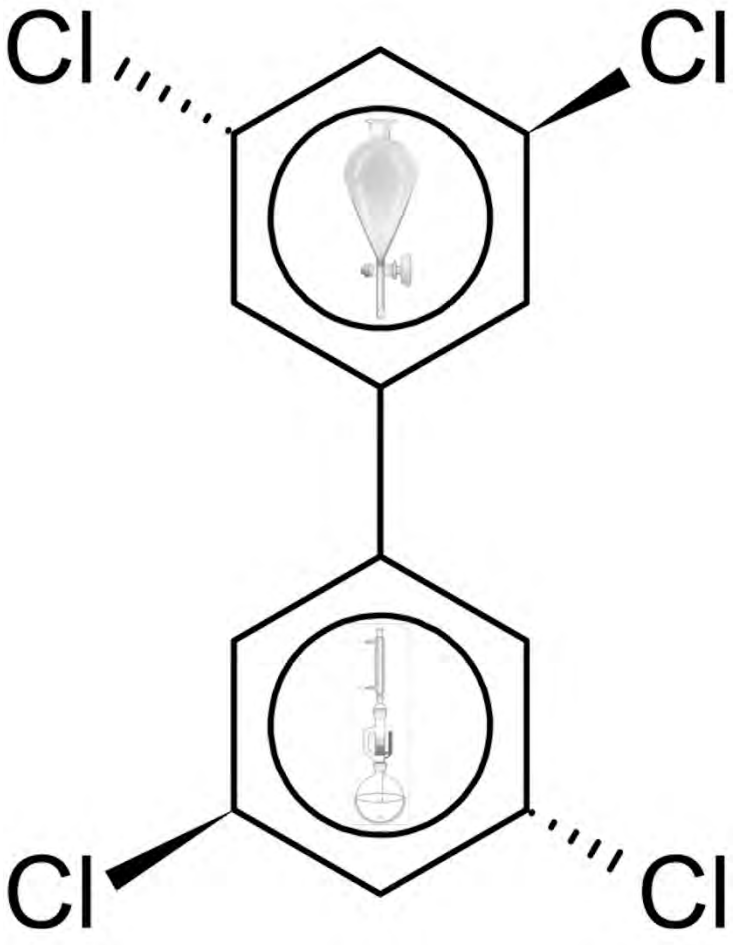
United States Environmental Protection Agency

Office of Water

www.epa.gov July 2021

Method 1628 Polychlorinated Biphenyl (PCB) Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Low-resolution GC/MS using Selected Ion Monitoring



U.S. Environmental Protection Agency
Office of Water (4303T)
Office of Science and Technology
Engineering and Analysis Division
1200 Pennsylvania Avenue, NW
Washington, DC 20460

EPA 821-R-21-002

Acknowledgements

This method was prepared under the direction of Adrian Hanley of the Engineering and Analysis Division, Office of Science and Technology, within EPA's Office of Water. EPA acknowledges the support of a number of organizations in the development and validation of this PCB congener method, including the developers of the original procedure, SGS-AXYS Analytical, Ltd., the members of EPA's workgroup, the laboratories that participated in the multi-laboratory method validation study, and EPA's support contractor staff at General Dynamics Information Technology, including Mirna Alpizar, Eric Boring, and Harry McCarty. (A more detailed list of individuals and organizations can be found in Reference 13).

Disclaimer

Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Contact

Please address questions, comments, or suggestions to:

CWA Methods Team, Engineering and Analysis Division (4303T)
Office of Science and Technology
U.S. Environmental Protection Agency
1200 Pennsylvania Avenue
Washington, DC 20460

https://www.epa.gov/cwa-methods
https://www.epa.gov/cwa-methods/forms/contact-us-about-cwa-analytical-methods

Table of Contents

Method 1628
Polychlorinated Biphenyl (PCB) Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Low-Resolution GC/MS Using Selected Ion Monitoring

July 2021

1.0 Scope and Application

1.1 Method 1628 is for determination of all 209 polychlorinated biphenyl (PCBs) congeners (see Table 1) in wastewater and other matrices, by low-resolution gas chromatography/mass spectrometry (GC/MS) using selected ion monitoring (SIM).

1.1.1 The method calibrates and quantifies 65 PCB congeners selected by EPA as priorities because of their:

- chromatographic retention times on the column used for this analysis (e.g., first and last eluting congeners in a level of chlorination),
- prevalence in environmental samples,
- high concentrations in Aroclors, and
- their toxicities (e.g., the World Health Organization's list of dioxin-like PCB congeners)

See Section 10.5 and Table 6 for the type of calibration and quantitation reference for each of the 65 congeners. Twenty-three congeners are calibrated by isotope dilution (the response of the target congener is compared to the response of its $^{13}C_{12}$-labeled analog). Fourteen congeners are calibrated by modified isotope dilution (a congener that coelutes with a congener calibrated by isotope dilution). Twenty-eight congeners are quantified by extracted internal standard quantification (the response of the target congener is compared to the response of the $^{13}C_{12}$-labeled analog of another congener in the same level of chlorination [LOC]).

1.1.2 The remaining 144 congeners are quantified indirectly using isotope dilution standards of similar congeners with the same level of chlorination. This approach may produce greater uncertainty in the results for these congeners; however, if any of the 144 congeners is a priority for a particular application, the method allows the flexibility to calibrate additional congeners using labeled standards for such congeners.

1.2 EPA developed this method for use in Clean Water Act (CWA) programs for wastewater, surface water, soil, sediment, biosolids, and tissue matrices. Other applications and matrices may be possible, which may or may not require modifications of sample preparation, chromatographic conditions, etc.

1.3 The detection limits and quantitation levels in this method may be dependent on the levels of interferences and laboratory background levels rather than instrumental limitations. The method detection limits (MDL) and minimum levels (ML) of quantitation given in Table 12 are the pooled MDL_s and ML values determined during the multi-laboratory method validation study (Reference 13).

1.4 The instrumental portions of this method are for use only by analysts experienced with low-resolution GC/MS SIM or under the close supervision of such qualified persons. Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in Section 9.2.

1.5 This method is "performance-based," which means that the analyst may make modifications to improve performance (e.g., overcome interferences, or improve the sensitivity, accuracy or precision of the results) without additional EPA review, *provided that* all performance criteria in this method are met. Requirements for establishing equivalency are in Section 9.1.2, and include 9.1.2.2 c. For CWA uses, additional flexibility is described at 40 CFR 136.6. The laboratory must document changes in performance, sensitivity, selectivity, precision, recovery, etc., that result from modifications within the scope of 40 CFR Part 136.6, and Section 9 of this method, and document how these modifications compare to the specifications in this method. Changes outside the scope of 40 CFR Part 136.6 and Section 9 of this method may require prior review or approval.

2.0 Summary of Method

Environmental samples are prepared and extracted using matrix-specific procedures. Sample extracts are subjected to various cleanup procedures designed to remove interferences and concentrated to a low final volume. Analyses are conducted by low-resolution GC/MS with selected ion monitoring (SIM) to identify and quantify the PCB congeners by monitoring a primary and a secondary ion for each native PCB and labeled compound.

2.1 Extraction

2.1.1 Aqueous samples containing less than one percent solids – An aliquot of labeled compound standard is spiked into the 1-L sample. The sample is extracted using a cartridge-based solid-phase extraction (SPE) system, a disk-based SPE system, or separatory funnel extraction (SFE). The extract is dried and concentrated for cleanup using rotary evaporation and nitrogen evaporation.

2.1.2 Aqueous samples containing greater than one percent solids – An aliquot of labeled compound standard is spiked into a 1-L sample. The sample is centrifuged, and the supernatant collected and extracted using a cartridge-based SPE system, a disk-based SPE system, or separatory funnel extraction (SFE). The particulate matter is extracted as a solid sample as described in Section 2.1.3. The two extracts are dried, combined, and concentrated for cleanup using rotary evaporation and nitrogen evaporation, prior to analysis.

2.1.3 Solid samples (excluding tissue) – A 10-g aliquot (dry-weight) of a homogenized soil/sediment sample is mixed with anhydrous granular sodium sulfate and allowed to dry. (For biosolid samples, a 5-g aliquot dry-weight is used.) An aliquot of labeled compound standard is spiked into the sample. The sample is transferred to a Soxhlet thimble and extracted for a minimum of 16 hours using 1:1 hexane:acetone in a Soxhlet extractor. The extract is dried and concentrated for cleanup using rotary evaporation and nitrogen evaporation.

2.1.4 Tissue – A 10-g aliquot (wet-weight) of homogenized sample is mixed with anhydrous granular sodium sulfate and allowed to dry. An aliquot of labeled compound standard is spiked into the sample. The sample is transferred to a Soxhlet thimble and extracted for a minimum of 16 hours using dichloromethane in a Soxhlet extractor. The extract is concentrated for cleanup using rotary evaporation and nitrogen evaporation.

2.2 The default cleanup for tissue samples is a gel permeation column, followed by Florisil® column cleanup. For aqueous and other solid matrices, the extract is treated with copper to remove sulfur, followed by Florisil® column cleanup. Additional cleanup procedures may be used prior to the Florisil® column cleanup as needed (i.e., if the extract appears discolored).

2.3 After cleanup, the extract is concentrated to 60 µL and 40 µL of non-extracted internal standard is then added. An aliquot of the extract is injected into the gas chromatograph (GC). The analytes are separated by the GC and detected by a low-resolution mass spectrometer (LRMS) operated in SIM mode.

2.4 An individual PCB congener is identified through peak analysis of the quantification and confirmation ions.

2.5 Quantitative determination of target compound concentration is made with respect to a labeled PCB congener; the concentrations are then used to convert raw peak areas in sample chromatograms to final concentrations.

2.5.1 Results for target compounds are recovery corrected by the method of quantification (i.e., isotope dilution, modified isotope dilution, or extracted internal standard quantification). Labeled compound recoveries are determined similarly against the non-extracted internal standard (i.e., a recovery standard) and are used as general indicators of overall analytical quality.

2.5.2 The results for six congeners (PCBs 35, 77, 81, 123, 126, and 157) are corrected for higher homolog interference by area subtraction (Section 16.3 and Table 11).

2.6 The quality of the analysis is assured through reproducible calibration and testing of the extraction, cleanup, and GC/MS systems.

3.0 Definitions

Definitions are provided in the glossary at the end of this method.

4.0 Contamination and interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and elevated baselines causing misinterpretation of chromatograms. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Where possible, reagents are cleaned by extraction or solvent rinse. Glassware should be purchased as certified clean or solvent-rinsed or baked in a kiln or furnace at 450 - 500 ºC in order to remove these and other contaminants. Baked glassware must be rinsed with dichloromethane prior to use.

4.2 Proper cleaning of glassware is extremely important, because glassware may not only contaminate the samples, but may also remove the analytes of interest by adsorption on the glass surface.

4.2.1 Glassware should be rinsed with solvent (using the same solvent that was last used on that piece of glassware) and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with fluoropolymer stopcocks, must be disassembled prior to detergent washing.

4.2.2 After detergent washing, glassware should be rinsed immediately, first with methanol, then with hot tap water followed by another methanol rinse, then acetone, and finished with a dichloromethane rinse.

4.2.3 Baking of glassware in a kiln or other high temperature furnace (450 - 500 ºC) may be warranted after particularly dirty samples are encountered. The kiln or furnace should be vented outside the laboratory, or to a trapping system, to prevent laboratory contamination by PCB vapors. Baking should be minimized, as repeated baking of glassware may cause active sites on the glass surface that may irreversibly adsorb PCBs.

4.2.4 Immediately prior to use, the Soxhlet glassware must be rinsed with dichloromethane.

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

4.3.1 The reference matrix must simulate, as closely as possible, the sample matrix being tested. Ideally, the reference matrix should not contain the PCBs in detectable amounts, but it should contain potential interferents in the concentrations expected to be found in the samples to be analyzed.

4.3.2 When a clean reference matrix that simulates the sample matrix under test is not available, reagent water (Section 7.6.1) can be used to simulate water samples; Ottawa sand or reagent-grade sand (Section 7.6.2) can be used to simulate soils; and a 10:90 w/w mixture of canola oil and Ottawa sand (Section 7.6.3) can be used to simulate tissues.

