
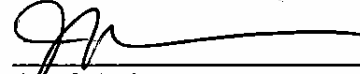




**Title: PCB Analysis by HRGC/HRMS  
[Methods 1668A & 1668C ]**

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## 1. SCOPE AND APPLICATION

- 1.1. This SOP is appropriate for the detection and quantitative measurement of mono- through deca-chlorinated biphenyls in a variety of matrices at low part-per-trillion to part-per-billion levels using high resolution gas chromatography and high resolution mass spectrometry, by Method 1668A or Method 1668C. Sample preparation procedures are defined in SOP WS-IDP-0013 (Method 1668A) and WS-IDP-0018 (Method 1668C)..
- 1.2. The CBs determined in this method are the 12 polychlorinated biphenyls (PCBs) designated as toxic by the World Health Organization (WHO). These are congeners 77, 81,105, 114, 118, 123, 126, 156, 157, 167, 169, and 189. This method also determines the remaining 197 PCBs, of which 128 are resolved on the SPB-octyl column to be determined as individual congeners. The remaining 69 congeners are determined as mixtures of isomers (co-elutions). This method can also be used to determine the 20 PCBs on the National Oceanic and Atmospheric Administration (NOAA) list.
- 1.3. The 12 toxic WHO congeners as well as the first and last eluted congener at each level of chlorination (LOC) are determined by the isotope dilution method using the corresponding labeled analogue. The remaining congeners are determined by using an average of the labeled analogues of each level of chlorination (LOC).
- 1.4. This method allows the determination of PCB toxicity equivalent factors (TEQPCB)
- 1.5. The calibration range of the procedure for 1 L water is 20 to 20000 ppq, 2 to 2000 ppt for 10.0 g soil, sediment or tissue, and 40-40,000 pg/train (assuming 1/2 sample to 20 uL F.V.) for air train samples for mono-deca PCBs. Analysis of dilutions of aliquots of the sample will permit measurements of concentrations above the upper method calibration limit. The practical limits of detection and quantitation may be different from the lower method calibration limit, depending on the complexity of the matrix and the level of PCB contamination of the reagent and absorbent used in the extraction and cleanup procedure.
- 1.6. The PCB naming convention is given in Table VII.
- 1.7. This SOP should be used by analysts who are experienced and skilled in HRGC/HRMS trace analysis.
- 1.8. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

## 2. SUMMARY OF METHOD

- 2.1. This procedure uses matrix specific extraction, analyte specific cleanup and HRGC/HRMS analysis techniques.

- 2.2. An aliquot of a matrix (water, soil, sediment, XAD Resin, filter) is spiked with the solution containing 27 isotopically  $^{13}\text{C}$ -labeled PCBs Isotope Dilution Analytes (IDAs) listed in Table I. The sample is then extracted according to matrix specific extraction procedures in SOP WS-IDP-0013 for Method 1668A or SOP WS-IDP-0018 for Method 1668C.
- 2.3. The preparation of the final extract for the instrumental analysis is accomplished by adding 5 isotopically ( $^{13}\text{C}$ ) labeled internal standards (ISS -Table I). The internal standard  $^{13}\text{C}$ -2,5-DiCB (EPA#9) is used to quantitate the mono and di chlorinated biphenyls and  $^{13}\text{C}$ -TrCB-19. The internal standard  $^{13}\text{C}$ -2,2',5,5'-TCB (EPA #52) is used to determine the percent recoveries of  $^{13}\text{C}$ -TrCB-37- biphenyls and tetra-chlorinated biphenyls. The  $^{13}\text{C}$ -2,2',4,5,5'-PeCB (EPA #101) is used to determine the percent recoveries of penta chlorinated biphenyls. The  $^{13}\text{C}$ -2,2',3,4,4',5'-HxCB (EPA #138) is used to determine the percent recoveries of the hexa chlorinated biphenyls. The  $^{13}\text{C}$ -2,2',3,3',5,5',6,6'-OCB (EPA #194) internal standard is used to determine the percent recoveries of hepta-deca chlorinated biphenyls.
- 2.4. One to two microliters (uL) of the final concentrated extract are injected into the HRGC/HRMS.
- 2.5. The identification of those PCB congeners for which a  $^{13}\text{C}$ -labeled isotope dilution analyte is available in the sample fortification solution (IDA solution) is based on their elution time compared to the corresponding labeled isotope dilution analyte and the simultaneous detection of the two most abundant ions in the molecular ion isotopic cluster.
- 2.6. The identification of those PCB congeners for which no  $^{13}\text{C}$ -labeled isotope dilution analyte is available in the sample fortification solution (IDA solution) is based on their elution time measured in the routine calibration standard and the simultaneous detection of the two most abundant ions in the molecular ion isotopic cluster. Confirmation is based on a comparison of the integrated ion abundance of the molecular ions to their theoretical abundance ratios (Table II).
- 2.7. Quantitation of the individual congeners for which a  $^{13}\text{C}$ -labeled isomer is available in the curve is achieved by using the Relative Response Factor (RRF) determined in the 5 point initial calibration curve (ICAL). The quantitation of specific congeners not included in the five point initial calibration solutions is calculated using a RF determined from a single point calibration of a solution containing all 209 PCB compounds. The quantitation of the total for each homologous series is achieved by using the average RRFs of the first and last eluting isomers in the curve at the corresponding chlorination level, determined by the ICAL using the average response of all  $^{13}\text{C}$ -labeled isotope dilution analytes in that homologous series. The sole exception is  $^{13}\text{C}_{12}$ -3,3',4,4',5-penta PCB (EPA #126) which is not used in the average response for the penta-PCB homologous series.

### 3. DEFINITIONS

- 3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).
- 3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.
- 3.3. Isotope Dilution Analyte (IDA) is used in this SOP to refer to what Method 1668 calls an Internal Standard.
- 3.4. Internal Standard (IS) is used in this SOP to refer to what Method 1668 calls a Recovery Standard.

#### **4. INTERFERENCES**

- 4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.
- 4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.
- 4.3. Reuse of glassware is to be minimized to avoid the risk of contamination.
- 4.4. Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCBs are often associated with other interfering chlorinated substances such as polychlorinated dioxins/furans (PCDDs/PCDFs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, methoxy biphenyl hydroxydiphenyl ethers, benzylphenyl ethers, brominated diphenyl ethers, polynuclear aromatics and pesticides that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.
- 4.5. A high-resolution capillary column (30 m SPB Octyl) is used to resolve as many PCB isomers as possible; however, no single column is known to resolve all isomers.

#### **5. SAFETY**

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-0002), 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. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used.
- 5.1.2. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.1.3. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

#### 5.2. 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 below 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.

<b>Table 1: Materials used in this SOP, and their Hazards.</b>			
<b>Material</b>	<b>Hazards</b>	<b>Exposure Limit (2)</b>	<b>Signs and symptoms of exposure</b>
Dodecane	Flammable	None listed	May cause respiratory tract, skin or eye irritation.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

## 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 10.1.

- 6.1.1. GC Column for mono- through deca- chlorinated biphenyls - 30 m x 0.25 mm i.d.; 0.25um fused silica capillary column (Supelco SPB-Octyl, or equivalent).
- 6.1.2. The SPB-Octyl column must resolve congeners 34 from 23 and congeners 187 from 182. The valley height must be less than 40% of the shorter of the two peaks when the diluted 209 congener solution is analyzed.
- 6.2. Mass Spectrometer - 28-40 eV electron impact ionization, shall be capable of repetitively and selectively monitoring 18 or more exact m/z ratios at high resolution (>10,000) during a period of approximately one second, and shall meet all of the performance specifications in Section 10.2.
- 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 and does not intercept the electron or ion beams.
- 6.4. Data System- Capable of collecting, recording and storing MS data. The system utilizes MassLynx version 4.1 software and Chrom Peak Review, version 2.1 or equivalent.

## 7. REAGENTS AND STANDARDS

- 7.1. Standard solutions - Purchased as solutions or mixtures with certification to their purity, concentration and authenticity, or prepared from materials of known purity and composition. If compound purity is 98 percent or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark in screw-capped vials with Teflon-lined caps in a refrigerator at 4 degrees C. A mark is placed on the vial at the level of the solution so that solvent evaporation loss can be detected. If solvent loss has occurred, the solution should be replaced.
- 7.2. Stock solutions
  - 7.2.1. Preparation - Prepare in isooctane or equivalent solvent per the steps below or purchase as dilute solutions (Cambridge Isotope Laboratories, Cambridge, MA, or equivalent).
  - 7.2.2. Stock standard solutions are prepared from dilutions of neat solutions. Dilutions are performed in volumetric flasks and transferred to a clean 15 mL vial or amber glass bottle with Teflon-lined cap.
- 7.3. Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration of performance test standards. Reference standards that can be used to determine the accuracy of calibration standards are available from Cambridge Isotope Laboratories.

