years. Movement of the data files to the server is the responsibility of the primary analyst. Server backup and storage is the responsibility of the IT department.

# 12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All PCB Aroclor and Congener results are reportable without qualification if analytical holding times are met, preservation requirements (including cooler temperatures) are met, and all QC criteria defined in the table below are met. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis.

| QC Parameter | Acceptance Criteria |
|---|---|
| Initial Calibration Curve | 20% RSD for all target analytes<br>If %RSD>20%, still acceptable if the average %RSD$\leq$ 20%<br>Correlation coefficient must be $\geq$ 0.99 – for linear model<br>The COD or $r^2$ must be $\geq$ 0.99 – for non-linear model |
| Independent Check Verification | +/- 15% recovery of the true values,<br>Sporadic marginal failure accepted for congeners |
| Continuing Calibration Verification | Analyzed every 12hr or at the minimum of every 20 samples, 15% D for all target analytes<br>If the %D > $\pm$ 15%, still acceptable if average %D$\leq$ 15 |
| Method Blank | No analyte at or above the reporting limit,<br>“B” qualify analyte if detected < 5x the blank contamination |
| Laboratory Control Sample | Soil/Tissue/Aqueous/PUF: 40-140%;<br>50% RPD |
| Matrix Spike / Matrix Spike Duplicate | Same as for LCS; 50% RPD between the duplicates. |
| Sample / Sample Duplicate | 50% RPD between the duplicates. |
| Surrogates | Soil: 30-150%; Aqueous: 30-150%; PUF: 60-140% |
| Internal Standards | 50% - 200% of the daily CCV area for the Internal Standards |
| SRM | Same as for LCS, 40% - 140% recovery |

Any deviations and observations made about the analyses must be documented in the instrument logbook and or project narrative. If a problem arises during analysis, document the problem and initiate corrective action. If there is a problem with a sample analysis which indicates that re-extraction should be performed, and if there is no additional sample available for re-extraction, the Project Manager needs to be informed immediately so that the client can be involved in the corrective action process. Re-extraction of a sample should be done within holding time if at all possible.

Evaluation of a sample result often requires knowledge about the results of all samples within a job. Therefore, use all of the data, if possible, from a job to judge whether or not corrective actions are needed. For example, if a sample is run and the surrogates are low, one might request a re-extraction immediately only to find that a MS/MSD were also done on this sample, with low surrogate and MS recoveries, confirming a matrix effect. In this case, a re-extraction is not required and the results would be reported with a narration explaining the evidence of a matrix effect on the low surrogate recoveries. The characteristics of the overall job may be invaluable in deciding how to report the data to the client.

If non-compliant PCB Aroclor or PCB Congener results are to be reported, the Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-

analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

# 13. Method Performance

## 13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

## 13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

### 13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

### 13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

# 14. Pollution Prevention and Waste Management

Once satisfactory analytical results have been generated, the extracts are held for 30 days, or longer if specified by a client contract, then discarded into a 55-gallon drum labeled “Vial Waste”.

Refer to Alpha’s Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

# 15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

G-006 Waste Management and Disposal SOP

G-008 Reagent, Solvent, and Standard Control SOP

SOP/08-01Document Control

SOP/08-03 Manual Integration

# 16. Attachments

Table I: Reporting Limits (RLs) and Method Detection Limits (MDLs) for PCB Aroclors and Congeners by GC/ECD

Table II: LCS/LCSD, MS/MSD and Precision and Accuracy Evaluation Criteria

Table III: Surrogate Recovery Evaluation Criteria

## Table I

## Reporting Limits (RLs) and Method Detection Limits (MDLs) for PCB Aroclors and Congeners by GC/ECD

| Compound | CAS Registry No | Aqueous in µg/L | | Solid in µg/Kg | | PUF in ng/PUF | |
|---|---|---|---|---|---|---|---|
| | | MDLs | RLs | MDL | RLs | MDL | RLs |
| Aroclor 1016 | 12674-11-2 | 0.0050 | 0.03 | 8.01 | 30 | 8.01 | 30 |
| Aroclor 1221 | 11104-28-2 | 0.0103 | 0.03 | 27.6 | 100 | 27.6 | 100 |
| Aroclor 1232 | 11141-16-5 | 0.0092 | 0.03 | 9.98 | 30 | 9.98 | 30 |
| Aroclor 1242 | 53469-21-9 | 0.0055 | 0.03 | 5.63 | 20 | 5.63 | 20 |
| Aroclor 1248 | 12672-29-6 | 0.0051 | 0.03 | 5.62 | 20 | 5.62 | 20 |
| Aroclor 1254 | 11097-69-1 | 0.0105 | 0.03 | 6.72 | 20 | 6.72 | 20 |
| Aroclor 1260 | 11096-82-5 | 0.0045 | 0.03 | 6.22 | 20 | 6.22 | 20 |
| Aroclor 1262 | 11100-14-4 | 0.0092 | 0.03 | 5.51 | 20 | 5.51 | 20 |
| Aroclor 1268 | 37324-23-5 | 0.0032 | 0.03 | 4.89 | 20 | 4.89 | 20 |