4.4 Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCBs. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxydiphenyl ethers, benzylphenyl ethers, brominated diphenyl ethers, polynuclear aromatics, polychlorinated naphthalenes, and pesticides. Because low levels of PCBs are measured by this method, elimination of interferences is essential. The cleanup steps given in Section 12.0 can be used to reduce or eliminate these interferences and thereby permit reliable determination of the PCBs at low part per trillion (ppt) levels in aqueous samples and at sub-part per billion (ppb) levels in solid matrices.

4.5 Each piece of reusable glassware may be numbered to associate that glassware with the processing of a particular sample. This may assist the laboratory in tracking possible sources of contamination for individual samples, identifying glassware associated with highly contaminated samples that may require extra cleaning, and determining when glassware should be discarded.

4.6 Cleanup of tissue – The natural lipid content of tissue can interfere in the analysis of tissue samples for the PCBs. The lipid contents of different species and portions of tissue can vary widely. Lipids are soluble to varying degrees in various organic solvents and may be present in sufficient quantity to overwhelm the various column chromatography procedures used for cleanup of sample extracts. Lipids must be removed by the gel permeation chromatography (GPC) procedure in Section 12.3. Florisil® (Section 12.1) is mandatory as the final cleanup step.

4.7 If the laboratory air is a potential source of PCB contamination, samples, reagents, glassware, and other materials should be dried in a glove box or other area free from contamination.

4.8 It is possible that loss of chlorine from a more highly chlorinated congener, as a result of fragmentation in the mass analyzer, may increase the response and produce an enhanced response for a less-chlorinated congener eluting at the same retention time. This effect may be significant for certain “toxic” congeners assigned toxicity equivalency factor values by the World Health Organization (WHO). Based on the chromatographic conditions in this method, PCBs 35, 77, 81,

123, 126, and 157 co-elute with a more highly chlorinated congener and the results can be corrected for the potential contribution of those congeners (see Table 11). However, the results for those six congeners should be interpreted as maximum values, due to the potential for interferences (see Section 16.1.5). There may be instances where the response for other congeners is elevated; however, the effect in relation to the overall PCB content will be negligible; therefore, the concentrations of those other congeners do not need to be qualified as maximum values. See the note in Section 9.1.2.3 regarding the use of different GC columns and conditions.

5.0 Safety

5.1 The toxicity or carcinogenicity of each chemical used in this method has not been precisely determined; however, each compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.1.1 PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. On the basis of the available toxicological and physical properties of the PCBs, pure standards should be handled only by highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks.

5.1.2 It is recommended that the laboratory purchase dilute standard solutions of the analytes in this method. However, if primary solutions are prepared, they must be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator must be worn when high concentrations are handled.

5.2 The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of safety data sheets (SDSs) should also be made available to all personnel involved in these analyses. It is also suggested that the laboratory perform personal hygiene monitoring of each analyst who uses this method and that the results of this monitoring be made available to the analyst. Additional information on laboratory safety can be found in References 1 - 4.

5.3 PCB standards, as well as field samples suspected of containing these compounds, must be handled using essentially the same techniques employed in handling radioactive or infectious materials. Well-ventilated, controlled access laboratories are required. Assistance in evaluating the health hazards of particular laboratory conditions may be obtained from certain consulting laboratories and from State Departments of Health or Labor, many of which have an industrial health service. Each laboratory must develop a strict safety program for handling these compounds.

5.3.1 Facility – When finely divided samples (dusts, soils, dry chemicals) are handled, all operations (including removal of samples from sample containers, weighing, transferring, and mixing) should be performed in a glove box demonstrated to be leak tight or in a fume hood demonstrated to have adequate air flow. Gross losses to the laboratory ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no inhalation hazards except in the case of an accident.

5.3.2 Protective equipment – Disposable plastic gloves, apron or lab coat, safety glasses or mask, and a glove box or fume hood adequate for radioactive work should be used. During analytical operations that may give rise to aerosols or dusts, personnel should wear respirators equipped with activated carbon filters. Eye protection (preferably full-face shields) must be worn while working with exposed samples or pure analytical standards. Latex gloves are commonly used to reduce exposure of the hands. When handling samples

suspected or known to contain high concentrations of the PCBs, an additional set of gloves can also be worn beneath the latex gloves.

5.3.3 Training – Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

5.3.4 Personal hygiene – Hands and forearms should be washed thoroughly after each manipulation and before breaks (coffee, lunch, and shift).

5.3.5 Confinement – Isolated work areas posted with signs, segregated glassware and tools, and plastic absorbent paper on bench tops will aid in confining contamination.

5.3.6 Effluent vapors – The effluent of the sample splitter from the gas chromatograph (GC) and from roughing pumps on the mass spectrometer (MS) should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols to condense PCB vapors.

5.3.7 Waste Handling – Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans. Janitors and other personnel should be trained in the safe handling of waste.

5.3.8 Decontamination

5.3.8.1 Decontamination of personnel – Use any mild soap with plenty of scrubbing action.

5.3.8.2 Glassware, tools, and surfaces – Satisfactory cleaning may be accomplished by rinsing with dichloromethane, then washing with any detergent and water. If glassware is first rinsed with solvent, the wash water may be disposed of in the sewer. Given the cost of disposal, it is prudent to minimize solvent wastes.

5.3.9 Laundry – Clothing known to be contaminated should be collected in plastic bags. Persons that convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows of the potential problem. The washer should be run through a cycle before being used again for other clothing.

5.3.10 Wipe tests – A useful method of determining cleanliness of work surfaces and tools is to perform a wipe test of the surface suspected of being contaminated.

5.3.10.1 Wipe an area approximately 10 x 10 cm using a piece of filter paper moistened with dichloromethane or other solvent.

5.3.10.2 Extract and analyze the wipe by GC with an electron capture detector or by this method.

5.3.10.3 Using the area wiped (e.g., 10 x 10 cm = 0.01 m^2), calculate the concentration in $\mu g/m^2$. A concentration less than 1 $\mu g/m^2$ indicates acceptable cleanliness; anything higher warrants further cleaning. More than 100 $\mu g/m^2$ constitutes an acute hazard and requires prompt cleaning before further use of the equipment or workspace and indicates that unacceptable work practices have been employed.

5.4 Biosolids samples may contain high concentrations of biohazards and must be handled with gloves. Containers must be opened in a fume hood or biological safety cabinet to prevent exposure. Laboratory staff should know and observe the safety procedures required in a microbiology laboratory that handles pathogenic organisms when handling biosolids samples.

6.0 Equipment and Supplies

Note: *Brand names, suppliers, and part numbers are for illustration purposes only and no endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here. Meeting the performance requirements of this method is the responsibility of the laboratory.*

6.1 Sampling equipment for discrete or composite sampling

6.1.1 Sample bottles and caps

6.1.1.1 Liquid samples (waters, sludges and similar materials containing 5 percent solids or less) – Sample bottle, amber glass, 1-L minimum, with screw cap and with at least a 53-400 mm opening. Use of pre-cleaned bottles purchased from suppliers who provide a certificate of analysis is encouraged.

6.1.1.2 Solid samples (soils, sediments, sludges, paper pulps, filter cake, compost, and similar materials that contain more than 5 percent solids) – Sample bottle or jar, wide mouth, amber glass, 500-mL minimum. Use of pre-cleaned bottles purchased from suppliers who provide a certificate of analysis is encouraged.

6.1.1.3 If amber bottles are not available, samples must be protected from light.

6.1.1.4 Bottle caps – Threaded to fit sample bottles. Caps must be lined with fluoropolymer.

6.1.1.5 Cleaning

If pre-cleaned bottles are not used, employ the steps below to clean bottles.

6.1.1.5.1 Bottles are detergent water washed, then solvent rinsed before use.

6.1.1.5.2 Liners are detergent water washed and rinsed with reagent water (Section 7.6.1).

6.1.2 Compositing equipment – Automatic or manual compositing system incorporating glass containers cleaned per bottle cleaning procedure above. Only glass or fluoropolymer tubing must be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only. Before use, the tubing must be thoroughly rinsed with methanol, followed by repeated rinsing with reagent water to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.

6.2 Equipment for glassware cleaning

Note: *If blanks from bottles or other glassware, or with fewer cleaning steps than required above, show no detectable PCB contamination, unnecessary cleaning steps and equipment may be eliminated.*

6.2.1 Laboratory sink with overhead fume hood

6.2.2 Kiln – Capable of reaching 450 ºC within 2 hours and maintaining 450 - 500 ºC within ±10ºC, with temperature controller and safety switch (Cress Manufacturing Co., Carson City, NV, B27HT-240, or equivalent). See instructions and precautions in Sections 4.1 and 4.2.3.

6.3 Equipment for preparation of reagents and samples

6.3.1 Laboratory fume hood of sufficient size to contain the sample preparation equipment listed below.

6.3.2 Glove box (optional)

6.3.3 Tissue homogenizer –Macro homogenizer (Pro Scientific Model PRO400DS, or equivalent) with stainless steel Macro-shaft and Turbo-shear blade.

6.3.4 Meat grinder – Hobart, or equivalent, with 3- to 5-mm holes in inner plate.

6.3.5 Equipment for determining percent moisture

6.3.5.1 Oven – Capable of maintaining a temperature of 110 ± 5 ºC

6.3.5.2 Desiccator

6.3.6 Balances

6.3.6.1 Analytical – Capable of weighing 0.1 mg

6.3.6.2 Top loading – Capable of weighing 10 mg

6.3.7 Aluminum foil, to wrap clear sample bottles and fish samples after collection

6.3.8 Disposable plastic spoons for aliquoting solids samples, sterile (Bel-Art Cat # H369400010 or equivalent)

6.3.9 Magnetic stirring plate, with pre-cleaned PTFE-coated magnetic stirring bars

6.3.10 Glass bottles – 1-L, pre-cleaned, PTFE-lined lid, used for storage of reagents

6.3.11 Beakers – 400- to 500-mL

6.3.12 Spatulas – Stainless steel

6.3.13 Round-bottom flask, stoppered

6.3.14 Volumetric flask, 10-mL, Class A

6.3.15 Gas-tight syringes – 5-µL, 10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, 1000-µL (Hamilton or equivalent), accurate to within ±1% of nominal volume with a precision of 1% at 80% of the total volume

6.3.16 Analog or digital vortex mixer – 120V, 4.9 mm orbit, (Fisher Scientific 02-215-414, or equivalent)

6.4 Water sample extraction equipment – Equipment is provided in this section for three types of extraction (separatory funnel extraction, automated cartridge-based SPE extraction, and automated disk-based extraction), but not all equipment is required and is dependent on the extraction procedure that is chosen.

6.4.1 pH meter – with combination glass electrode. Calibrate prior to each use according to manufacturer's instructions.