- 7.4. Sealed ampoules may be used until the manufacturer's expiration date is exceeded.
- 7.4.1. If no expiration date is provided, then the expiration date will be 10 years from the date the ampoule is opened.
- 7.4.2. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.
- 7.5. All calibration, daily isotope dilution analyte (IDA) standards, daily clean up recovery surrogate (SU) standards, internal (IS) standards, and daily target analyte (TA) spiking solutions are stable for one year from preparation.
- 7.5.1. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration.
- 7.5.2. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot, second vendor, or SRM.
- 7.6. Secondary standard - Using stock solutions, prepare secondary standard solutions containing the compounds and concentrations shown in Table I in dodecane.
- 7.7. Labeled compound stock standard - From stock standard solutions prepared as above, or from purchased mixtures, prepare this standard to contain the labeled compounds at the concentrations shown in Table I in isooctane.
- 7.8. Isotope Dilution Analyte (IDA) Standard - Prepare at the concentration shown in Table I in isooctane.
- 7.9. Calibration standards (CS-1 through CS-6) - Combine the solutions in Tables I and V to produce the six calibration solutions shown in Table V in dodecane. These solutions permit the relative response (labeled to unlabeled) and response factor to be measured as a function of concentration. The 209 congener standard (CS-4) is used for continuing calibration verification (CCV).
- 7.9.1. A second source standard with all 209 congeners is used for initial calibration verification (ICV).
- 7.10. GC retention time window defining mix (WDM) (209 congener standard CS-4) - Used to define the beginning and ending retention times for mono-deca chlorinated homologue groups.
- 7.11. Standard solutions will be periodically assayed against reference standards. Continued use of standard solutions past the initially indicated expiration date is acceptable if

concentrations are verified versus the reference standard. Upon acceptable verification, a new expiration/evaluation date will be noted in the standards reagent database.

## 8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in the document. The complexity of the method is such that, in order to obtain reliable results, testers should be trained and experienced with sampling and preservation procedures.
- 8.2. There are no demonstrated maximum holding times associated with the PCBs in aqueous, solid, semi-solid, tissues, or other matrices. If stored in the dark at 0-6°C and preserved if required, aqueous samples may be stored for up to one year. Similarly, if stored in the dark at <-10 °C, solids, semi-solid, multi-phase, and tissue samples may be stored for up to one year.
- 8.3. All extracts must be stored capped at room temperature and completely analyzed within 45 days of extraction.

## 9. QUALITY CONTROL

- 9.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of reference matrix processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the acceptance limit or when surrogate recoveries are outside control limits. The associated samples will be evaluated for adverse impact, and flagged or qualified as appropriate. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. For any analyte detected in the blank in any of the homologous series of PCBs, the detection limit in the samples run with that blank for the specific isomer or the total for that homologous series is increased to 5 times of the contamination level. Alternatively, both QC and sample results may be reported and qualified as necessary.

**NOTE: When laboratory contamination is suspected sample results can be assumed to be maximum possible concentrations. It may be useful to consult project action limits to see if these concentrations may be acceptable to the client. This does NOT preclude the immediate need to initiate contamination clean-up procedures in the sample preparation area.**

- 9.1.1. Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any target analytes of interest at a concentration greater than ½ the reporting limit.



- 9.2. A laboratory control sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, sodium sulfate, extraction thimble, filter paper, etc.) spiked with 100 uL of IDA solution (Table 1) and 100 uL of TA solution (Table VI). The LCS must be processed in the same manner and at the same time as the associated samples.
- 9.2.1. **Method 1668A** - Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside 50-150%, or the control limits provided on the LIMS, or by the client. The associated samples will be evaluated for adverse impact, and flagged or qualified as appropriate. Re-extraction of the blank, other batch QC, and the affected samples are required when the LCS is deemed unacceptable
- 9.2.2. **Method 1668C** - Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside the control limits in Table VIII.
- 9.3. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair may be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. MS/MSD are prepared and analyzed at the request of the client. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analyte recoveries and precision must be within control limits 50-150%, or the control limits provided on the LIMS, or by the client. The result obtained from MS and MSD samples analysis should agree within 50 percent relative difference. Corrective actions must be documented on a Non-Conformance memo. Outliers will be flagged and narrated as appropriate.
- 9.4. The IDA recovery for each sample and method blank should be between 25 and 150 percent (15 and 150 percent for  $^{13}\text{C}$ -PCB-1 and  $^{13}\text{C}$ -PCB-3) for Method 1668A and within the recovery limits outlined in Table VIII for Method 1668C. Signal-to-noise of IDA's is also evaluated to assess data usability. Signal-to-noise should exceed 10:1 for all IDA's.
- 9.5. Positive results for the 12 coplanar PCBs are reported at the lower calibration limit of 2.0 pg/g for solids assuming a 10.0 g aliquot, 20 pg/L for aqueous assuming a 1.0 liter aliquot, and 40 pg/sample for air train samples assuming a one half split of the sample. For all remaining congeners the reporting limit is ten times the lower calibration limit, unless otherwise specified. Detection limits are reported on a sample specific basis and all results are recovery corrected per the isotope dilution technique. For an analyte reported as 'Not Detected' the associated reporting limit represents its maximum possible concentration.

## 10. CALIBRATION

- 10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".
- 10.2. Chromatographic/Mass Spectrometric Conditions and Data Acquisition Parameters
  - 10.2.1. Gas Chromatograph
    - Column coating SPB-Octyl
    - Film thickness: 0.25 um
    - Column dimension: 30 m X 0.25mm
    - Injector temperature: 260°C
    - Splitless valve time: 1.0
    - Interface temperature: 270°C
    - Recommended Temperature program: (see Table III)
  - 10.2.2. The mass spectrometer is operated in selective ion monitoring (SIM) mode. The ions listed in Table IV for each of the six SIM descriptors are monitored. Some of the ions are monitored in more than one SIM descriptor due to overlapping of the GC elution windows of the different homologous series.
  - 10.2.3. The mass spectrometer is tuned by using a PFK molecular bleed. The instrument is tuned to a resolving power of 10,000 (10 percent valley) at m/z 293.9165, or other appropriate PFK mass, at 8 kV. The intensity of this peak is qualitatively checked at 6 and 8 kV.  
  
**NOTE: Commercially available PFK can contain varying levels of contamination. A minor PFK mass (223.9872) is known to interfere with the dichloro PCB secondary quantitation ion (M+2). If this interferent is present it may not be possible to meet the S/N and ion ratio criteria for CS-2 In these cases the calibration/verification is considered acceptable if the CS-3 through CS-6 levels meet all criteria and the CS-2 meets RT criteria and the primary quantitation ion meets 10:1 signal to noise.**
  - 10.2.4. A hardcopy of the PFK reference peaks covering the mass range of 6 descriptors is produced, at the start of a run sequence, to document the mass resolution at that range.
  - 10.2.5. A mass resolution is also taken at the completion of a 12 hour period. The mass resolution must have a resolving power of at least 5,000. If the end mass resolution cannot be taken the data will be evaluated for usability and flagged or narrated appropriately. Also a Non-Conformance memo will also be completed.
- 10.3. Window Defining Mixture (WDM)

A solution containing all 209 PCBs is injected to determine the elution window for each homologous series. Two ions for each homologous series are monitored in each corresponding SIM descriptor.

NOTE: Injection of the WDM/CCV begins the 12 hour analysis bracket. All samples must be injected within 12 hours.

10.3.1. The WDM/CCV is also used to verify that acquisition windows for each function group are correct and to monitor and verify column performance criteria is achieved.

#### 10.4. Initial Calibration

10.4.1. Calibration is required prior to starting sample analysis. Initial calibration is also required if the routine (daily) calibration fails to meet the criteria for acceptable routine calibration.

10.4.2. A minimum of five high-resolution concentration solutions (listed in Table V) are used for initial calibration. (CS-1 optional)

10.4.3. The relative response factors (RRF) for native target analytes (TA) [RRF(n)] relative to their appropriate IDAs and the relative response factor of <sup>13</sup>C- IDA [RRF(m)] relative to the internal standards (IS) are calculated according to the following formulae:

$$\text{Equation 1} \quad RRF(n) = \frac{A_{TA} \times Q_{IDA}}{Q_{TA} \times A_{IDA}}$$

$$\text{Equation 2} \quad RRF(m) = \frac{A_{IS} \times Q_{rs}}{Q_{is} \times A_{rs}}$$

Where:

$A_{TA}$  = Sum of the integrated ion areas of the quantitation ions for unlabeled PCBs,

$A_{IDA}$  = Sum of the integrated ion areas of the quantitation ions for the labeled isotope dilution analytes.

$A_{IS}$  = Sum of the integrated ion areas of the quantitation ions for the labeled internal standards,

$Q_{IDA}$  = Quantity of the isotope dilution analyte injected (pg),

$Q_{IS}$  = Quantity of the internal standard injected (pg), and

$Q_{TA}$  = Quantity of the unlabeled PCB target injected (pg)

The RRF (n) and RRF (m) are dimensionless quantities; the units used to express  $Q_{IDA}$ ,  $Q_{IS}$ , and  $Q_{TA}$  must be the same.