| IUPAC Name | BZ # | CAS Registry No. | Aqueous RL in µg/L | Solid RL in µg/Kg |
|---|---|---|---|---|
| 2,4'-Dichlorobiphenyl | BZ 8 | 34883-43-7 | 0.003 | 2.0 |
| 2,2',5-Trichlorobiphenyl | BZ 18 | 37680-65-2 | 0.001 | 1.0 |
| 2,4,4'-Trichlorobiphenyl | BZ 28 | 7012-37-5 | 0.001 | 1.0 |
| 2,2',3,5'-Tetrachlorobiphenyl | BZ 44 | 41464-39-5 | 0.001 | 1.0 |
| 2,2',5,5'-Tetrachlorobiphenyl | BZ 52 | 35693-99-3 | 0.001 | 1.0 |
| 2,3',4,4'-Tetrachlorobiphenyl | BZ 66 | 32598-10-0 | 0.001 | 1.0 |
| 3,3'4,4'-Tetrachlorobiphenyl | BZ 77 | 32598-13-3 | 0.001 | 1.0 |
| 2,2',4,4',5-Pentachlorobiphenyl | BZ 99 | 38380-01-7 | 0.001 | 1.0 |
| 2,2',4,5,5'-pentachlorobiphenyl | BZ 101 | 37680-73-2 | 0.001 | 1.0 |
| 2,3,3',4,4'-pentachlorobiphenyl | BZ 105 | 32598-14-4 | 0.001 | 1.0 |
| 2,3',4,4'5-Pentachlorobiphenyl | BZ 118 | 31508-00-6 | 0.001 | 1.0 |
| 3,3',4,4',5-Pentachlorobiphenyl | BZ 126 | 57465-28-8 | 0.001 | 1.0 |
| 2,2',3,3',4,4'-Hexachlorobiphenyl | BZ 128 | 38380-07-3 | 0.001 | 1.0 |
| 2,2',3,4,4',5'-Hexachlorbiphenyl | BZ 138 | 35065-28-2 | 0.001 | 1.0 |
| 2,2',4,4',5,5'-Hexachlorobiphenyl | BZ 153 | 35065-27-1 | 0.001 | 1.0 |
| 3,3',4,4',5,5'-hexachlorobiphenyl | BZ 169 | 32774-16-6 | 0.001 | 1.0 |
| 2,2',3,3',4,4',5-Heptachlorobiphenyl | BZ 170 | 35065-30-6 | 0.001 | 1.0 |
| 2,2',3,4,4',5,5'-Heptachlorobiphenyl | BZ 180 | 35065-29-3 | 0.001 | 1.0 |
| 2,2',3,4',5,5'6-Heptachlorobiphenyl | BZ 187 | 52663-68-0 | 0.001 | 1.0 |
| 2,2',3,3',4,4',5,6-Octachlorobiphenyl | BZ 195 | 52663-78-2 | 0.001 | 1.0 |
| 2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl | BZ 206 | 40186-72-9 | 0.001 | 1.0 |
| 2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl | BZ 209 | 2051-24-3 | 0.001 | 1.0 |

**Note:** The reporting limits used for "Solid" refer to the matrices of soil, sediment, tissue, and any other possible non-aqueous matrix. The table above represents an example of reporting limits generally used in this analysis. The RLs for this analysis can vary based on sample matrix, amount extracted, final volume and cleanup schemes. Adjustments to these limits can be made to satisfy specific workplan or data quality objective requirements and specialty matrices.

## Table II

## LCS/LCSD, MS/MSD and Precision and Accuracy Evaluation Criteria

| Spiked Component | Aqueous/Solid/Tissue/PUF (% recovery) | RPD |
|---|---|---|
| AR1016 | 40-140 | 50 |
| AR1260 | 40-140 | 50 |
| Congener | 40-140 | 50 |

## Table III

## Surrogate Recovery Evaluation Criteria

| Surrogate | Aqueous/Solid/Tissue (% recovery) | PUF (% recovery) |
|---|---|---|
| 2,4,5,6-tetrachloro-m-ylene(TMX) | 30-150 | 60-120 |
| Decachlorobiphenyl (DCB) | 30-150 | 60-120 |
| DBOB | 30-150 | NA |
| BZ 198 | 30-150 | NA |

**NOTE:** Depending on project specific requirements, MS/LCS/Surrogate recoveries may be validated against a specified project QAPP, 8082 SW-846, TO-10A, CLP OLM04.2, DoD QSM 5.3, or in-house determined limits.