6.4.2 pH paper – capable of covering a pH range of 1 – 14 (Hydrion® Papers, or equivalent)

6.4.3 Graduated cylinder – 1-L capacity

6.4.4 Liquid/liquid extraction – Separatory funnels, 2-L, with fluoropolymer stopcocks

6.4.5 Erlenmeyer flask - 125-, 250-, and 500-mL, with 19/22 mm neck joint and ground-glass-stopper

6.4.6 Automated cartridge-based SPE extraction system (FMS, Inc. Billerica, MA, EZPrep 123, or equivalent)

6.4.6.1 Extractor system and controller

6.4.6.2 Cartridge adaptors - 6 mL

6.4.6.3 Extraction cartridge - 1 g divinylbenzene (DVB), 60-angstrom pore diameter, 85-µm particle size, FMS part# SPE-CAR1-DVB, or equivalent

6.4.6.4 Cap adaptor

6.4.6.5 Connector tubing

6.4.7 Automated disk-based SPE extraction system (Biotage® Horizon 5000, or equivalent)

6.4.7.1 Extractor system and controller

6.4.7.2 Extraction disk - 0.5 g DVB, 47-mm diameter, Atlantic® part# 47-2346-06, or equivalent

6.4.7.3 Cap adaptor

6.4.7.4 Disk holder assembly (90-mm diameter)

6.4.7.5 Filter paper (Atlantic® fast flow pre-filter or equivalent, glass fiber, 1.0-µm and 5.0-µm, 90-mm diameter, part#: FFAP-100-HS1 and FFAP-100-HS5, or equivalent)

6.5 Solid and tissue sample extraction equipment

Note: *Other extraction equipment and techniques may be useful for solid and tissue samples, including Soxhlet-Dean-Stark (SDS), automated Soxhlet, and others. Laboratories wishing to utilize such techniques are responsible for demonstrating equivalent performance, as described in 40 CFR 136.7 and Section 9 of this procedure. Such demonstrations **must** be performed on reference matrices with similar water contents (soils, sediments, and biosolids) or lipid contents (tissues) in order to be considered valid.*

6.5.1 Soxhlet apparatus

Note: *The extractor and thimble described below are suitable for most solid samples. For very wet sediment samples, or in instances where greater sensitivity is to be achieved by using larger sample sizes, the use of bigger glass thimbles and extractor bodies may be necessary in order to accommodate the mass of the sample and the amount of sodium sulfate required to address the moisture in the sample.*

6.5.1.1 Soxhlet – 50-mm ID, 200-mL extractor capacity with 500-mL flask (Wilmad-LabGlass LG-6900-108, or equivalent, with a 500-mL round-bottom flask)

6.5.1.2 Thimble – 130 mm long by 45 mm ID, medium porosity frit, 10-15 µm (Wilmad-LabGlass LG-7070-124, or equivalent)

6.5.1.3 Heating mantle – Hemispherical, to fit 500-mL round-bottom flask (Wilmad-LabGlass LG-8800-112, or equivalent)

6.5.1.4 Variable transformer – Powerstat (or equivalent), 120-volt, 10-amp (Wilmad-LabGlass LG-8965-100, or equivalent)

6.5.1.5 A recirculating water pump and chiller are recommended, as use of tap water for cooling the condensers wastes large volumes of water.

6.6 Filtration apparatus

6.6.1 Silanized glass wool – stored in a clean glass jar and rinsed with toluene (2 times) and hexane (2 times) prior to use.

6.6.2 Glass funnel – 125- to 250-mL

6.6.3 Filter paper

6.6.3.1 Ahlstrom, glass fiber filter, 161 grade, 1.1-µm pore size, 42.5-mm diameter (Ahlstrom 1610-0425, or equivalent)

6.6.3.2 Pall, glass fiber, A/E grade, 1.0-µm pore size, 102-mm diameter (Pall 61633, or equivalent)

6.7 Centrifuge apparatus

6.7.1 Centrifuge – Capable of rotating 500-mL centrifuge bottles at 5,000 rpm minimum (Accuspin™ Model 400, or equivalent)

6.7.2 Centrifuge bottle – 500-mL glass

6.7.3 Centrifuge tubes – 12- to 15-mL glass, with screw caps, used to concentrate extracts

6.8 Cleanup apparatus

6.8.1 Automated gel permeation chromatograph (LabTech, Inc., Hopkinton, MA, AutoClean GPC, or equivalent)

6.8.1.1 Column – 600-700 mm long × 25 mm ID glass, packed with 70 g of 200-400 mesh S-X3 Bio-Beads® (Bio-Rad Laboratories, Hercules, CA, Support #1522750 or equivalent)

6.8.1.2 Syringe – 10-mL, gas tight, with PTFE Luer lock fitting (Hamilton 81620, or equivalent)

6.8.1.3 Syringe filter holder – stainless steel or polypropylene holder with glass-fiber or fluoropolymer filters (Pall AP- 4310, or equivalent)

6.8.1.4 UV detector – capable of reading 254-nm wavelength

6.8.2 Pipettes

6.8.2.1 Disposable, Pasteur, 5-mm ID x150-mm long (Fisher Scientific 13-678-6A, or equivalent)

6.8.2.2 Disposable, serological, 25-mL, 8- to 10- mm ID, (Fisher Scientific 13-678-36D or equivalent)

6.8.3 Glass chromatographic columns

6.8.3.1 10-mm ID x 150-mm long, with coarse-glass frit or glass-wool plug and 150-mL reservoir

6.8.3.2 10-mm ID x 300-mm long, with coarse-glass frit or glass-wool plug and 300-mL reservoir

6.8.3.3 20-mm ID x 300-mm long, with coarse-glass frit, 250-mL reservoir, and glass or fluoropolymer stopcock

6.8.4 Oven – For baking and storage of adsorbents, capable of maintaining a constant temperature (± 5 ºC) in the range of 105 - 250 ºC

6.9 Concentration apparatus

6.9.1 Rotary evaporator – Buchi Rotavapor™ R-300, Fisher Scientific 05-001-022 or equivalent, equipped with a variable temperature water bath capable of ≤ 30 °C

6.9.1.1 Vacuum source for rotary evaporator equipped with shutoff valve at the evaporator and vacuum gauge

6.9.1.2 A recirculating water pump and chiller are recommended, as use of tap water for cooling the evaporator wastes large volumes of water and can lead to inconsistent performance as water temperatures and pressures vary.

6.9.1.3 Round-bottom flask – 100-, 250-, and 500-mL or larger, with ground-glass fitting compatible with the rotary evaporator

6.9.2 Nitrogen evaporation apparatus – Equipped with water bath controlled in the range of 30 - 60 ºC (N-EVAP, Organomation Associates, Inc., Berlin, MA, Cat. No. 11250, or equivalent), installed in a fume hood

6.10 Vials

6.10.1 Autosampler vials – amber, crimp-top or screw-cap, tapered vial and reusable insert compatible with the GC autosampler, with a volume of at least 1,000 µL (to allow for dilution on an 800-µL extract in the vial), Thermo Fisher Scientific, Waltham, MA, Chromacol™ 1.1-CTV(A), or equivalent

6.10.2 PCB standard storage vials – amber, 15 mL with fluoropolymer-lined screw cap

6.11 Gas chromatograph – The GC system must be capable of temperature programming and have a flow controller that maintains a constant column flowrate throughout the temperature program operations. The system must be suitable for splitless or on-column injection and have all required accessories including syringes, analytical columns, regulators, and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-PTFE thread sealants or flow controllers with rubber components are not to be used. The GC system must meet all of the performance specifications in Section 10.0.

6.12 GC column – Agilent J&W DB-5 chromatography column (60-m length, 0.25-mm ID, 0.10-µm film thickness, Agilent 122-5061) or equivalent

6.13 Mass spectrometer –Capable of operation at 70 eV (nominal) electron energy in the electron ionization (EI) mode, and selectively monitoring at least two *m/z*s for each target analyte and standard at unit mass resolution during a period of less than 1.5 seconds, and must meet the tuning acceptance criteria (Section 10.2).

6.14 GC/MS interface – The laboratory may use any GC/MS interface that provides acceptable sensitivity and QC. However, direct insertion of the GC column into the mass spectrometer source is the recommended interface.

6.15 Data system – Capable of collecting, recording, storing, and processing MS data

6.15.1 Data acquisition – The signal at each exact mass to charge ratios (*m/z*) must be collected repetitively throughout the monitoring period and stored on a mass storage device.

6.15.2 Response factors and multipoint calibrations – The data system must record and maintain lists of response factors (response ratios for isotope dilution) and multipoint calibrations. Computations of relative standard deviation (RSD) are used to test calibration linearity.

6.15.3 Multiple ion monitoring (MIM) or selected ion monitoring (SIM) – The data system must be capable of acquiring data for two characteristic ions for each native congener and labeled compound.

7.0 Reagents and standards

7.1 pH adjustment

7.1.1 Sodium hydroxide, NaOH, Analytical Reagent (AR) Grade, (1 M) - dissolve 40 g sodium hydroxide pellets in 1 L of reagent water, using a magnetic stirrer with stirring bar (Section 6.3.9). Extract the solution twice with 100 mL dichloromethane and once with 100 mL hexane. Remove the organic layer after each extraction. Store in an amber jar with PTFE-lined lid (Section 6.3.10). Typically, 3 L are prepared. Shelf life for this reagent is 3 months from the date of preparation. The reagent may be used after the expiration date; however, the solution must be re-extracted, as described above, and a new expiration date assigned.

7.1.2 Sulfuric acid - (H_2SO_4, conc., Seastar Chemicals, quartz distilled, or equivalent) is used as received.

7.1.3 Hydrochloric acid (HCl) – Reagent grade, either 25% or concentrated

7.2 Solution drying, matrix preparation, and solvent evaporation

7.2.1 Solution drying – Sodium sulfate, reagent grade, granular, anhydrous (12-60 mesh, J.T. Baker or equivalent) - bake at approximately 400 ºC for 1 hour minimum, cool in a desiccator, and store in a pre-cleaned glass bottle with screw cap (Section 6.3.10) that prevents moisture from entering. If, after heating, the sodium sulfate develops a noticeable grayish cast (due to the presence of carbon in the crystal matrix), that batch of reagent is not suitable for use and should be discarded. Extraction with dichloromethane (as opposed to simple rinsing) and baking at a lower temperature may produce sodium sulfate that is suitable for use. The baked sodium sulfate has a shelf life of 1 month. The reagent may be used after the expiration date; however, it must be re-baked and a new expiration date assigned.

7.2.2 Solid and tissue drying – Sodium sulfate, reagent grade, powdered, treated, and stored as in Section 7.2.1.

7.2.3 Pre-purified nitrogen – Clean dry nitrogen filtered through a column of activated carbon or purchased as ultra-high purity nitrogen (≥ 99.998% purity).

7.3 Extraction

7.3.1 Solvents – Acetone, toluene, hexane, methanol, and dichloromethane, distilled in glass, pesticide quality, lot-certified to be free of interferences. All solvent mixtures used in the analyses are made by mixing the appropriate quantities on a volume basis. Shelf life is approximately 2 years for solvent mixtures and must be discarded after this shelf life.

Note: *Some solvents may need to be re-distilled to eliminate PCB backgrounds.*

7.3.2 Anti-bumping granules (Millipore Sigma 107913, or equivalent) are baked for 5 hours at a temperature up to 130 °C prior to use and stored in a clean, baked jar.

7.4 GPC calibration solution – Prepare a solution containing 2.5 mg/mL corn oil, 0.05 mg/mL bis (2-ethylhexyl) phthalate (BEHP), 0.01 mg/mL methoxychlor, 0.002 mg/mL perylene, and 0.008 mg/mL sulfur, or at concentrations appropriate to the response of the detector. The GPC calibration

solution may be purchased in concentrated form (AccuStandard® CLP-027-R2) and diluted 100-fold with 1:1 dichloromethane:hexane.

7.5 Adsorbents for sample cleanup

Note: *Commercially available absorbents of similar compositions to the ones listed in this section, including pre-packed columns or cartridges, may be used provided that equivalency is established according to Section 9.1.2.*

7.5.1 Silica gel

7.5.1.1 Activated silica gel (SiliCycle SiliaFlash®, R10040B, G60, 60–200 µm particle size [70 – 230 mesh], 60 Å pore diameter, or equivalent) - heat for 8 hours at 450 °C and store, under nitrogen, in a stoppered round-bottom flask (Section 6.3.13). Shelf life for activated silica gel is 3 months. The reagent may be used after the expiration date; however, it must be re-baked and a new expiration date assigned.

7.5.1.2 22% H_2SO_4 acidic silica - add H_2SO_4 (concentration equivalent to 22% of final weight of acidified silica) to activated silica (Section 7.5.1.1) and agitate until the mixture is homogenous. Store in a stoppered round-bottom flask. Shelf life is approximately 2 months and must be discarded after this shelf life.

7.5.1.3 44% H_2SO_4 acidic silica - add H_2SO_4 (conc., equivalent to 44% of final weight of acidified silica) to activated silica (Section 7.5.1.1) and agitate until the mixture is homogenous. Stored in a stoppered round-bottom flask. Shelf life is approximately 2 months and must be discarded after this shelf life.

7.5.1.4 28% NaOH basic silica - add 1M NaOH solution (equivalent to 28% of final weight of activated silica) to baked silica (Section 7.5.1.1) and agitate until the mixture is homogenous. Store in a stoppered round-bottom flask. Shelf life is approximately 2 months and the reagent must be discarded after this shelf life.