The RRF for the total PCBs for each homologous series is calculated as the average of RRFs of the first and last eluting isomers of the corresponding

homologous series. The sole exception is  $^{13}\text{C}_{12}$ -3,3',4,4',5-penta PCB (EPA #126) which is not used in the average response for the penta-PCB homologous series.

- 10.4.4. The initial calibration is accepted if the relative standard deviation (% RSD) for the Toxic and LOC native compounds listed in Method 1668A does not exceed  $\pm 20$  percent. The second source criterion for the natives (TA) is  $\pm 30\%$  deviation from the curve. The first eluting congeners for each chlorination level are not Toxic congeners, and are only used for retention time reference and in the estimated calculation of homolog concentrations. The calibration for the labeled isotope dilution analytes (IDAs) is acceptable if the %RSD does not exceed  $\pm 40$  percent.

*Note- The incorporation of alternate calibration analytes and acceptance criteria may be implemented on a project specific basis.*

- 10.4.5. The signal to noise ratio (s/n) for the GC signals present in every selected ion current profile must be  $> 2.5:1$ .
- 10.4.6. An injection of the 209 PCB congener mix will be performed annually at concentration at or near the laboratory reporting limit for the congeners not included in the multi-point calibration curve. Solution must be 1 – 2 times the reporting limit. For DoD compliant work a quarterly LOD check at the CS-2 level and an LOQ check at 1-4X the DL level must be performed.

## 10.5. Daily Calibration Check (CS4)

### 10.5.1. 1668A Calibration Criteria

10.5.1.1. Daily calibration check is required every 12 hours. The daily calibration check is acceptable if the % Difference in RRF for the Toxic and LOC native compounds listed in Method 1668A are not greater than  $\pm 30\%$  from the initial calibration. The first eluting congeners for each chlorination level are not Toxic congeners, and are only used for retention time reference and in the estimated calculation of homolog concentrations. The daily calibration check of IDAs is acceptable if the RRFs are not greater than  $\pm 50\%$  of the mean RRF calculated from the initial calibration curve, unless otherwise directed in the client's SOW. The daily calibration check of the cleanup recovery surrogates (SU) is acceptable if the RRFs are not greater than  $-40\%$  or  $+30\%$  of the mean RRF calculated from the initial calibration curve, unless otherwise directed in the client's SOW. The ratio of the ions for the target analytes (TA) and isotope dilution analytes (IDAs) must be within the limits specified in Table II. If the daily calibration check fails to meet the above criteria, a new initial calibration curve is required.

10.5.1.2. For 1668C QC acceptance criteria see Table VIII.

10.5.2. The valley height between the shorter of the two peaks for the congener pairs 34/23 and 187/182 must be less than 40%. Congeners 156 and 157 must co-elute within 2 seconds of the peak maximum.-

10.5.3. If the criteria in Section 10.5.2 are not met, maintenance must be performed. If a second injection fails the column should be replaced.

10.5.3.1. The retention times and response factors should be updated after the criteria for Section 10.5.2 is met.

## 11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Non-Conformance Memo shall be filed in the project file.

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

11.2. Analysis

11.2.1. 1-2 uL of the final 20 uL extract of the sample is injected into the GC.

11.2.2. Acquire SIM data under the same acquisition and mass spectrometer conditions that the initial calibration curve was acquired.

## 12. CALCULATIONS/DATA REDUCTION

12.1. For a gas chromatographic peak to be identified as a PCB peak, the following criteria must be met:

12.1.1. For the PCB congeners (a total of 27 congeners) which have an isotopically labeled IDAs in the sample extract, the retention time must be within -1 to +3 seconds of the related IDA.

12.1.2. For the Toxic/LOC congeners (27 congeners) that are present in the five initial calibration solutions, the retention time must be within 0.0005 retention time units of the relative limits measured in the routine calibration (CS4 standard).

12.1.3. For the remaining 182 PCB congeners (mono-nona) which do not have an isotopically labeled internal standard and are not in the five point initial calibration

solutions, the retention time should be within 0.005 retention time units of the relative times measured in the 209 PCB single point calibrations solution. If mono-nona totals are being reported for all or just the remaining isomers the retention time for these compounds should be within the corresponding homologous retention time windows which are established by analyzing either a calibration solution or the 209 single point calibration solution..

- 12.1.4. For the two ions monitored for each analyte, the apex of the peaks must occur within +/- 2 seconds of each other.
- 12.1.5. The ratio of the relative intensity of the selected isotopic ions is required to be within the limits (Table II).
- 12.1.6. A GC/MS peak must be 2.5 times higher than the noise level for positive identification of a PCB compound, and 10 times higher than the noise level for all labeled compounds.
- 12.1.7. For total isomers to be positively identified they must be within the retention time window of their respective homologous series as specified by the 209 PCB calibration standard.
- 12.1.8. The loss of one or more chlorines from high chlorinated congeners may contribute to the less-chlorinated congeners peaks that elute at the same retention time which can have an adverse effect on the quantitation of the less-chlorinated congeners. Also in the analysis of total PCBs the extra erroneous peaks in the chromatogram of less-chlorinated congeners produced by the fragment of the high-chlorinated congeners may lead to a high bias in the concentration of the less-chlorinated congeners. If identification is ambiguous, an experienced analyst will determine the presence or absence of the congeners to be reported and the data flagged or narrated appropriately. (The flags will include but are not limited to an elevated detection limit or an estimated positive concentration).
- 12.2. For gas chromatographic peaks that have met the criteria in Section 12.1, the concentration of the PCBs is calculated by using the following formula:

**Equation 3**

$$C_{TA} = \frac{A_{TA} \times Q_{IDA}}{A_{IDA} \times W \times RRF_{(TA)}}$$

Where:

- $C_{TA}$  = Concentration of unlabeled TA PCB congener,  
 $A_{TA}$  = Sum of the integrated ion areas of quantitation ions for unlabeled PCBs,  
 $A_{IDA}$  = Sum of the integrated ion areas of the quantitation ions for the labeled IDAs,  
 $Q_{IDA}$  = Quantity, in pg, of the IDAs added to the sample before extraction,  
 $W$  = Sample size in g (if solid) or L (if liquid), and

$RR_{(TA)}$  = Calculated mean relative response factor for the TA.

12.3. The percent recovery of the IDAs is calculated by using the following formula:

**Equation 4**                      IDA Percent Recovery =  $\frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS} \times RRF_{(IDA)}} \times 100$

Where:

$A_{IDA}$  = Sum of the integrated ion areas of the quantitation ions for the labeled isotope dilution analyte (IDA).

$A_{IS}$  = Sum of the integrated ion areas of the quantitation ions for the labeled internal standard (IS).

$Q_{IDA}$  = Quantity, in pg, of the isotope dilution analyte (IDA) added to the sample before extraction,

$Q_{IS}$  = Quantity, in pg, of the internal standard (IS) added to the cleaned up sample extract before HRGC/HRMS analysis, and

$RRF_{(IDA)}$  = Mean relative response factor for the labeled IDA relative to the appropriate internal standard

12.4. The total concentration for each homologous series of PCBs calculated by summing up the concentration of all positively identified isomers of each homologous series.

12.5. Target compounds that exceed the upper calibration range of the calibration solutions, will be qualified as estimated unless otherwise directed by specific project request. A dilution factor appropriate to bringing the toxic congeners within the calibration range should be used. For other congeners a dilution that, at a minimum, brings the response to a concentration within the detector's response range is acceptable. If possible, the dilution should bring the concentration within the calibration range of the initial calibration solutions. Results for PCB congeners in a sample that has been diluted are reported at the least dilute level at which the area at the quantitation m/z is in the linear response range and the corresponding labeled compound recovery is within the acceptance range.

12.6. Reporting Results

12.6.1. Unless otherwise directed TestAmerica Sacramento will report results in the following units: aqueous samples (pg/L), solids and sediments (pg/g), air samples (pg/sample). Tissues are reported in pg/g wet weight.

12.6.2. Unless otherwise requested the Toxic/LOC congeners will be reported to an RL consistent with the CS-2 calibration level. Other congeners will be reported to an RL of 10X the CS-2 level. Reporting limits for coeluting groups will be multiplied by the number of congeners present. EPA CLP work will be reported per Exhibit C Table 1 of the statement of Work (CBC1-x). Results below the reporting limit will be reported only upon specific request and the reporting process must be agreed upon with the client before samples are processed.

### 13. METHOD PERFORMANCE

#### 13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-0006. MDLs are available in the Quality Assurance department.

#### 13.2. Initial Demonstration

Each analyst must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration standard.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the historical acceptance criteria.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4. A passing PT sample can be substituted for the 4 aliquots in Section 13.2.1.

#### 13.3. Training Qualification

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.

### 14. POLLUTION CONTROL

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."

14.1. 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.



- 14.2. Do not allow waste solvent to evaporate in fume hoods. All solvent waste is stored in capped containers unless transfers are being made.