7.5.2 Florisil® – 2.0-2.1% Deactivated (Supelco® 46382 or US Silica, Pesticide grade, 149-250 µm particle size or 60/100 mesh) - activate by heating at 450 °C for a minimum of 8 hours, cool to room temperature and deactivate with reagent water typically 2% by weight). Store under nitrogen in a stoppered round-bottom flask and allow to equilibrate for 24 hours. Determine the cutpoints prior to use. Shelf life is 3 months from the date of deactivation. The reagent may be used after the expiration date; however, new cutpoints must be redetermined and a new expiration date assigned.

7.5.3 Alumina – 1% Deactivated (Fisher Scientific, A950, Basic Brockman Activity 1, 60–325 mesh) - bake for a minimum of 8 hours at 450°C, cool to room temperature and deactivate with reagent water (typically 1% by weight). Store under nitrogen in a stoppered round-bottom flask and allow to equilibrate for 24 hours. Cutpoints must be determined prior to use. Typical cutpoints are as follows: hexane (10 mL, discard); 1:1 dichloromethane: hexane (45 mL, collect). After deactivation, shelf life is 6 months. The alumina may be used after the expiration date; however, new cutpoints must be determined and a new expiration date assigned.

7.5.4 Bio-Beads® – (Bio-Rad 1522750, S-X3, 200 - 400 mesh, ≤ 2,000 molecular weight limit, or equivalent) - soak in 1:1 dichloromethane:hexane for 24 hours prior to column preparation. Biobead® columns are re-used after cleaning procedures have been carried out. Refer to manufacturer's instructions for the preparation, use, and cleaning of Bio-Bead® columns.

7.5.5 Activated copper – (Fisher Scientific C434-500, Alfa Aesar AA4245530, or equivalent) Either copper foil or powder may be used. Copper must be activated immediately prior to use. Cut copper foil (~200 g) into approximately 1-cm squares or use 200 g of copper powder. To activate, place the quantity of copper needed for sulfur removal in a ground-glass-stoppered Erlenmeyer flask or bottle and cover with methanol. Add HCl (Section 7.1.3) dropwise (0.5 - 1.0 mL) while swirling, until the copper brightens. Pour off the methanol/HCl and rinse 3 times with acetone, then 3 times with hexane. For copper foil, cover with hexane after the final rinse and store in a stoppered flask under nitrogen until used. For the powder, evaporate to dryness on a rotary evaporator and store in a stoppered flask under nitrogen until used.

7.5.6 Potassium silicate - Dissolve 56 g of high purity potassium hydroxide (Aldrich, or equivalent) in 300 mL of methanol in a 750- to 1000-mL flatbottom flask. Add 100 g of activated silica gel (Section 7.5.1.1) plus a stirring bar and stir on an explosion-proof hot plate at 60-70 ºC for 1-2 hours. Decant the liquid and rinse the potassium silicate twice with 100-mL portions of methanol, followed by a single rinse with 100 mL of methylene chloride. Spread the potassium silicate on solvent-rinsed aluminum foil and dry for 2-4 hours in a hood. Activate overnight at 200-250 ºC prior to use. Potassium silicate is used in the packing of the anthropogenic isolation column (Section 12.5.4).

7.6 Reference matrices – Matrices in which the PCBs and interfering compounds are not detected by this method

7.6.1 Reagent water – purified water produced by passing through activated charcoal

7.6.2 High-solids reference matrix – Ottawa or reagent-grade sand

7.6.3 Tissue reference matrix – 10:90 w/w mixture of canola oil and Ottawa sand (simulating a tissue sample with 10% lipids), or clean fish tissue

7.7 Standard solutions – Prepare from materials of known purity and composition or purchase as solutions or mixtures with certification to their purity, concentration, and authenticity. If the chemical purity is 98 % or greater, the weight may be used without correction to calculate the concentration of the standard. Observe the safety precautions in Section 5 and the recommendation in Section 5.1.2.

Note: *Native PCB standards are available from several suppliers. $^{13}C_{12}$-labeled congeners are available from Cambridge Isotope Laboratories and Wellington Laboratories and may be available from other suppliers. Listing of these suppliers does not constitute a recommendation or endorsement for use.*

7.7.1 For preparation of stock solutions from neat materials, dissolve an appropriate amount of assayed reference material in solvent. For example, weigh 10 to 20 mg of an individual congener to three significant figures in a 10-mL volumetric flask and fill to the mark with acetone or other water-miscible solvent. After the compound is completely dissolved, transfer the solution to a clean 15-mL vial with PTFE-lined cap.

7.7.2 When not being used, store standard solutions in the dark at room temperature in screw-capped vials with fluoropolymer-lined caps. Place a mark on the vial at the level of the solution so that solvent loss by evaporation can be detected. Replace the solution if solvent loss has occurred.

7.8 Labeled compound standard solution –The labeled compound standard solution is prepared to have the nominal concentrations of isotopically labeled compounds presented in Table 4 and contains all of the labeled compounds used for isotope dilution and extracted internal standard (EIS) quantitation. A 32-µL aliquot of labeled compound standard solution (equivalent to 40 ng of each labeled compound) is added to each sample QC sample and method blank prior to extraction.

Note: *The labeled compound standard solution should be prepared in acetone for aqueous samples to promote dissolution of the labeled compound. If the solution was prepared in another solvent, then it should be diluted into 1 mL of acetone and then quantitatively transferred to the sample with acetone rinses.*

This spiking solution may be prepared at another concentration and another spiking volume may be employed, as long as the mass of labeled compound spiked into the sample is 40 ng.

7.9 Non-extracted internal standard (NIS) solution –The NIS solutions are prepared to have the nominal concentrations of isotopically labeled compounds presented in Table 4. A 40-µL aliquot of NIS solution is added to the final sample extract and to each calibration standard (if not already included in the calibration mixture purchased from the vendor or if the calibration mixture was prepared from neat materials) just prior to instrumental analysis. The mass of NIS added is designed to match the mass of labeled compound in the final extract.

7.10 Calibration standard solutions – A series of six calibration solutions containing native analytes, labeled compound standards, and labeled NISs is used to establish linearity of the analytical instrument (this is the default calibration procedure). The calibration solutions are prepared to have the nominal concentrations presented in Table 7. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the labeled compound and NISs remain constant. A mid-level calibration solution must be analyzed at least every 12 hours on an ongoing basis for the purpose of calibration verification. A mid-level calibration verification (CV) standard must also be analyzed at the end of the analytical batch but before the end of the 12 hours in order to bracket the analyses.

7.11 Native standard solution – The native spiking solution is prepared to have the nominal concentrations presented in Table 4. An aliquot of native spiking solution is added to all IPR and OPR samples. An aliquot equivalent to 16 ng of each target analyte is added to the sample (200 µL is added if the native standard solution is prepared at the concentrations presented in Table 4).

Note: *This spiking solution may be prepared at another concentration and another spiking volume may be employed as long as the mass of native compound spiked into the sample is 16 ng.*

7.12 Retention time marker solution – A separate calibration standard containing all 209 PCB congeners, as well as isotopically labeled PCBs is used to develop retention time (RT) markers and is analyzed each time a new initial calibration is performed.

7.12.1 Prepare the nine solutions with the congeners listed in Table 5 at 10 µg/mL in isooctane or nonane, or purchase AccuStandard® C-CS-01, C-CS-02, C-CS-03, C-CS-04, C-CS-05, C-CS-06, C-CS-07, C-CS-08, and C-CS-09, or equivalent.

7.12.2 Prepare the retention time marker solution by combining aliquots of the nine solutions (Section 7.12.1), labeled compound standard solution (Section 7.8) and NIS solution (Section 7.9). Typically, this solution is prepared with the native congeners from the individual congener solutions (Table 5) at 10 ng/mL and the labeled compounds and NISs at 400 ng/mL.

7.13 Stability of solutions – Standard solutions used for quantitative purposes (Sections 7.8 through 7.12) should be assayed periodically (e.g., every 6 months) against certified standard reference materials (SRMs) from the National Institute of Science and Technology (NIST), if available, or certified reference materials from a source that will attest to the authenticity and concentration, to assure that the composition and concentrations have not changed more than 5% from the certified value.

8.0 Sample collection, preservation, storage, and holding times

8.1 Collect samples in amber glass containers following conventional sampling practices (Reference 5). Other sample collection techniques, or sample volumes may be used, if documented.

8.2 Aqueous samples

8.2.1 Samples that flow freely are collected as grab samples or in refrigerated bottles using automatic sampling equipment. Collect one liter of sample, or a larger or smaller volume sufficient to meet project needs.

8.2.2 Maintain aqueous samples protected from light (in amber glass containers) at less than 6 ºC (but keep from freezing) from the time of collection until sample preparation at the laboratory.

8.3 Solid, mixed-phase, semi-solid, and oily samples, excluding tissue

8.3.1 Collect samples as grab samples using amber wide-mouth jars. Do not fill jars to the top to prevent glass breakage when sample is frozen at the laboratory.

8.3.2 Maintain solid, semi-solid, oily, and mixed-phase samples protected from light (in amber glass containers) at less than 6 ºC from the time of collection until receipt at the laboratory. Store solid, semi-solid, oily, and mixed-phase samples in amber glass containers at less than -10 ºC. Care must be taken when storing glass jars in the freezer to prevent breakage and leakage of sample.

8.4 Fish and other tissue samples

8.4.1 Fish may be cleaned, filleted, or processed in other ways in the field, such that the laboratory may expect to receive whole fish, fish fillets, or other tissues for analysis.

8.4.2 Collect fish, wrap in aluminum foil, and maintain at less than 6 ºC from the time of collection until receipt at the laboratory, to a maximum time of 24 hours. If a longer transport time is necessary, freeze the sample. Ideally, fish should be frozen upon collection and shipped to the laboratory on dry ice.

8.4.3 Maintain tissue samples protected from light (in amber glass containers) at less than -10 ºC until prepared. Store unused samples in amber glass containers or wrapped in aluminum foil at less than -10 ºC.

8.5 Holding times

8.5.1 There are no demonstrated maximum holding times associated with the PCB congeners in aqueous, solid, semi-solid, tissue, or other sample matrices. If stored in amber glass containers or containers wrapped in aluminum foil at less than 6 ºC, aqueous samples may

be stored for up to one year. Similarly, if stored in the dark at less than -10 ºC, solid, semi-solid, multi-phase, and tissue samples may be stored for up to one year.

8.5.2 Store sample extracts in amber glass containers at less than -20 ºC until analyzed. If stored in amber glass containers at less than -20 ºC, sample extracts may be stored for 1 year.

9.0 Quality Control

9.1 Each laboratory that uses this method is required to operate a formal quality assurance program (Reference 6). The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

If the method is to be applied to a sample matrix other than water (e.g., soils, filter cake, compost, tissue) the most appropriate alternate reference matrix (Sections 7.6.2 - 7.6.3) is substituted for the reagent water matrix (Section 7.6.1) in all performance tests.

9.1.1 The laboratory must make an initial demonstration of the ability to generate acceptable precision and recovery with this method. This demonstration is given in Section 9.2.

9.1.2 In recognition of advances that are occurring in analytical technology, and to overcome matrix interferences, the laboratory is permitted certain options to improve separations or lower the costs of measurements. These options include alternative extraction, concentration, and cleanup procedures, and changes in sample volumes, columns, and detectors. Alternative determinative techniques, such as substitution of spectroscopic or immunoassay techniques for GC/MS technology, and changes that degrade method performance, are *not* allowed without prior review and approval.

Note: *For additional details about the flexibility to make modifications without prior EPA review, see 40 CFR Part 136.6.*

9.1.2.1 Each time a modification is made to this method, the laboratory is required to repeat the procedure in Section 9.2. If calibration will be affected by the change, the instrument must be recalibrated per Section 10.0. Once the modification is demonstrated to produce results equivalent or superior to results produced by this method as written, that modification may be used routinely thereafter, so long as the other requirements in this method are met (e.g., labeled compound recovery).

9.1.2.2 The laboratory is required to maintain records of modifications made to this method. These records include the following, at a minimum:

a) The names, titles, business addresses, and telephone numbers of the analyst(s) that performed the analyses and modification, and of the quality control officer that witnessed and will verify the analyses and modifications.

b) A listing of pollutant(s) measured, by name and CAS Registry number.

c) A narrative stating reason(s) for the modifications (see Section 1.6).

d) Results from all quality control (QC) tests comparing the modified method to this method, including:

i. Calibration (Section 10)
ii. Calibration verification (Section 15.3)
iii. Initial precision and recovery (Section 9.2.1)
iv. Labeled compound recovery (Section 9.3)
v. Analysis of blanks (Section 9.5)
vi. Accuracy assessment (Section 9.4)

e) Data that will allow an independent reviewer to validate each determination by tracing the instrument output (peak height, area, or other signal) to the final result. These data are to include:

i. Sample numbers and other identifiers
ii. Extraction dates
iii. Analysis dates and times
iv. Analysis sequence/run chronology
v. Sample weight or volume (Section 11)
vi. Extract volume prior to each cleanup step (Section 12)
vii. Extract volume after each cleanup step (Section 12)
viii. Final extract volume prior to injection (Section 13)
ix. Injection volume (Section 14.4)
x. Dilution data, differentiating between dilution of a sample or extract (Section 16.4)
xi. Instrument identification
xii. Column (dimensions, liquid phase, solid support, film thickness, etc.)
xiii. Operating conditions (temperatures, temperature program, flow rates)
xiv. Detector (type, operating conditions, etc.)
xv. Chromatograms, printer tapes, and other recordings of raw data
xvi. Quantitation reports, data system outputs, and other data to link the raw data to the results reported

9.1.2.3 Alternative GC columns and column systems – If a column or column system other than those specified in this method is used, that column or column system must meet all the requirements of this method, meaning that it can separate at least as many congeners as the method-specified column and that no additional co-elutions occur.

Note: *The use of alternative GC columns or temperature programs will likely result in a different elution order and different coelutions. These coelutions will need to be tested to determine which higher chlorinated congeners will contribute to the responses of specific lower chlorinated congeners and to provide a correction factor to be applied to the responses of the affected lower chlorinated congeners (see Section 16.3).*

9.1.3 Analyses of method blanks are required on an on-going basis to demonstrate the extent of background contamination in any reagents or equipment used to prepare and analyze field samples (Section 4.3). The procedures and criteria for analysis of a method blank are described in Section 9.5.

9.1.4 The laboratory must spike all samples with labeled compounds to monitor method performance. This test is described in Section 9.3. When results of these spikes indicate

atypical method performance for samples, the samples are diluted to evaluate whether the performance issue is caused by the sample matrix. Procedures for dilution are given in Section 16.4.

9.1.5 The laboratory must, on an ongoing basis, demonstrate through calibration verification and the analysis of the ongoing precision and recovery standard (OPR) and blanks that the analytical system is in control. These procedures are given in Sections 15.1 through 15.8.

9.1.6 The laboratory should maintain records to define the quality of data generated. Development of accuracy statements is described in Section 9.4.

9.2 Initial Demonstration of Capability

9.2.1 Initial precision and recovery (IPR) – To establish the ability to generate acceptable precision and recovery, the laboratory must perform the following operations for each sample matrix type to which the method will be applied by that laboratory.

9.2.1.1 Extract, concentrate, and analyze four aliquots of the matrix type (e.g., aqueous, soil, sediment, biosolids, tissue) to be tested (Section 7.6.1 through 7.6.3), spiked with 200 µL of the native standard solution (Section 7.11) and 32 µL of the labeled compound standard solution (Section 7.8). At least one method blank of the matrix being analyzed, must be included. All sample processing steps that are to be used for processing samples, including preparation and extraction (Sections 11.2 – 11.4), cleanup (Section 12.0) and concentration (Section 13.0), must be included in this test.

9.2.1.2 Using results of the set of four analyses, compute the average percent recovery (R) of the extracts and the relative standard deviation (RSD) of the concentration for each compound, by isotope dilution for PCBs with a labeled analog, and by non-extracted internal standard (NIS) for PCBs without a labeled analog and for the labeled analogs.

9.2.1.3 For each native and labeled compound, compare RSD and % recovery with the corresponding limits for initial precision and recovery in Table 8 and Table 9. If RSD and R for all compounds meet the acceptance criteria, system performance is acceptable, and analysis of blanks and samples may begin. If, however, any individual RSD exceeds the precision limit or any individual R falls outside the range for recovery, system performance is unacceptable for that compound. Correct the problem and repeat the test (Section 9.2).

9.2.2 Method detection limit (MDL) – The laboratory must establish MDLs for the analytes of interest using the MDL procedure at 40 CFR Part 136, Appendix B (Reference 12). An MDL must be determined for each of the 209 congeners. Co-eluting congeners (at the same LOC) are treated as a single analyte and the same MDL is applied to all congeners that co-elute together (i.e., one MDL is determined for PCB-5/8, one for PCB 4/10, etc.). The minimum level of quantification (ML) is then calculated using the approach described in Section 23 or from another recognized source. MDLs and MLs determined in a given laboratory may differ but should generally be in the range of those in Table 12.

9.3 To assess method performance on the sample matrix, the laboratory must spike all samples with the labeled compound standard solution (Section 7.8) and all sample extracts with the NIS spiking solution (Section 7.9).

9.3.1 Analyze each sample according to the procedures in Sections 11.0 through 17.0.

9.3.2 Compute the percent recovery of the labeled compound using the non-extracted internal standard method (Section 16.2) and the equation in Section 15.5.2.

9.3.3 The recovery of each labeled compound must be within the limits in Table 10 (see Note 1 below the table). Given that there are 29 labeled compounds being tested simultaneously, some failures of individual labeled compounds are not unexpected. If the recoveries of more than three compounds fall outside of these limits, additional cleanup procedures must be employed to attempt to bring the recoveries within the expected ranges. None of the three or fewer failed compounds may have a recovery less than 5%. If the recoveries cannot be brought within the expected ranges after all cleanup procedures have been employed, water samples are diluted, and smaller amounts of soils, sludges, sediments, and other matrices are analyzed, per Section 16.4.

9.4 Recovery of labeled compounds from samples must also be assessed and records maintained.

9.4.1 After the analysis of 30 samples of a given matrix type (water, soil, biosolids, tissues, etc.) for which the labeled compounds pass the tests in Section 9.3, compute the average percent recovery (R) and the standard deviation of the percent recovery (S_R) for the labeled compounds only. Express the assessment as a percent recovery interval from $R - 2S_R$ to $R + 2S_R$ for each matrix. For example, if $R = 90\%$ and $S_R = 10\%$ for five analyses of soil, the recovery interval would be expressed as 70 to 110%.

9.4.2 Update the accuracy assessment for each labeled compound in each matrix on a regular basis.

9.4.3 Examine the frequencies at which labeled compounds fail to determine if there are consistent patterns among the congeners that may indicate sample processing procedures that may need to be revised. For example if the failures are more common for the congeners in the lowest two homologs, it may be an issue of extract concentration, whereas failures in other homologs could be related to cutpoints for specific cleanup steps or other procedures.

9.5 Method blanks – A reference matrix method blank is analyzed with each sample batch (Section 4.3) to demonstrate freedom from contamination. The matrix for the method blank must be similar to the sample matrix for the batch, e.g., a 1-L reagent water blank (Section 7.6.1), a high-solids reference matrix blank (Section 7.6.2), or a tissue blank (Section 7.6.3).

9.5.1 Analyze the extract (Sections 11.2.1.4, 11.2.2.3, 11.2.3.3, 11.2.4.3, 11.3.3, and 11.4.5) of the method blank aliquot after the analysis of the OPR (Section 15.5) and solvent blank (Section 15.7).

9.5.2 If any PCB is found in the blank at 1) at a concentration greater than the ML for the analyte, 2) at a concentration greater than one-third the regulatory compliance limit, or 3) at a concentration greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, analysis of samples must be halted, and the problem corrected. (The laboratory may adopt more stringent acceptance limits for the method blank, at their discretion.) If the contamination is traceable to the extraction batch, samples affected by the blank must be re-extracted and the extracts re-analyzed. If, however, continued re-testing results in repeated blank contamination, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with blank

contamination for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.

9.6 The specifications contained in this method can be met if the apparatus used is calibrated properly and then maintained in a calibrated state. The standards used for initial calibration (Section 10.5), calibration verification (Section 15.3), and for initial (Section 9.2.1) and ongoing (Section 15.5) precision and recovery should be prepared from the same source, so that the most precise results will be obtained. A GC/MS instrument will provide the most reproducible results if dedicated to the settings and conditions required for determination of PCBs by this method.

9.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when the internal standard method is used.

9.8 Matrix spikes generally are **not** required for isotope dilution methods because any deleterious effects of the matrix should be evident in the recoveries of the labeled compounds spiked into every sample. However, because many of the congeners are quantified by a non-analogous labeled compound (e.g., PCB-18 is quantified by ^{13}C-PCB-28), the analysis of matrix spike samples may help diagnose matrix interferences for specific congeners. If the laboratory chooses to perform matrix spikes, they may use recoveries of 25 – 150% as interim acceptance limits for all matrices, based on the performance exhibited during the multi-laboratory validation study (Reference 13).

10.0 Calibration and Standardization

The processes of calibration and standardization for this method differ from those in full-scan GC/MS methods. For the purpose of this method, "calibration" refers to multiple aspects of the procedure, including:

- Establishing the GC/MS operating conditions
- Adjusting the mass resolution of the detector system to achieve unit mass resolution for a reference compound (called "tuning" here)
- Establishing mass accuracy of the peak maximum of (± 0.3 Daltons or atomic mass units) for PFTBA or other manufacturer-recommended reference compound (called "mass accuracy calibration" here)
- Establishing accurate retention times, relative retention times (RRTs), and retention time windows for all 209 PCB congeners (called "retention time calibration" here)
- Establishing the response ratios (RRs) response factors (RFs) for all the target analytes and labeled compounds through the analyses of at least 6 solutions (called "initial calibration" here)

Collectively, these processes are performed at the time that the instrument is initially set up and then verified periodically during routine analyses (see Section 10.6). The process begins with establishing the instrument operating conditions.

10.1 Establishing GC/MS operating conditions

The GC and MS operating conditions may be optimized for compound separation and sensitivity. The same optimized operating conditions must be used for the analysis of all standards, blanks, IPR and OPR standards, and samples. The conditions below are a suggested starting point and may need to be adjusted for the specific GC column and GC/MS system employed.

GC Temperature Program		**General GC Conditions**	
Initial Temperature (°C)	50	Injector Temperature (°C)	280
Hold time (min)	0.95	Injector	Splitless, 2 min
Rate (°C min^{-1})	13.3	Carrier Gas	Helium
Temperature (°C)	150	Maximum Temperature (°C)	325
Hold time (min)	0	Flow Rate	1.0 mL/min
Rate (°C min^{-1})	3.5		
Temperature (°C)	250		
Hold time (min)	0	**MS Conditions**	
Rate (°C min^{-1})	57.8	Source Temperature (°C)	286
Temperature (°C)	320	Quadrupole Temperature (°C)	150
Hold time (min)	9.5	Electron Energy (eV)	70
Total Run time (min)	48.0	Mass Resolution	Unit

10.2 Mass Spectrometer (MS) Resolution (Tuning)

Prior to the analysis of any samples, including IPR/OPR, blanks, or calibration standards, the MS system must be tuned using a suitable calibrant such as perfluorotributylamine (PFTBA) or an alternative calibrant recommended by the manufacturer. Tuning involves checking the mass resolution, and the mass peak relative responses at the time that the instrument is first set up and periodically during routine analyses. Refer to the manufacturer's instructions for additional specifics.

10.2.1 After the instrument operating conditions have been established and optimized in Section 10.1, perform a manual tune using PFTBA, or use the manufacturer's "auto tune" procedure as needed to optimize the mass spectrometer.