## 15. WASTE MANAGEMENT

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 SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

- 15.1. Autovials contaminated with dodecane. As the autovials are removed from the instrument after analysis, they are stored in vial files in the instrument lab for at least ninety days, depending on client requirements. After at least ninety days, the vial files are transferred to the waste disposal area where they are drummed and shipped as PCB waste after no more than ninety days.
- 15.2. Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials that may or may not be contaminated/hazardous. Place contaminated materials into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

## 16. REFERENCES/CROSS REFERENCES

- 16.1. State of California Air Resources Board Method 428: Determination of Polychlorinated Dibenzo-p-dioxin (PCDD), Polychlorinated Dibenzofuran (PCDF), and Polychlorinated Biphenyl Emissions from Stationary Sources, September 12, 1990.
- 16.2. EPA Method 1668: Toxic polychlorinated Biphenyls by Isotope Dilution High Resolution Gas Chromatography/High resolution Mass Spectrometry, March 1997.
- 16.3. Method 1668, Revision A (Method 1668A): Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, August 2003.
- 16.4. :Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, April 2010.

## 17. METHOD MODIFICATIONS

- 17.1. Deviations from reference Method 1668A
  - 17.1.1. The acceptance criteria of not greater than +/- 20% and +/- 30% for the Initial Calibration and Continuing Calibration Check respectively, includes the Toxic and

LOC native compounds listed in Method 1668A and 1668C,

- 17.1.2. The retention time for PCB-209 does not have to be greater than 55 minutes if all criteria listed in Section 10.5 is achieved. The laboratory uses GC conditions different than those recommended in Method 1668A.
- 17.1.3. The retention time windows used to identify the chlorination levels is established from the analysis of the native window defining mix included in the calibration curve and native spiking mix.
- 17.1.4. The laboratory routinely uses a SPB-Octyl column for primary analysis.

## **18. ATTACHMENTS**

- 18.1. Table I- Composition of the Sample Fortification Solutions
- 18.2. Table II- Ion-Abundance Ratio Acceptable Ranges
- 18.3. Table III- GC Temperature Program
- 18.4. Table IV- Ions Monitored for HRGC/HRMS Analysis of PCBs
- 18.5. Table V- High-Resolution Concentration Calibration Solution
- 18.6. Table VI- Composition of the Matrix Spike Fortification Solution
- 18.7. Table VII- Analyte List
- 18.8. Table VIII – Acceptance Criteria for VER, IPR, OPR, and Labeled Compounds in Samples

## **19. REVISION HISTORY**

- 19.1. WS-ID-0013, Revision 4.3, Effective 05/10/2013
  - 19.1.1. Incorporated SOP (WS-ID-0018 – Method 1668C) into this SOP.
  - 19.1.2. Added Table VIII with Method 1668C acceptance criteria.
  - 19.1.3. Editorial changes.
- 19.2. WS-ID-0013, Revision 4.2, Effective 03/19/2013
  - 19.2.1. Updated selected ion masses in Table IV
  - 19.2.2. Editorial changes..

- 19.3. WS-ID-0013, Revision 4.1, Effective 11/11/2011
  - 19.3.1. Updated Table(s) 2, 3, and 4.
  - 19.3.2. Editorial changes.
- 19.4. WS-ID-0013, Revision 4, Effective 8/31/2010
  - 19.4.1. Deleted the last sentence in Section 2.6.
  - 19.4.2. Deleted all references to the DB-5 column type in Sections 4.5, 6.2.1 and 10.2.1.
  - 19.4.3. Deleted all references to extraction equipment or requirements.
  - 19.4.4. Deleted Section 6.2.3.
  - 19.4.5. Deleted Reference 16.1.
  - 19.4.6. Editorial changes.
  - 19.4.7. Tables I, II, III, V and VI were updated.

19.5.

20.

**TABLE I**  
**Composition of the Sample Fortification Solutions**

	Isotope Dilution Analytes (IDA's) Concentration (pg/uL in Isooctane)	Internal Standards (IS's) Solution (pg/uL in Dodecane)	Clean-up Recovery Surrogates (SU) (pg/uL in Dodecane)
<sup>13</sup> C <sub>12</sub> -2-MonoPCB (1)	20	--	--
<sup>13</sup> C <sub>12</sub> -4-MonoPCB (3)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,2-DiPCB (4)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,5-DiPCB (9)	--	100	--
<sup>13</sup> C <sub>12</sub> -4,4'-DiPCB (15)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,2',6'-TriPCB (19)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,4,4'-TriPCB (28)	--	--	100
<sup>13</sup> C <sub>12</sub> -3,4,4'-TriPCB (37)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,2',5,5'-TetraPCB (52)	--	100	--
<sup>13</sup> C <sub>12</sub> -2,2',6,6'-TetraPCB (54)	20	--	--
<sup>13</sup> C <sub>12</sub> -3,3',4,4'-TetraPCB (77)	20	--	--
<sup>13</sup> C <sub>12</sub> -3,4,4',5-TetraPCB (81)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,2',4,5,5'-PentaPCB (101)	--	100	--
<sup>13</sup> C <sub>12</sub> -2,2',4,6,6'-PentaPCB (104)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4'-PentaPCB (105)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,3,3',5,5'-PentaPCB (111)	--	--	100
<sup>13</sup> C <sub>12</sub> -2,3,4,4',5-PentaPCB (114)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,3',4,4',5-PentaPCB (118)	20	--	--
<sup>13</sup> C <sub>12</sub> -2',3,4,4',5-PentaPCB (123)	20	--	--
<sup>13</sup> C <sub>12</sub> -3,3',4,4',5-PentaPCB (126)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,2',3',4,4',5'-HexaPCB (138)	--	100	--
<sup>13</sup> C <sub>12</sub> -2,2',4,4',6,6'-HexaPCB (155)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4',5-HexaPCB (156)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4',5'-HexaPCB (157)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,3',4,4',5,5'-HexaPCB (167)	20	--	--
<sup>13</sup> C <sub>12</sub> -3,3',4,4',5,5'-HexaPCB (169)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,2',3,3',5,5',6-HeptaPCB (178)	--	--	100
<sup>13</sup> C <sub>12</sub> -2,2',3,4',5,6,6'-HeptaPCB (188)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4',5,5'-HeptaPCB (189)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,2',3,3',4,4',5,5'-OctaPCB (194)	--	100	--
<sup>13</sup> C <sub>12</sub> -2,2',3,3',5,5',6,6'-OctaPCB (202)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4',5,5',6-OctaPCB (205)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,2',3,3',4,4',5,5',6-NonaPCB (206)	20	--	--
<sup>13</sup> C <sub>12</sub> -2,2',3,3',4,5,5',6,6'-NonaPCB (208)	20	--	--
<sup>13</sup> C <sub>12</sub> -DecaPCB (209)	20	--	--

**TABLE II**  
**Ion-Abundance Ratio Acceptable Ranges**

Number of Halogen Atoms	Theoretical Ion Type	Ratio	Control Limits	
			Lower	Upper
1 Cl	M/M+2	3.13	2.66	3.60
2 Cl	M/M+2	1.56	1.33	1.79
3 Cl	M/M+2	1.04	0.88	1.20
4 Cl	M/M+2	0.77	0.65	0.89
5 Cl	M/M+2	1.55	1.32	1.78
6 Cl	M+2/M+4	1.24	1.05	1.43
7 Cl	M+2/M+4	1.05	0.89	1.21
8 Cl	M+2/M+4	0.89	0.76	1.02
9 Cl	M+2/M+4	0.77	0.65	0.89
10 Cl	M+4/M+6	1.16	0.99	1.33

**TABLE III**  
**GC Temperature Program**

Parameter	SPB-Octyl
Run time (m)	71.6
Initial Temp (°C)	150
Initial Time (m)	2.0
Rate #1 (°C/m)	2.0
Temp #1 (°C)	256
Time #1 (m)	0.0
Rate #2 (°C/m)	15
Temp #2 (°C)	275
Time #2 (m)	12
Rate #3 (°C)	120
Final Temp (°C)	120
Final Time (m)	2

**TABLE IV**  
**Ions Monitored for HRGC/HRMS Analysis of PCBs**

Descriptor	Accurate Mass	Ion I.D.	Analyte
1	188.0393	M	MonoCB
	190.0363	M+2	MonoCB
	200.0795	M	MonoCB IDA
	202.0766	M+2	MonoCB IDA
	218.9856	Lock	PFK
	222.0003	M	DiCB
	223.9974	M+2	DiCB
	225.9944	M + 4	DiCB
	234.0406	M	DiCB IDA
	236.0376	M+2	DiCB IDA
2	222.0003	M	DiCB
	223.9974	M+2	DiCB
	225.9944	M + 4	DiCB
	234.0406	M	DiCB IDA
	236.0376	M+2	DiCB IDA
	242.9856	Lock	PFK
	255.9613	M	TriCB
	257.9584	M+2	TriCB
	259.9554	M + 4	TriCB
	268.0016	M	TriCB IDA
	269.9986	M+2	TriCB IDA
	289.9224	M	TetraCB
	291.9194	M+2	TetraCB
	293.9165	M + 4	TetraCB
	301.9626	M	TetraCB IDA, IS
	303.9597	M+2	TetraCB IDA, IS
3	255.9613	M	TriCB
	257.9584	M+2	TriCB
	259.9554	M + 4	TriCB
	268.0016	M	TriCB IDA
	269.9986	M+2	TriCB IDA
	280.9825	Lock	PFK
	289.9224	M	TetraCB
	291.9194	M+2	TetraCB
	293.9165	M + 4	TetraCB
	301.9626	M	TetraCB IDA, IS
	303.9597	M+2	TetraCB IDA
	323.8834	M	PentaCB
	325.8804	M+2	PentaCB
	327.8775	M + 4	PentaCB
	337.9207	M+2	PentaCB IDA, IS
	339.9178	M + 4	PentaCB IDA, IS