10.2.2 Check the instrument mass resolution to ensure that it achieves at least unit resolution. When using PFTBA, unit resolution is demonstrated when a signal at *m/z* 70 is visible adjacent to *m/z* 69, *m/z* 220 is visible as a distinct peak adjacent to *m/z* 219, and *m/z* 503 is separated from *m/z* 502 with a valley of no higher than 80% (with respect to *m/z* 503). If using an alternative reference calibrant, select characteristic *m/z* across the calibrated mass range and evaluate their responses to ensure unit resolution.

10.2.3 When using PFTBA, if distinct signals are not visible for *m/z* 70 and 69, and *m/z* 220 and 219, or the valley between the signals for *m/z* 503 and 502 is greater than 80% (Section 10.2.2), then adjust the MS operating conditions and perform another tune (Section 10.2.1) and evaluate the new responses. If using an alternative reference calibrant, evaluate the responses of those characteristic *m/z* selected in Section 10.2.2 in a similar manner. If the requirements cannot be met again, more significant adjustments may be required. Consult the manufacturer's instructions.

10.2.4 Using PFTBA, determine the relative abundances by dividing the abundance of *m/z* 219 and *m/z* 502 by the abundance of the *m/z* 69 peak. Perform similar calculations for the characteristic *m/zs* when using an alternative reference calibrant.

10.3 Mass accuracy calibration

Mass accuracy calibration must be performed when the instrument is initially set up, using PFTBA or another reference calibrant. Using the results from PFTBA, verify the mass calibration by measuring the amount of peak drift from the expected masses for *m/zs* 69, 219, and 502. If the peak

apex has shifted more than approximately 0.3 Dalton (atomic mass unit), then the mass calibration will need to be adjusted. If using an alternative reference calibrant, measure the peak drift from the expected masses for the characteristic *m/zs* for that calibrant.

10.4 Retention time calibration for the PCB congeners

10.4.1 Separately inject each of the diluted individual congener solutions (Section 7.12). All native congeners for which there is a labeled analog should elute slightly before or simultaneously with the labeled analog. If not, then the laboratory should check to make sure the native and labeled congeners were assigned correctly. Store the retention time (RT) and relative retention time (RRT) for each congener in the data system. The RRT is calculated by dividing the native congener RT by the labeled compound RT.

Calculate an RRT window using RTs for the native congeners that are ± 3 seconds (equivalent to ± 0.05 min) from the stored RT (e.g., use the two extremes for each RT). For example, if the stored RT for a native congener was 26.30 min and the RT of the labeled compound was 26.29 min, the RRT rounds to 1.000. The RRT window is calculated by dividing both 26.25 (for the low limit) and 26.35 (for the high limit) by 26.29. In this scenario, the RRT window would be 0.9985 - 1.002.

10.4.2 Inject the diluted combined 209 congener solution (Section 7.12). If necessary, adjust the chromatographic conditions and scan descriptors until the RRTs for all congeners are approximately within the windows established in 10.4.1.

10.4.3 After the column performance tests are passed (Section 10.4.2), calculate and store the RTs and RRTs for the resolved congeners and the RTs and RRTs for the congeners that co-elute.

10.4.4 GC resolution check

Based on the chromatographic conditions in Section 10.1 and the example retention times in Table 2, PCBs 28 and 31 are the two native congeners in the same homolog that elute closest together. If they are not completely resolved, then the height of the valley (the space between the two peaks, measured from the baseline to the low point of the valley) must be less than 80% of the height of the smaller of either PCB 28 or 31 (see Figure 1). If this criterion is not achieved, then adjust chromatographic conditions accordingly. Regardless of the separation achieved for PCBs 28 and 31, also examine the retention times of all other congeners that should be resolved (according to Table 2) to ensure that they are adequately separated (Figure 2).

10.5 Initial multi-point calibration of target analytes

Prior to the analysis of samples, and after the tune and mass accuracy calibration have met all criteria in Sections 10.2 and 10.3, the GC/MS system must be calibrated at a minimum of 6 standard concentrations (Section 7.10 and Table 7) that encompass the working concentration range of the instrument. Those initial calibration solutions contain target compounds, the suite of labeled compounds used for isotope dilution quantitation, those used for extracted internal standard (EIS) quantitation, and the NISs listed in Table 7.

Note: *Six calibration standards are the minimum number that should be used in the initial calibration; however, the laboratory may use more standards, as long as the criteria in Section 10.5.3.3 can be met.*

This method procedure calibrates and quantifies 65 target PCB congeners by one of three approaches (Table 6):

- True isotope dilution quantification (tID), whereby the response of the target congener is compared to the response of its $^{13}C_{12}$-labeled analog. Twenty-three target congeners are quantified in this way.

- Modified isotope dilution (mID), when one or more congeners in the same LOC coelute with a congener that has a $^{13}C_{12}$-labeled analog. Fourteen target congeners are quantified in this way (6 with $^{13}C_{12}$-labeled analogs and 8 that coelute with one of those 6).

- Extracted internal standard quantification (EIS), whereby the response of the target congener (or one or more congeners in the sample LOC coelute) is compared to the response of the $^{13}C_{12}$-labeled analog of another congener in the same LOC with which it coelutes. Twenty-eight target congeners are quantified in this way.

The remaining 144 congeners are quantified indirectly using isotope dilution standards of similar congeners with the same level of chlorination. The response factor is assumed to be the same as the reference isotope dilution standard. This approach may produce greater uncertainty in the results for these congeners than using any of the three approaches described above but calibrating all of the congeners would make the level of effort more burdensome for the laboratories that are the intended users of this procedure. These congeners were seen less often and/or at lower concentrations in the environmental databases surveyed and the original Aroclor formulations.

Note: *If any of these congeners is a priority to a specific data user, the laboratory is encouraged to calibrate additional congeners using any of the three approaches listed above. This is allowed under the flexibility of the method.*

SIM analysis is required to achieve better sensitivity than full-scan analysis. The two ions (called Q1 and Q2) to be monitored for each native congener, labeled compound used for isotope dilution quantitation or for extracted internal standard (EIS) quantitation, and NIS compound are listed in Table 2. The ratio of the two ions (Q1/Q2) is an important aspect of identifying each target analyte, as described in Section 16.1.4. However, only the Q1 for each analyte is used in the calculations of the response ratios and response factors in Section 10.5.3.2. and the determination of analyte concentrations in Section 16.2.

10.5.1 Initial calibration frequency

The GC/MS system must be calibrated when it is initially set up and whenever the laboratory takes corrective action that might change or affect the initial calibration criteria, or if the CV technical acceptance criteria have not been met.

10.5.2 Initial calibration procedure

Prepare calibration standards containing the native compounds, all the labeled compounds, and the NISs at the concentrations described in Table 7. Analyze each calibration standard by injecting 1.0 or 2.0 µL.

Note: *The same injection volume must be used for all standards, samples, blanks, and QC tests.*

10.5.3 Initial calibration calculations

10.5.3.1 Instrument sensitivity

Sufficient instrument sensitivity is established if a signal-to-noise ratio ≥ 3:1 for PCB-118 can be achieved when analyzing the 10 ng/mL CS1 standard (Table 7). The signal-to-noise ratios of other congeners may be checked, but if PCB-118 meets this criterion, the sensitivity of the instrument is acceptable.

10.5.3.2 Response Ratios (RR) and Response Factors (RF)

The response ratio (RR) for each congener calibrated by isotope dilution is calculated according to the equation below, separately for each of the calibration standards, using the areas of the quantitation ions (Q1) with the *m/z* shown in Table 2. RR is used for the 23 congeners quantified by true isotope dilution and the 14 congeners quantified by modified isotope dilution.

$$RR = \frac{Area_n\ C_l}{Area_l\ C_n}$$

where:

$Area_n$ = The measured area of the Q1 *m/z* for the native (unlabeled) PCB

$Area_l$ = The measured area at the Q1 *m/z* for the labeled PCB used for isotope dilution quantitation

C_l = The concentration of the labeled compound used for isotope dilution quantitation in the calibration standard (ng)

C_n = The concentration of the native compound in the calibration standard

Similarly, the response factor (RF) for each unlabeled congener calibrated by extracted internal standard is calculated according to the equation below. RF is used for the 28 congeners quantified by extracted internal standard.

$$RF = \frac{Area_s\ C_{eis}}{Area_{eis}\ C_n}$$

where:

$Area_s$ = The measured area of the Q1 *m/z* for the target (unlabeled) PCB

$Area_{eis}$ = The measured area at the Q1 *m/z* for the labeled PCB used as the extracted internal standard (EIS)

C_{eis} = The concentration of the labeled PCB used as the extracted internal standard (EIS) in the calibration standard (ng)

C_n = The concentration of the target (unlabeled) PCB in the calibration standard (ng)

A response factor (RF_s) is calculated for each labeled compound in the calibration standard using the equation below. RF_s is used for the 29 labeled compounds quantified by non-extracted internal standard.

$$RF_s = \frac{Area_l\ C_{nis}}{Area_{nis}\ C_l}$$

where:

$Area_l$ = The measured area of the Q1 *m/z* for the labeled PCB standard added to the sample before extraction

$Area_{nis}$ = The measured area at the Q1 *m/z* for the labeled PCB used as the non-extracted internal standard (NIS)

C_{nis} = The concentration of the labeled compound used as the non-extracted internal standard (NIS) in the calibration standard (ng)

C_l = The concentration of the labeled PCB standard added to the sample before extraction (ng)

10.5.3.3 Instrument Linearity

Calculate the relative standard deviation (RSD) or the relative standard error (RSE) of the RR or RF values of the six initial calibration standards for each native congener and labeled compound. The RSD or RSE must be ≤ 20% to establish instrument linearity. Alternatively, employ a linear or quadratic regression for the calibration, based on the manufacturer's instructions, and use the RSE as the calibration metric. If using a quadratic regression, a calibration standard at a seventh concentration must be used to maintain sufficient degrees of freedom.

10.5.3.4 Establishing mean responses for non-extracted internal standards

Calculate the mean area of the response for each of the three non-extracted internal standards in each of the initial calibration standards. Store the mean responses for use later in tracking the overall sensitivity of the GC/MS system (see Sections 10.6.4, 15.3.4, and 15.9). There are no QC acceptance criteria associated with these mean responses or their variability in the initial calibration itself.

10.5.4 Initial calibration corrective actions

If the instrument sensitivity or the instrument linearity criteria for initial calibration are not met, inspect the system for problems and take corrective actions to achieve the criteria. This may require the preparation and analysis of fresh calibration standards. All initial calibration criteria must be met before any samples or required blanks are analyzed.

10.6 Verification of MS tuning, mass calibration, retention times, and calibration of target analytes

During routine operation, the various calibrations described in Sections 10.2 to 10.5 must be verified at the frequencies described below.

10.6.1 Tuning verification

Unlike the full-scan GC/MS instruments used in early EPA GC/MS methods, modern GC/MS systems are considerably more stable. As a result, the tune with PFTBA or an alternative calibrant does not require daily verification. The tune should be checked by performing the procedure in Section 10.2:

- At least once a week during periods when samples are being analyzed
- Any time that mass accuracy cannot be verified (see Section 10.6.2)
- After any major instrument maintenance activities

10.6.2 Mass accuracy and resolution verification

Mass accuracy and resolution calibration must be verified at the beginning of each 12-hour period during which samples or standards are analyzed, using PFTBA or another reference calibrant, using the same procedure described in Section 10.3. If the peak drift is greater

than 0.3 Da, then corrective action is required. When using PFTBA, if the relative abundance of *m/z* 219 and *m/z* 502 against *m/z* 69 is not within 50 – 150% when compared to the reference tune (Section 10.2.4), or the valley between *m/z* 502 and *m/z* 503 is greater than 80%, corrective action is required. If using an alternative reference calibrant, verify the mass accuracy and resolution using the expected masses for the characteristic *m/z* for that calibrant.

10.6.3 Retention time verification for all 209 PCB congeners

Retention times for all 209 PCB congeners do not require periodic verification. However, any time that the GC column is replaced, or major instrument maintenance is performed, the retention times of all 209 congeners must be determined again using the procedures in Section 10.4.