Descriptor	Accurate Mass	Ion I.D.	Analyte
4	289.9224	M	TetraCB
	291.9194	M+2	TetraCB
	293.9165	M + 4	TetraCB
	301.9626	M	TetraCB IDA, IS
	303.9597	M+2	TetraCB IDA
	323.8834	M	PentaCB
	325.8804	M+2	PentaCB
	327.8775	M + 4	PentaCB
	330.9792	Lock	PFK
	337.9207	M+2	PentaCB IDA, IS
	339.9178	M + 4	PentaCB IDA, IS
	359.8415	M+2	HexaCB
	361.8385	M+4	HexaCB
	363.8356	M + 6	HexaCB
	371.8817	M+2	HexaCB IDA
	373.8788	M+4	HexaCB IDA
	393.8025	M+2	HeptaCB
	395.7995	M+4	HeptaCB
	397.7966	M + 6	HeptaCB
	405.8428	M+2	HeptaCB IDA
407.8398	M+4	HeptaCB IDA	
5	354.9792	Lock	PFK
	359.8415	M+2	HexaCB
	361.8385	M+4	HexaCB
	363.8356	M + 6	HexaCB
	371.8817	M+2	HexaCB IDA
	373.8788	M+4	HexaCB IDA
	393.8025	M+2	HeptaCB
	395.7995	M+4	HeptaCB
	397.7966	M + 6	HeptaCB
	405.8428	M+2	HeptaCB IDA
	407.8398	M+4	HeptaCB IDA
	427.7635	M+2	Octa CB
	429.7606	M+4	Octa CB
	431.7576	M + 6	Octa CB
	439.8038	M+2	OctaCB IDA
	441.8008	M+4	OctaCB IDA
454.9782	Lock	PFK	
6	393.8025	M+2	HeptaCB
	395.7995	M+4	HeptaCB
	397.7966	M + 6	HeptaCB
	405.8428	M+2	HeptaCB IDA
	407.8398	M+4	HeptaCB IDA
	427.7635	M+2	Octa CB

Descriptor	Accurate Mass	Ion I.D.	Analyte
	429.7606	M+4	Octa CB
	431.7576	M + 6	Octa CB
	439.8038	M+2	OctaCB IDA
	441.8008	M+4	OctaCB IDA
	442.9728	QC	PFK
	454.9782	Lock	PFK
	461.7246	M + 2	NonaCB
	463.7216	M + 4	NonaCB
	465.7187	M + 6	NonaCB
	473.7648	M + 2	NonaCB IDA
	475.7619	M + 4	NonaCB IDA
	495.6856	M + 2	DecaCB
	497.6826	M + 4	DecaCB
	499.6797	M + 6	DecaCB
	507.7258	M + 2	DecaCB IDA
	509.7229	M + 4	DecaCB IDA
	511.7199	M + 6	DecaCB IDA



**TABLE V**  
**High-Resolution Concentration Calibration Solution**

Compound	IUPAC #(s)	Concentration (pg/ $\mu$ L in dodecane)					
		Optional CS1	CS2	CS3	CC4	CS5	CS6
Target Analytes (TA's)							
MonoPCB	1,3	0.2	1	5	50	400	1000
DiPCB	4,15	0.2	1	5	50	400	1000
TriPCB	19,37	0.2	1	5	50	400	1000
TetraPCB	54,77,81	0.2	1	5	50	400	1000
PentaPCB	104,105,114,118,123,126	0.2	1	5	50	400	1000
HexaPCB	155, 156,157,167,169	0.2	1	5	50	400	1000
HeptaPCB	188,189	0.2	1	5	50	400	1000
OctaPCB	202,205	0.2	1	5	50	400	1000
NonaPCB	206,208	0.2	1	5	50	400	1000
DecaPCB	209	0.2	1	5	50	400	1000
Isotope Dilution Analytes (IDA's)							
<sup>13</sup> C-MonoPCB	1,3	100	100	100	100	100	100
<sup>13</sup> C-DiPCB	4,15	100	100	100	100	100	100
<sup>13</sup> C-TriPCB	19,37	100	100	100	100	100	100
<sup>13</sup> C-TetraPCB	54,77, 81	100	100	100	100	100	100
<sup>13</sup> C-PentaPCB	104,105,114,118,123,126	100	100	100	100	100	100
<sup>13</sup> C-HexaPCB	155,156,157,167,169	100	100	100	100	100	100
<sup>13</sup> C-HeptaPCB	188,189	100	100	100	100	100	100
<sup>13</sup> C-OctaPCB	202,205	100	100	100	100	100	100
<sup>13</sup> C-NonaPCB	206,208	100	100	100	100	100	100
<sup>13</sup> C-DecaPCB	209	100	100	100	100	100	100
Internal Standards (IS's)							
<sup>13</sup> C-DiPCB	9	100	100	100	100	100	100
<sup>13</sup> C-TetraPCB	52	100	100	100	100	100	100
<sup>13</sup> C-PentaPCB	101	100	100	100	100	100	100
<sup>13</sup> C-HexaPCB	138	100	100	100	100	100	100
<sup>13</sup> C-OctaPCB	194	100	100	100	100	100	100
Cleanup Recovery Surrogates (SU's)							
<sup>13</sup> C-TriPCB	28	100	100	100	100	100	100
<sup>13</sup> C-PentaPCB	111	100	100	100	100	100	100
<sup>13</sup> C-HeptaPCB	178	100	100	100	100	100	100

**Table VI**  
**Composition of the Matrix Spike Fortification Solution**

Compound (Unlabelled)	IUPAC #(s)	Concentration (pg/uL in Dodecane)
MonoPCB	1,3	20
DiPCB	4,15	20
TriPCB	19,37	20
TetraPCB	54,77,81	20
PentaPCB	104,105,114, 118,123,126	20
HexaPCB	155, 156,157,167,169	20
HeptaPCB	188,189	20
OctaPCB	202,205	20
NonaPCB	206,208	20
DecaPCB	209	20

**Table VII  
Analyte List**

SYNAME	Compound	CAS No	SYNAME	Compound	CAS No
PCB 1 (BZ)	2-Chlorobiphenyl	2051-60-7	PCB 106 (BZ)	2,3,3',4,5-Pentachlorobiphenyl	70424-69-0
PCB 2 (BZ)	3-Chlorobiphenyl	2051-61-8	PCB 107 (BZ)		
PCB 3 (BZ)	Biphenyl, 4-chloro	2051-62-9	109 (IUPAC)	2,3,3',4',5-Pentachlorobiphenyl	70424-68-9
PCB 4 (BZ)	2,2'-Dichlorobiphenyl	13029-08-8	PCB 108 (BZ)		
PCB 5 (BZ)	2,3-Dichlorobiphenyl	16605-91-7	107 (IUPAC)	2,3,3',4,5'-Pentachlorobiphenyl	70362-41-3
PCB 6 (BZ)	2,3'-Dichlorobiphenyl	25569-80-6	PCB 109 (BZ)		
PCB 7 (BZ)	2,4-Dichlorobiphenyl	33284-50-3	108 (IUPAC)	2,3,3',4,6-Pentachlorobiphenyl	74472-35-8
PCB 8 (BZ)	2,4'-Dichlorobiphenyl	34883-43-7	PCB 110 (BZ)	2,3,3',4',6-Pentachlorobiphenyl	38380-03-9
PCB 9 (BZ)	2,5-Dichlorobiphenyl	34883-39-1	PCB 111 (BZ)	2,3,3',5,5'-Pentachlorobiphenyl	39635-32-0
PCB 10 (BZ)	2,6-Dichlorobiphenyl	33146-45-1	PCB 112 (BZ)	2,3,3',5,6-Pentachlorobiphenyl	74472-36-9
PCB 11 (BZ)	3,3'-Dichlorobiphenyl	2050-67-1	PCB 113 (BZ)	2,3,3',5',6-Pentachlorobiphenyl	68194-10-5
PCB 12 (BZ)	3,4-Dichlorobiphenyl	2974-92-7	PCB 114 (BZ)	2,3,4,4',5-Pentachlorobiphenyl	74472-37-0
PCB 13 (BZ)	3,4'-Dichlorobiphenyl	2974-90-5	PCB 115 (BZ)	2,3,4,4',6-Pentachlorobiphenyl	74472-38-1
PCB 14 (BZ)	3,5-Dichlorobiphenyl	34883-41-5	PCB 116 (BZ)	2,3,4,5,6-Pentachlorobiphenyl	18259-05-7
PCB 15 (BZ)	4,4'-Dichlorobiphenyl	2050-68-2	PCB 117 (BZ)	2,3,4',5,6-Pentachlorobiphenyl	68194-11-6
PCB 16 (BZ)	2,2',3-Trichlorobiphenyl	38444-78-9	PCB 118 (BZ)	2,3',4,4',5-Pentachlorobiphenyl	31508-00-6
PCB 17 (BZ)	2,2',4-Trichlorobiphenyl	37680-66-3	PCB 119 (BZ)	2,3',4,4',6-Pentachlorobiphenyl	56558-17-9
PCB 18 (BZ)	2,2',5-Trichlorobiphenyl	37680-65-2	PCB 120 (BZ)	2,3',4,5,5'-Pentachlorobiphenyl	68194-12-7
PCB 19 (BZ)	2,2',6-Trichlorobiphenyl	38444-73-4	PCB 121 (BZ)	2,3',4,5',6-Pentachlorobiphenyl	56558-18-0
PCB 20 (BZ)	2,3,3'-Trichlorobiphenyl	38444-84-7	PCB 122 (BZ)	2',3,3',4,5-Pentachlorobiphenyl	76842-07-4
PCB 21 (BZ)	2,3,4-Trichlorobiphenyl	55702-46-0	PCB 123 (BZ)	2',3,4,4',5-Pentachlorobiphenyl	65510-44-3
PCB 22 (BZ)	2,3,4'-Trichlorobiphenyl	38444-85-8	PCB 124 (BZ)	2',3,4,5,5'-Pentachlorobiphenyl	70424-70-3
PCB 23 (BZ)	2,3,5-Trichlorobiphenyl	55720-44-0	PCB 125 (BZ)	2',3,4,5,6'-Pentachlorobiphenyl	74472-39-2
PCB 24 (BZ)	2,3,6-Trichlorobiphenyl	55702-45-9	PCB 126 (BZ)	3,3',4,4',5-Pentachlorobiphenyl	57465-28-8
PCB 25 (BZ)	2,3',4-Trichlorobiphenyl	55712-37-3	PCB 127 (BZ)	3,3',4,5,5'-Pentachlorobiphenyl	39635-33-1
PCB 26 (BZ)	2,3',5-Trichlorobiphenyl	38444-81-4	PCB 128 (BZ)	2,2',3,3',4,4'-Hexachlorobiphenyl	38380-07-3
PCB 27 (BZ)	2,3',6-Trichlorobiphenyl	38444-76-7	PCB 129 (BZ)	2,2',3,3',4,5-Hexachlorobiphenyl	55215-18-4
PCB 28 (BZ)	2,4,4'-Trichlorobiphenyl	7012-37-5	PCB 130 (BZ)	2,2',3,3',4,5'-Hexachlorobiphenyl	52663-66-8
PCB 29 (BZ)	2,4,5-Trichlorobiphenyl	15862-07-4	PCB 131 (BZ)	2,2',3,3',4,6-Hexachlorobiphenyl	61798-70-7
PCB 30 (BZ)	2,4,6-Trichlorobiphenyl	35693-92-6	PCB 132 (BZ)	2,2',3,3',4,6'-Hexachlorobiphenyl	38380-05-1
PCB 31 (BZ)	2,4',5-Trichlorobiphenyl	16606-02-3	PCB 133 (BZ)	2,2',3,3',5,5'-Hexachlorobiphenyl	35694-04-3
PCB 32 (BZ)	2,4',6-Trichlorobiphenyl	38444-77-8	PCB 134 (BZ)	2,2',3,3',5,6-Hexachlorobiphenyl	52704-70-8
PCB 33 (BZ)	2',3,4-Trichlorobiphenyl	38444-86-9	PCB 135 (BZ)	2,2',3,3',5,6'-Hexachlorobiphenyl	52744-13-5
PCB 34 (BZ)	2',3,5-Trichlorobiphenyl	37680-68-5	PCB 136 (BZ)	2,2',3,3',6,6'-Hexachlorobiphenyl	38411-22-2
PCB 35 (BZ)	3,3',4-Trichlorobiphenyl	37680-69-6	PCB 137 (BZ)	2,2',3,4,4',5-Hexachlorobiphenyl	35694-06-5
PCB 36 (BZ)	3,3',5-Trichlorobiphenyl	38444-87-0	PCB 138 (BZ)	2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2
PCB 37 (BZ)	3,4,4'-Trichlorobiphenyl	38444-90-5	PCB 139 (BZ)	2,2',3,4,4',6-Hexachlorobiphenyl	56030-56-9
PCB 38 (BZ)	3,4,5-Trichlorobiphenyl	53555-66-1	PCB 140 (BZ)	2,2',3,4,4',6'-Hexachlorobiphenyl	59291-64-4
PCB 39 (BZ)	3,4',5-Trichlorobiphenyl	38444-88-1	PCB 141 (BZ)	2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6
PCB 40 (BZ)	2,2',3,3'-Tetrachlorobiphenyl	38444-93-8	PCB 142 (BZ)	2,2',3,4,5,6-Hexachlorobiphenyl	41411-61-4
PCB 41 (BZ)	2,2',3,4-Tetrachlorobiphenyl	52663-59-9	PCB 143 (BZ)	2,2',3,4,5,6'-Hexachlorobiphenyl	68194-15-0
PCB 42 (BZ)	2,2',3,4'-Tetrachlorobiphenyl	36559-22-5	PCB 144 (BZ)	2,2',3,4,5',6-Hexachlorobiphenyl	68194-14-9
PCB 43 (BZ)	2,2',3,5-Tetrachlorobiphenyl	70362-46-8	PCB 145 (BZ)	2,2',3,4,6,6'-Hexachlorobiphenyl	74472-40-5
PCB 44 (BZ)	2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	PCB 146 (BZ)	2,2',3,4',5,5'-Hexachlorobiphenyl	51908-16-8
PCB 45 (BZ)	2,2',3,6-Tetrachlorobiphenyl	70362-45-7	PCB 147 (BZ)	2,2',3,4',5,6-Hexachlorobiphenyl	68194-13-8
PCB 46 (BZ)	2,2',3,6'-Tetrachlorobiphenyl	41464-47-5	PCB 148 (BZ)	2,2',3,4',5,6'-Hexachlorobiphenyl	74472-41-6
			PCB 149 (BZ)	2,2',3,4',5',6-Hexachlorobiphenyl	38380-04-0
			PCB 150 (BZ)	2,2',3,4',6,6'-Hexachlorobiphenyl	68194-08-1
			PCB 151 (BZ)	2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5