10.6.4 Calibration verification

Except during analyses immediately after a multi-point initial calibration (Section 10.5), the calibration is verified every 12 hours through the analysis of a mid-level calibration solution. Calibration verification uses the mean RRs or RFs determined from the initial calibration to calculate the analyte concentrations in the verification standard. See Sections 14.5 and 15.3.

In addition, the GC resolution of PCBs 28 and 31 must be checked every 12 hours during the analysis of the mid-level calibration verification solution, using the same procedure described in Section 10.4.4. If the requirements in Section 10.4.4 cannot be met, then corrective action is required.

In order to track potential changes in instrument sensitivity over time, compare the area responses of each of the three non-extracted internal standards in the calibration verification to their mean responses in the most recent initial calibration (see Section 10.6.4). Calculate the percent difference (%D) as follows, using the mean area from the initial calibration as the "true value":

$$\%D = \frac{CALVER\ Area_{NIS} - Mean\ ICAL\ Area_{NIS}}{Mean\ ICAL\ Area_{NIS}}\ x\ 100$$

The %D for each non-extracted internal standard in the calibration verification must meet the requirements in Section 15.3.3 (within the range of -50% to +100% of their mean responses in the most recent initial calibration).

11.0 Sample preparation and extraction

This section describes the sample preparation procedures for aqueous samples (Section 11.2), solid (soil, biosolids, or sediment) samples (Section 11.3) and tissue samples (Section 11.4). Section 12 describes the extract cleanup options for each sample type. For aqueous samples (or samples that are mostly aqueous) that contain particles, percent solids are determined using the procedures in Section 11.1. Because PCBs may be bound to suspended particles, the preparation of aqueous samples is dependent on the solids content of the sample. Aqueous samples containing one percent solids or less are prepared and extracted per one of the three procedures in Section 11.2.1 – 11.2.3. For aqueous samples containing greater than one percent solids, a sample aliquot sufficient to provide 10 g of dry solids is used, as described in Section 11.2.4.

11.1 Determination of percent solids

11.1.1 Determination of percent suspended solids – Aqueous liquids and multi-phase samples consisting of mainly an aqueous phase

11.1.1.1 Desiccate and weigh a glass fiber filter (Section 6.6.3.1) to three significant figures.

11.1.1.2 Filter 10.0 ± 0.02 mL (Section 6.8.2.2) of well-mixed sample through the filter.

11.1.1.3 Dry the filter a minimum of 12 hours at 110 ± 5 ºC and cool in a desiccator.

11.1.1.4 Calculate percent solids as follows:

$$\%\ solids = \frac{weight\ of\ sample\ aliquot\ after\ drying\ (g) - weight\ of\ filter\ (g)}{10\ g}\ x\ 100$$

11.1.2 Non-aqueous liquids, solids, semi-solid samples, and multi-phase samples in which the main phase is not aqueous (does not include tissues)

11.1.2.1 Weigh 5 to 10 g of sample to three significant figures in a tared beaker.

11.1.2.2 Dry a minimum of 12 hours at 110 ± 5 ºC, and cool in a desiccator.

11.1.2.3 Calculate percent solids as follows:

$$\%\ solids = \frac{weight\ of\ sample\ aliquot\ after\ drying\ (g)}{weight\ of\ sample\ aliquot\ before\ drying\ (g)}\ x\ 100$$

11.2 Water sample preparation and extraction

Aqueous samples containing one percent suspended solids or less and QC aliquots are prepared and extracted using one of the following extraction techniques: separatory funnel extraction (Section 11.2.1), automated cartridge-based SPE (Section 11.2.2), or automated disk-based SPE (Section 11.2.3). These extraction procedures were found to provide comparable results in a single-laboratory study. In addition, a procedure is provided to separate particulates for aqueous samples containing > 1% solids in Section 11.2.4. The particulates from those samples can then be prepared using the solid extraction procedures (Section 11.3) and the extracts of the two components combined before cleanup. Other extraction procedures for aqueous samples, such as continuous liquid-liquid extraction, may be used, provided that the requirements in Section 9 of this method have been met.

11.2.1 Separatory funnel extraction of aqueous samples ≤ 1% solids

11.2.1.1 Weigh the sample bottle with its cap, on a top loading balance to ± 1 g. After extraction, re-weigh the sample bottle and convert the weight to volume assuming a density of 1.00 g/mL.

11.2.1.2 Spike 32 µL of the labeled compound spiking solution (Section 7.8) into the sample bottle. (Other volumes and spiking solution concentrations may be employed as long as the mass of labeled compound spiked into the sample equals the mass contained in the calibration standards.) Cap the bottle and mix the

sample by careful shaking for 30 seconds. Allow the sample to equilibrate for 30 minutes, with occasional shaking.

11.2.1.3 For each sample or sample batch (to a maximum of 20 samples) to be extracted during the same 12-hour shift, place two 1.0-L aliquots of reagent water in clean sample bottles or flasks. Spike 32 µL of the labeled compound standard solution (Section 7.8) into both reagent water aliquots as described in Section 11.2.1.2. One of these aliquots will serve as the method blank (Section 9.5). Spike 200 µL of the native standard solution (Section 7.11) into the other reagent water aliquot. This aliquot will serve as the OPR (Section 15.5). Cap the bottle and mix the sample by careful shaking for 30 seconds. Allow the samples to equilibrate for 30 minutes, with occasional shaking.

11.2.1.4 Pour the spiked sample into a 2-L separatory funnel. Rinse the bottle or flask twice with 5 mL of reagent water and add these rinses to the separatory funnel. Add 100 mL of dichloromethane to the empty sample bottle. Seal the bottle and shake 60 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking vigorously for two minutes. Allow the mixture to equilibrate for at least 10 minutes. Collect the dichloromethane layer in an Erlenmeyer flask.

11.2.1.5 Extract the water sample two more times with 100-mL portions of dichloromethane. Combine the dichloromethane layers. Discard the aqueous layer.

Note: *If an emulsion forms and is more than one-third the volume of the solvent layer, employ one of the following mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, use of phase separation paper, centrifugation, use of an ultrasonic bath with ice, addition of NaCl, or other physical methods. Alternatively, solid-phase cartridge (Section 11.2.2), disk-based (Section 11.2.3), or other extraction technique may be used to prevent emulsion formation. Any alternative technique is acceptable so long as the requirements in Section 9.2 are met.*

11.2.1.6 Dry the extract by filtration into a round-bottom flask through a solvent-rinsed glass funnel with a glass wool plug and approximately half full of granular anhydrous sodium sulfate (Section 7.2.1). Rinse the anhydrous sodium sulfate with dichloromethane to ensure quantitative transfer.

11.2.1.7 Concentrate the extract by rotary evaporation (water bath < 30 °C) to 1-2 mL (Section 13.1). Transfer the extract to a centrifuge tube (Section 6.7.3) with hexane rinses. Concentrate the extract to 300 µL under a gentle stream of nitrogen (Section 13.2). Make the volume to 1 mL with hexane. (Other concentration equipment may be used, provided that the requirements in Section 9 of this method have been met.) Proceed to Section 12.0 for extract cleanup.

11.2.2 Aqueous sample extraction using automated cartridge-based extractor (samples ≤1% solids)

Note 1: *For aqueous samples with > 1% solids, the supernatant portion of sample can be extracted by this procedure once the particulate portion has been separated as described in Section 11.2.4.*

Note 2: *Consult manufacturer's manual for instructions that are specific to the brand and model of the automated cartridge SPE system being used.*

This extraction procedure is suitable for aqueous samples with ≤ 1% solids, including drinking water, non-potable water, effluents, and aqueous sludge. The typical sample size is 1 L. Larger sample sizes will require additional parts.

11.2.2.1 Weigh the sample bottle with its cap, on a one-place top loader balance to ± 1 g. After extraction, re-weigh the sample bottle and convert the weight to volume assuming a density of 1.00 g/mL.

11.2.2.2 Spike the sample with 32 µL of labeled compound standard solution (Section 7.8). (Other volumes and spiking solution concentrations may be employed as long as the mass of labeled compound spiked into the sample equals the mass contained in the calibration standards.) Cap the bottle and mix the sample by careful shaking for 30 seconds. Allow the sample to equilibrate for 30 minutes, with occasional shaking.

11.2.2.3 For each sample or sample batch (to a maximum of 20 samples) to be extracted during the same 12-hour shift, place two 1.0-L aliquots of reagent water in clean sample bottles or flasks. One of these aliquots will serve as the method blank (Section 9.5). Spike 32 µL of the labeled compound standard solution (Section 7.8) into both reagent water aliquots. Spike 200 µL of the native standard solution (Section 7.11) into reagent water aliquot. This aliquot will serve as the OPR (Section 15.5). Shake the sample for 30 seconds and allow to equilibrate for 30 minutes.

11.2.2.4 Acidify the sample with 25% or concentrated hydrochloric acid (Section 7.1.3) to a pH of 2. Do not let the sample sit more than 30 minutes before extraction.

11.2.2.5 Prepare the SPE system following the manufacturer's instructions. Rinse the sample bottle lid twice with acetone (each about 1 mL) and add it into the sample. Put some glass wool into the DVB cartridge (Section 6.4.6.3). More glass wool may be needed for dirtier samples; however, do not over compact the glass wool. Attach the sample bottle and cartridge to the extractor.

11.2.2.6 Follow the manufacturer's instructions to start the extraction. The pre-wet, extraction, rinse, and elution steps are to be performed according to the manufacturer's instructions.

11.2.2.7 The final extract contains 25 mL hexane as bottle rinse, 15 mL of hexane as additional elution, and 1-2 mL of water. Quantitatively transfer the extract to a 250-mL Erlenmeyer flask with hexane and dry over anhydrous sodium sulfate (Section 7.2.1) for at least 30 minutes. Transfer the dried extract into a 250-mL round-bottom flask.

11.2.2.8 Concentrate the extract by rotary evaporation (water bath <30 °C) to 1-2 mL (Section 13.1). Transfer the extract to a centrifuge tube (Section 6.7.3) with hexane rinses. Concentrate the extract to 300 µL under a gentle stream of nitrogen (Section 13.2). Make the volume to 1 mL with hexane. (Other concentration equipment may be used, provided that the requirements in Section 9 of this method have been met.) Proceed to Section 12.0 for extract cleanup.

11.2.3 Aqueous sample extraction using automated disk-based extractor (samples ≤ 1% solids)

This extraction procedure is suitable for aqueous samples with ≤ 1% solids including drinking water, non-potable water, effluents, and aqueous sludge. The typical sample size is 1 L.

Note 1: *For aqueous samples with > 1% solids, the supernatant portion of sample can be extracted by this procedure once the particulate portion has been separated as described in Section 11.2.4.*

Note 2: *Consult manufacturer's manual for instructions specific to the brand and model of the automated disk SPE system being used.*

11.2.3.1 Weigh the sample bottle with the cap, on a one-place top loader balance to ± 1 g. After extraction, re-weigh the sample bottle and convert the weight to volume assuming a density of 1.00 g/mL.

11.2.3.2 Spike the sample with 32 µL of labeled compound standard solution (Section 7.8). (Other volumes and spiking solution concentrations may be employed as long as the mass of labeled compound spiked into the sample equals the mass contained in the calibration standards.) Cap the bottle and mix the sample by careful shaking for 30 seconds. Allow the sample to equilibrate for 30 minutes, with occasional shaking.

11.2.3.3 For each sample or sample batch (to a maximum of 20 samples) to be extracted during the same 12-hour shift, place two 1.0-L aliquots of reagent water in clean sample bottles or flasks. Spike 32 µL of the labeled compound standard solution (Section 7.8) into both reagent water aliquots. One of these aliquots will serve as the method blank (Section 9.5). Spike 200 µL of the native standard solution (Section 7.11) into one reagent water aliquot. This aliquot will serve as the OPR (Section 15.5). Shake the sample for 30 seconds and allow to equilibrate for 30 minutes.

11.2.3.4 Acidify the sample with 25% or concentrated hydrochloric acid (Section 7.1.3) to a pH of 2. Do not let the sample sit more than 30 minutes before extraction.