SYNAME	Compound	CAS No	SYNAME	Compound	CAS No
PCB 47 (BZ)	2,2',4,4'-Tetrachlorobiphenyl	2437-79-8	PCB 152 (BZ)	2,2',3,5,6,6'-Hexachlorobiphenyl	68194-09-2
PCB 48 (BZ)	2,2',4,5'-Tetrachlorobiphenyl	70362-47-9	PCB 153 (BZ)	2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1
PCB 49 (BZ)	2,2',4,5'-Tetrachlorobiphenyl	41464-40-8	PCB 154 (BZ)	2,2',4,4',5,6'-Hexachlorobiphenyl	60145-22-4
PCB 50 (BZ)	2,2',4,6'-Tetrachlorobiphenyl	62796-65-0	PCB 155 (BZ)	2,2',4,4',6,6'-Hexachlorobiphenyl	33979-03-2
PCB 51 (BZ)	2,2',4,6'-Tetrachlorobiphenyl	68194-04-7	PCB 156 (BZ)	2,3,3',4,4',5'-Hexachlorobiphenyl	38380-08-4
PCB 52 (BZ)	2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	PCB 157 (BZ)	2,3,3',4,4',5'-Hexachlorobiphenyl	69782-90-7
PCB 53 (BZ)	2,2',5,6'-Tetrachlorobiphenyl	41464-41-9	PCB 158 (BZ)	2,3,3',4,4',6'-Hexachlorobiphenyl	74472-42-7
PCB 54 (BZ)	2,2',6,6'-Tetrachlorobiphenyl	15968-05-5	PCB 159 (BZ)	2,3,3',4,5,5'-Hexachlorobiphenyl	39635-35-3
PCB 55 (BZ)	2,3,3',4'-Tetrachlorobiphenyl	74338-24-2	PCB 160 (BZ)	2,3,3',4,5,6'-Hexachlorobiphenyl	41411-62-5
PCB 56 (BZ)	2,3,3',4'-Tetrachlorobiphenyl	41464-43-1	PCB 161 (BZ)	2,3,3',4,5,6'-Hexachlorobiphenyl	74472-43-8
PCB 57 (BZ)	2,3,3',5'-Tetrachlorobiphenyl	70424-67-8	PCB 162 (BZ)	2,3,3',4',5,5'-Hexachlorobiphenyl	39635-34-2
PCB 58 (BZ)	2,3,3',5'-Tetrachlorobiphenyl	41464-49-7	PCB 163 (BZ)	2,3,3',4',5,6'-Hexachlorobiphenyl	74472-44-9
PCB 59 (BZ)	2,3,3',6'-Tetrachlorobiphenyl	74472-33-6	PCB 164 (BZ)	2,3,3',4',5',6'-Hexachlorobiphenyl	74472-45-0
PCB 60 (BZ)	2,3,4,4'-Tetrachlorobiphenyl	33025-41-1	PCB 165 (BZ)	2,3,3',5,5',6'-Hexachlorobiphenyl	74472-46-1
PCB 61 (BZ)	2,3,4,5'-Tetrachlorobiphenyl	33284-53-6	PCB 166 (BZ)	2,3,4,4',5,6'-Hexachlorobiphenyl	41411-63-6
PCB 62 (BZ)	2,3,4,6'-Tetrachlorobiphenyl	54230-22-7	PCB 167 (BZ)	2,3',4,4',5,5'-Hexachlorobiphenyl	52663-72-6
PCB 63 (BZ)	2,3,4',5'-Tetrachlorobiphenyl	74472-34-7	PCB 168 (BZ)	2,3',4,4',5',6'-Hexachlorobiphenyl	59291-65-5
PCB 64 (BZ)	2,3,4',6'-Tetrachlorobiphenyl	52663-58-8	PCB 169 (BZ)	3,3',4,4',5,5'-Hexachlorobiphenyl	32774-16-6
PCB 65 (BZ)	2,3,5,6'-Tetrachlorobiphenyl	33284-54-7	PCB 170 (BZ)	2,2',3,3',4,4',5'-Heptachlorobiphenyl	35065-30-6
PCB 66 (BZ)	2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	PCB 171 (BZ)	2,2',3,3',4,4',6'-Heptachlorobiphenyl	52663-71-5
PCB 67 (BZ)	2,3',4,5'-Tetrachlorobiphenyl	73575-53-8	PCB 172 (BZ)	2,2',3,3',4,5,5'-Heptachlorobiphenyl	52663-74-8
PCB 68 (BZ)	2,3',4,5'-Tetrachlorobiphenyl	73575-52-7	PCB 173 (BZ)	2,2',3,3',4,5,6'-Heptachlorobiphenyl	68194-16-1
PCB 69 (BZ)	2,3',4,6'-Tetrachlorobiphenyl	60233-24-1	PCB 174 (BZ)	2,2',3,3',4,5,6'-Heptachlorobiphenyl	38411-25-5
PCB 70 (BZ)	2,3',4',5'-Tetrachlorobiphenyl	32598-11-1	PCB 175 (BZ)	2,2',3,3',4,5',6'-Heptachlorobiphenyl	40186-70-7
PCB 71 (BZ)	2,3',4',6'-Tetrachlorobiphenyl	41464-46-4	PCB 176 (BZ)	2,2',3,3',4,6,6'-Heptachlorobiphenyl	52663-65-7
PCB 72 (BZ)	2,3',5,5'-Tetrachlorobiphenyl	41464-42-0	PCB 177 (BZ)	2,2',3,3',4',5,6'-Heptachlorobiphenyl	52663-70-4
PCB 73 (BZ)	2,3',5',6'-Tetrachlorobiphenyl	74338-23-1	PCB 178 (BZ)	2,2',3,3',5,5',6'-Heptachlorobiphenyl	52663-67-9
PCB 74 (BZ)	2,4,4',5'-Tetrachlorobiphenyl	32690-93-0	PCB 179 (BZ)	2,2',3,3',5,6,6'-Heptachlorobiphenyl	52663-64-6
-PCB 75 (BZ)	2,4,4',6'-Tetrachlorobiphenyl	32598-12-2	PCB 180 (BZ)	2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3
PCB 76 (BZ)	2',3,4,5'-Tetrachlorobiphenyl	70362-48-0	PCB 181 (BZ)	2,2',3,4,4',5,6'-Heptachlorobiphenyl	74472-47-2
PCB 77 (BZ)	3,3',4,4'-Tetrachlorobiphenyl	32598-13-3	PCB 182 (BZ)	2,2',3,4,4',5,6'-Heptachlorobiphenyl	60145-23-5
PCB 78 (BZ)	3,3',4,5'-Tetrachlorobiphenyl	70362-49-1	PCB 183 (BZ)	2,2',3,4,4',5',6'-Heptachlorobiphenyl	52663-69-1
PCB 79 (BZ)	3,3',4,5'-Tetrachlorobiphenyl	41464-48-6	PCB 184 (BZ)	2,2',3,4,4',6,6'-Heptachlorobiphenyl	74472-48-3
PCB 80 (BZ)	3,3',5,5'-Tetrachlorobiphenyl	33284-52-5	PCB 185 (BZ)	2,2',3,4,5,5',6'-Heptachlorobiphenyl	52712-05-7
PCB 81 (BZ)	3,4,4',5'-Tetrachlorobiphenyl	70362-50-4	PCB 186 (BZ)	2,2',3,4,5,6,6'-Heptachlorobiphenyl	74472-49-4
PCB 82 (BZ)	2,2',3,3',4'-Pentachlorobiphenyl	52663-62-4	PCB 187 (BZ)	2,2',3,4',5,5',6'-Heptachlorobiphenyl	52663-68-0

SYNAME	Compound	CAS No	SYNAME	Compound	CAS No
PCB 83 (BZ)	2,2',3,3',5'- Pentachlorobiphenyl	60145-20-2	PCB 188 (BZ)	2,2',3,4',5,6,6'- Heptachlorobiphenyl	74487-85-7
PCB 84 (BZ)	2,2',3,3',6'- Pentachlorobiphenyl	52663-60-2	PCB 189 (BZ)	2,3,3',4,4',5,5'- Heptachlorobiphenyl	39635-31-9
PCB 85 (BZ)	2,2',3,4,4'- Pentachlorobiphenyl	65510-45-4	PCB 190 (BZ)	2,3,3',4,4',5,6-Heptachlorobiphenyl	41411-64-7
PCB 86 (BZ)	2,2',3,4,5-Pentachlorobiphenyl	55312-69-1	PCB 191 (BZ)	2,3,3',4,4',5',6- Heptachlorobiphenyl	74472-50-7
PCB 87 (BZ)	2,2',3,4,5'- Pentachlorobiphenyl	38380-02-8	PCB 192 (BZ)	2,3,3',4,5,5',6-Heptachlorobiphenyl	74472-51-8
PCB 88 (BZ)	2,2',3,4,6-Pentachlorobiphenyl	55215-17-3	PCB 193 (BZ)	2,3,3',4',5,5',6- Heptachlorobiphenyl	69782-91-8
PCB 89 (BZ)	2,2',3,4,6'- Pentachlorobiphenyl	73575-57-2	PCB 194 (BZ)	2,2',3,3',4,4',5,5'- Octachlorobiphenyl	35694-08-7
PCB 90 (BZ)	2,2',3,4',5'- Pentachlorobiphenyl	68194-07-0	PCB 195 (BZ)	2,2',3,3',4,4',5,6- Octachlorobiphenyl	52663-78-2
PCB 71 (BZ)	2,3',4',6-Tetrachlorobiphenyl	41464-46-4	PCB 176 (BZ)	2,2',3,3',4,6,6'- Heptachlorobiphenyl	52663-65-7
PCB 72 (BZ)	2,3',5,5'-Tetrachlorobiphenyl	41464-42-0	PCB 177 (BZ)	2,2',3,3',4',5,6- Heptachlorobiphenyl	52663-70-4
PCB 73 (BZ)	2,3',5',6-Tetrachlorobiphenyl	74338-23-1	PCB 178 (BZ)	2,2',3,3',5,5',6- Heptachlorobiphenyl	52663-67-9
PCB 74 (BZ)	2,4,4',5-Tetrachlorobiphenyl	32690-93-0	PCB 179 (BZ)	2,2',3,3',5,6,6'- Heptachlorobiphenyl	52663-64-6
PCB 75 (BZ)	2,4,4',6-Tetrachlorobiphenyl	32598-12-2	PCB 180 (BZ)	2,2',3,4,4',5,5'- Heptachlorobiphenyl	35065-29-3
PCB 76 (BZ)	2',3,4,5-Tetrachlorobiphenyl	70362-48-0	PCB 181 (BZ)	2,2',3,4,4',5,6-Heptachlorobiphenyl	74472-47-2
PCB 77 (BZ)	3,3',4,4'-Tetrachlorobiphenyl	32598-13-3	PCB 182 (BZ)	2,2',3,4,4',5,6'- Heptachlorobiphenyl	60145-23-5
PCB 78 (BZ)	3,3',4,5-Tetrachlorobiphenyl	70362-49-1	PCB 183 (BZ)	2,2',3,4,4',5',6- Heptachlorobiphenyl	52663-69-1
PCB 79 (BZ)	3,3',4,5'-Tetrachlorobiphenyl	41464-48-6	PCB 184 (BZ)	2,2',3,4,4',6,6'- Heptachlorobiphenyl	74472-48-3
PCB 80 (BZ)	3,3',5,5'-Tetrachlorobiphenyl	33284-52-5	PCB 185 (BZ)	2,2',3,4,5,5',6-Heptachlorobiphenyl	52712-05-7
PCB 81 (BZ)	3,4,4',5-Tetrachlorobiphenyl	70362-50-4	PCB 186 (BZ)	2,2',3,4,5,6,6'-Heptachlorobiphenyl	74472-49-4
PCB 82 (BZ)	2,2',3,3',4- Pentachlorobiphenyl	52663-62-4	PCB 187 (BZ)	2,2',3,4',5,5',6- Heptachlorobiphenyl	52663-68-0
PCB 83 (BZ)	2,2',3,3',5- Pentachlorobiphenyl	60145-20-2	PCB 188 (BZ)	2,2',3,4',5,6,6'- Heptachlorobiphenyl	74487-85-7
PCB 84 (BZ)	2,2',3,3',6- Pentachlorobiphenyl	52663-60-2	PCB 189 (BZ)	2,3,3',4,4',5,5'- Heptachlorobiphenyl	39635-31-9
PCB 85 (BZ)	2,2',3,4,4'- Pentachlorobiphenyl	65510-45-4	PCB 190 (BZ)	2,3,3',4,4',5,6-Heptachlorobiphenyl	41411-64-7
PCB 86 (BZ)	2,2',3,4,5-Pentachlorobiphenyl	55312-69-1	PCB 191 (BZ)	2,3,3',4,4',5',6- Heptachlorobiphenyl	74472-50-7
PCB 87 (BZ)	2,2',3,4,5'- Pentachlorobiphenyl	38380-02-8	PCB 192 (BZ)	2,3,3',4,5,5',6-Heptachlorobiphenyl	74472-51-8
PCB 88 (BZ)	2,2',3,4,6-Pentachlorobiphenyl	55215-17-3	PCB 193 (BZ)	2,3,3',4',5,5',6- Heptachlorobiphenyl	69782-91-8
PCB 89 (BZ)	2,2',3,4,6'- Pentachlorobiphenyl	73575-57-2	PCB 194 (BZ)	2,2',3,3',4,4',5,5'- Octachlorobiphenyl	35694-08-7

SYNAME	Compound	CAS No	SYNAME	Compound	CAS No
PCB 90 (BZ)	2,2',3,4',5-Pentachlorobiphenyl	68194-07-0	PCB 195 (BZ)	2,2',3,3',4,4',5,6-Octachlorobiphenyl	52663-78-2
PCB 91 (BZ)	2,2',3,4',6-Pentachlorobiphenyl	68194-05-8	PCB 196 (BZ)	2,2',3,3',4,4',5,6'-Octachlorobiphenyl	42740-50-1
PCB 92 (BZ)	2,2',3,5,5'-Pentachlorobiphenyl	52663-61-3	PCB 197 (BZ)	2,2',3,3',4,4',6,6'-Octachlorobiphenyl	33091-17-7
PCB 93 (BZ)	2,2',3,5,6-Pentachlorobiphenyl	73575-56-1	PCB 198 (BZ)	2,2',3,3',4,5,5',6-Octachlorobiphenyl	68194-17-2
PCB 94 (BZ)	2,2',3,5,6'-Pentachlorobiphenyl	73575-55-0	PCB 199 (BZ)	2,2',3,3',4,5,6,6'-Octachlorobiphenyl	52663-73-7
PCB 95 (BZ)	2,2',3,5,6-Pentachlorobiphenyl	38379-99-6	200 (IUPAC)	2,2',3,3',4,5',6,6'-Octachlorobiphenyl	40186-71-8
PCB 96 (BZ)	2,2',3,6,6'-Pentachlorobiphenyl	73575-54-9	PCB 200 (BZ)	2,2',3,3',4,5',6,6'-Octachlorobiphenyl	40186-71-8
PCB 97 (BZ)	2,2',3',4,5-Pentachlorobiphenyl	41464-51-1	PCB 201 (BZ)	2,2',3,3',4,5,5',6'-Octachlorobiphenyl	52663-75-9
PCB 98 (BZ)	2,2',3',4,6-Pentachlorobiphenyl	60233-25-2	199 (IUPAC)	2,2',3,3',5,5',6,6'-Octachlorobiphenyl	2136-99-4
PCB 99 (BZ)	2,2',4,4',5-Pentachlorobiphenyl	38380-01-7	PCB 202 (BZ)	2,2',3,4,4',5,5',6-Octachlorobiphenyl	52663-76-0
PCB 100 (BZ)	2,2',4,4',6-Pentachlorobiphenyl	39485-83-1	PCB 203 (BZ)	2,2',3,4,4',5,6,6'-Octachlorobiphenyl	74472-52-9
PCB 101 (BZ)	2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	PCB 204 (BZ)	2,3,3',4,4',5,5',6-Octachlorobiphenyl	74472-53-0
PCB 102 (BZ)	2,2',4,5,6-Pentachlorobiphenyl	68194-06-9	PCB 205 (BZ)	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9
PCB 103 (BZ)	2,2',4,5,6-Pentachlorobiphenyl	60145-21-3	PCB 206 (BZ)	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl	52663-79-3
PCB 104 (BZ)	2,2',4,6,6'-Pentachlorobiphenyl	56558-16-8	PCB 207 (BZ)	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl	52663-77-1
PCB 105 (BZ)	2,3,3',4,4'-Pentachlorobiphenyl	32598-14-4	PCB 208 (BZ)	2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl	2051-24-3
PCB 105 (BZ)	2,3,3',4,4'-Pentachlorobiphenyl	32598-14-4	PCB 209 (BZ)	Decachlorobiphenyl	2051-24-3

**Table VIII**  
**Method 1668C QC Acceptance Criteria for VER, IPR, OPR and Labeled compounds in samples**

Congener Name	Test Concentration (ng/ml)	VER (%)	IPR RSD (%)	IPR Mean Recovery (%)	OPR Recovery (%)	Labeled compound Recovery in Samples (%)
PCB-1	100	75-125	25	70-130	60-135	NA
PCB-3	100	75-125	25	70-130	60-135	
PCB-4	100	75-125	25	70-130	60-135	
PCB-15	100	75-125	25	70-130	60-135	
PCB-19	100	75-125	25	70-130	60-135	
PCB-37	100	75-125	25	70-130	60-135	
PCB-54	100	75-125	25	70-130	60-135	
PCB-77	100	75-125	25	70-130	60-135	
PCB-81	100	75-125	25	70-130	60-135	
PCB-104	100	75-125	25	70-130	60-135	
PCB-105	100	75-125	25	70-130	60-135	
PCB-114	100	75-125	25	70-130	60-135	
PCB-118	100	75-125	25	70-130	60-135	
PCB-123	100	75-125	25	70-130	60-135	
PCB-126	100	75-125	25	70-130	60-135	
PCB-155	100	75-125	25	70-130	60-135	
PCB-156	100	75-125	25	70-130	60-135	
PCB-157	100	75-125	25	70-130	60-135	
PCB-167	100	75-125	25	70-130	60-135	
PCB-169	100	75-125	25	70-130	60-135	
PCB-188	100	75-125	25	70-130	60-135	
PCB-189	100	75-125	25	70-130	60-135	
PCB-202	100	75-125	25	70-130	60-135	
PCB-205	100	75-125	25	70-130	60-135	
PCB-206	100	75-125	25	70-130	60-135	
PCB-208	100	75-125	25	70-130	60-135	
PCB-209	100	75-125	25	70-130	60-135	
Internal Standards						
<sup>13</sup> C <sub>12</sub> -PCB-1	100	50-145	70	20-135	15-145	5-145
<sup>13</sup> C <sub>12</sub> -PCB-3	100	50-145	70	20-135	15-145	5-145
<sup>13</sup> C <sub>12</sub> -PCB-4	100	50-145	70	20-135	15-145	5-145
<sup>13</sup> C <sub>12</sub> -PCB-15	100	50-145	70	20-135	15-145	5-145
<sup>13</sup> C <sub>12</sub> -PCB-19	100	50-145	70	20-135	15-145	5-145
<sup>13</sup> C <sub>12</sub> -PCB-37	100	50-145	70	20-135	15-145	5-145
<sup>13</sup> C <sub>12</sub> -PCB-54	100	50-145	70	20-135	15-145	5-145
<sup>13</sup> C <sub>12</sub> -PCB-77	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-81	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-104	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-105	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-114	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-118	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-123	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-126	100	50-145	50	45-135	40-145	10-145

<sup>13</sup> C <sub>12</sub> -PCB-155	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-156	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-157	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-167	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-169	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-188	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-189	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-202	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-205	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-206	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-208	100	50-145	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-209	100	50-145	50	45-135	40-145	10-145
Cleanup Standards						
<sup>13</sup> C <sub>12</sub> -PCB-28	100	65-135	70	20-135	15-145	5-145
<sup>13</sup> C <sub>12</sub> -PCB-111	100	75-125	50	45-135	40-145	10-145
<sup>13</sup> C <sub>12</sub> -PCB-178	100	75-125	50	45-135	40-145	10-145