11.2.3.5 Follow the manufacturer's instructions for system operation and monitor all pressure setpoints.

11.2.3.6 Rinse the sample bottle cap twice with acetone (each about 1 mL) and add it into the sample. Load the bottle to the extractor and install the appropriate DVB extraction disk (Section 6.4.7.2) and filters (1 µm, 5 µm, or both from Section 6.4.7.5) into the SPE disk holder assembly. Seat the disk holder onto the extractor platform. Follow the manufacturer's instructions to load and run the method. The pre-wet, extraction, rinse, and elution steps are to be performed according to the manufacturer's instructions.

11.2.3.7 The final extract collected contains ~20 mL acetone, ~90 mL of hexane and ~10 mL of water. Quantitively transfer the extract to a 500-mL Erlenmeyer flask with hexane. Add ~50 mL of hexane and dry over anhydrous sodium sulfate (Section 7.2.1) for at least 30 minutes. Transfer the dried extract to a 250-mL round-bottom flask.

11.2.3.8 Concentrate the extract by rotary evaporation (water bath < 30 °C) to ~20 mL. Add ~ 50 mL hexane and dry the extract over anhydrous sodium sulfate. Concentrate the extract by rotary evaporation (water bath < 30 °C) to 1-2 mL (Section 13.1). Transfer the extract to a centrifuge tube (Section 6.7.3) with hexane rinses and add ~ 100 µL (10 drops) of toluene or nonane. Concentrate the extract to 50 µL under a gentle stream of nitrogen (Section 13.2). Make the volume to 1 mL with hexane. (Other concentration equipment may be used, provided that the requirements in Section 9 of this method have been met.) Proceed to Section 12.0 for extract cleanup.

11.2.4 Preparation of aqueous samples containing > 1% solids

11.2.4.1 Weigh the sample bottle with lid on a one-place top loader balance to ± 1 g. After extraction, re-weigh the sample bottle and convert the weight to volume assuming a density of 1.00 g/mL.

11.2.4.2 Spike the sample with 32 µL of labeled compound standard solution (Section 7.8). (Other volumes and spiking solution concentrations may be employed as long as the mass of labeled compound spiked into the sample equals the mass contained in the calibration standards.) Cap the bottle and mix the sample by careful shaking for 30 seconds. Allow the sample to equilibrate for 30 minutes, with occasional shaking.

11.2.4.3 For each sample or sample batch (to a maximum of 20 samples) to be extracted during the same 12-hour shift, place two 1.0-L aliquots of reagent water in clean sample bottles or flasks. Spike 32 µL of the labeled compound standard solution (Section 7.8) into both reference matrix aliquots as described in Section 11.2.4.2. One of these aliquots will serve as the method blank (Section 9.5). Spike 200 µL of the native standard solution (Section 7.11) into one reference matrix aliquot as described in Section 11.2.4.2. This aliquot will serve as the OPR (Section 15.5). Extract, cleanup, and concentrate both aliquots as described in Sections 11.2.4.4 - 11.2.4.7, 12.0 and 13.0.

11.2.4.4 Transfer a portion of the sample to a clean 500-mL glass jar with a clean solvent-rinsed 102-mm filter paper (Section 6.6.3.2) in the bottom of the jar. Spin the sample in the centrifuge at 1500 rpm until the solids have settled. Decant the supernatant into a separatory funnel. Repeat the procedure until the entire sample has been centrifuged, leaving the filter paper in place throughout the process.

11.2.4.5 Once the entire sample has been processed, dry the particulate and filter in the jar by mixing them with anhydrous sodium sulfate (Section 7.2.1). Transfer the filter paper/solids to a Soxhlet thimble and extract the sample (Section 11.3.2-11.3.4) but do **not** add additional labeled compound standard solution. After extraction, proceed with combining the extracts of the two fractions, as described in Section 11.2.4.7.

11.2.4.6 Rinse the empty jar with reagent water and dichloromethane with 1-2 mL of each solvent. Add the rinses to the separatory funnel/Erlenmeyer flask containing the supernatant. Extract the supernatant by separatory funnel (as described in Sections 11.2.1.4 – 11.2.1.6), automated cartridge-based SPE (as described in Sections 11.2.2.4 – 11.2.2.7) or automated disk-based SPE (as described in Sections 11.2.3.4 – 11.2.3.7). After extraction, proceed with combining the extracts of the two fractions, as described in Section 11.2.4.7.

11.2.4.7 Quantitatively transfer the filter paper/solid extract (Section 11.3.5) with dichloromethane and combine it with the supernatant extract (Section 11.2.4.6). Concentrate the combined extract by rotary evaporation to 1 - 2 mL (Section 13.1). Transfer the combined extract to a centrifuge tube (Section 6.7.3) and add ~100 µL of toluene or nonane. Concentrate the extract to 50 µL under a gentle stream of nitrogen (Section 13.2). Make the volume to 1 mL with hexane. (Other concentration equipment may be used, provided that the requirements in Section 9 of this method have been met.) Proceed to Section 12.0 for extract cleanup.

11.3 Solid sample preparation and extraction (excluding tissues)

11.3.1 Weigh a well-mixed aliquot of each sample (of the same matrix type) sufficient to provide 10 g of dry solids (based on the solids determination in Section 11.1.1) into a clean beaker containing 75 - 100 grams of powdered sodium sulfate (Section 7.2.2). Stir the mixture well with a spatula and allow the mixture to dry to a free-flowing powder for a minimum of 30 minutes.

Note 1: *For very wet sediments, it may be preferable to add the anhydrous sodium sulfate in portions, stirring well after each addition.*

***Note 2*:** *Biosolids can contain PCBs at very high levels and 10 g (dry-weight) may be too much sample for the cleanup procedures to handle; therefore, it is recommended that 5 g (dry-weight) be used. The same weight of reagent grade sand and at least 75 g of sodium sulfate should be used for the associated method blank and OPR.*

11.3.2 Spike the sample with 32 µL of labeled compound standard solution (Section 7.8) and allow to equilibrate for at least 30 minutes. (Other volumes and spiking solution concentrations may be employed as long as the mass of labeled compound spiked into the sample equals the mass contained in the calibration standards.)

11.3.3 For each sample or sample batch (to a maximum of 20 samples) to be extracted during the same 12-hour shift, weigh two 10-g aliquots of the appropriate reference matrix (Section 7.6.2) and 75 g of sodium sulfate into clean beakers or glass jars. Stir the mixture well with a spatula. Spike 32 µL of the labeled compound standard solution (Section 7.8) into both reference matrix aliquots as described in Section 11.3.2. (Other volumes and spiking solution concentrations may be employed as long as the mass of labeled compound spiked into the sample equals the mass contained in the calibration standards.) One of these aliquots will serve as the method blank (Section 9.5). Spike 200 µL of the native standard solution (Section 7.11) into one reference matrix aliquot as described in Section 11.3.2. This aliquot will serve as the OPR (Section 15.5). Extract, cleanup, and concentrate both aliquots as described in Sections 11.3.4 – 11.3.5, 12.0, and 13.0.

11.3.4 Place a layer of silica (2 g, neutral, baked) into a clean Soxhlet thimble. Quantitatively transfer the mixture (Section 11.3.2 or 11.3.3) into the thimble. Lightly tapping the outside of the container may help transfer any solids that cling to sides of the beaker. If any solid still does not transfer, scrape the sides of the beaker with a spatula or add an additional portion of sodium sulfate until a freely flowing powder is achieved. Place the thimble into the Soxhlet body. Rinse the beaker with several small portions of 1:1 hexane:acetone and add the rinses to the thimble. Add 300 mL of 1:1 hexane:acetone and 4 - 5 anti-bumping granules to the Soxhlet's round-bottom flask. Extract the mixture for a minimum of 16 hours; adjust as necessary to achieve a reflux rate of at least 4 cycles per hour.

Note: *As noted in Section 6.5, very wet samples may require more sodium sulfate drying agent than will fit in the Soxhlet apparatus called out in that section. If a larger thimble and extractor are required, use a 5-g layer of neutral baked silica and extract the sample with 600 mL of solvent, using a larger round-bottom flask on the Soxhlet extractor.*

11.3.5 Allow the mixture to cool. Concentrate the extract by rotary evaporation (water bath < 30 °C) to ~20 mL. Add ~ 50 mL hexane and dry the extract by adding anhydrous granular sodium sulfate (up to 10 g may be required).

11.3.6 Quantitatively transfer to a round-bottom flask with hexane. Concentrate the extract by rotary evaporation (water bath < 30 °C) to 1 - 2 mL (Section 13.1). Transfer the extract to a centrifuge tube (Section 6.7.3) with hexane rinses and add ~100 µL of toluene or nonane. Concentrate the extract to 300 µL under a gentle stream of nitrogen (Section 13.2). Make the volume to 1 mL with hexane. (Other concentration equipment may be used, provided that the requirements in Section 9 of this method have been met.) Proceed to Section 12.0 for extract cleanup.

11.4. Fish and other tissue sample preparation and extraction - Prior to processing tissue samples, the laboratory must determine the exact tissue to be analyzed. Common requests for analysis of fish tissue include whole fish-skin on, whole fish-skin removed, edible fish fillets (filleted in the field or by the laboratory), specific organs, and other portions. Once the appropriate tissue has been determined, the sample must be homogenized.

11.4.1 Samples are homogenized while still frozen, where practical. If the laboratory must dissect the whole fish to obtain the appropriate tissue for analysis, the unused tissues may be rapidly refrozen and stored in a clean glass jar for subsequent use. Care must be taken when storing glass jars in the freezer to prevent breakage and leakage of sample.

11.4.2 Homogenize the sample using either a tissue homogenizer (Section 6.3.3) or a meat grinder (Section 6.3.4). Cut tissue that is too large to feed into the grinder into smaller pieces. To assure homogeneity when using a meat grinder, after the entire sample has been processed, mix the ground tissue with a spoon, transfer back to the grinder, and repeat the grinding at least two more times until the homogenize tissue has a consistent texture and color.

11.4.3 Weigh 10 g of homogenized tissue sample into a beaker containing 75 - 100 g of anhydrous granular sodium sulfate (Section 7.2.2). Stir the mixture well with a spatula and allow the mixture to dry to a free-flowing powder for a minimum of 30 minutes.

11.4.4 Spike the sample with 32 µL of labeled compound standard solution (Section 7.8) and allow to equilibrate for at least 30 minutes. (Other volumes and spiking solution concentrations may be employed as long as the mass of labeled compound spiked into the sample equals the mass contained in the calibration standards.)

11.4.5 For each sample or sample batch (to a maximum of 20 samples) to be extracted during the same 12-hour shift, weigh two 10-g aliquots of the tissue reference matrix (Section 7.6.3) and 75 g of sodium sulfate into a 400- to 500-mL beaker. Stir the mixture well with a spatula. Spike 32 µL of the labeled compound standard solution (Section 7.8) into both reference matrix aliquots as described in Section 11.4.4. (Other volumes and spiking solution concentrations may be employed as long as the mass of labeled compound spiked into the sample equals the mass contained in the calibration standards.) One of these aliquots will serve as the method blank (Section 9.5). Spike 200 µL of the native standard solution (Section 7.11) into one reference matrix aliquot as described in Section 11.4.4. This aliquot will serve as the OPR (Section 15.5). Extract, cleanup, and concentrate both aliquots as described in Sections 11.4.6, 11.4.7, 12.0, and 13.0.

11.4.6 Place a layer of silica (2 g, neutral, baked) into a clean Soxhlet thimble. Quantitatively transfer the mixture (sample or batch QC) into the thimble. Lightly tapping the outside of the container may help transfer any solids that cling to sides of the beaker. If any solid still does not transfer, scrape the sides of the beaker with a spatula or add an additional portion of sodium sulfate until a freely flowing powder is achieved. Place the thimble into the Soxhlet body. Rinse the beaker with several small portions of dichloromethane and add the rinses to the thimble. Add 300 mL dichloromethane and 4 – 5 anti-bumping granules (Section 7.3.2) to the Soxhlet's round-bottom flask. Extract the mixture for a minimum of 16 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the mixture to cool.

Note: *As noted in Section 6.5, larger tissue samples may require more sodium sulfate drying agent than will fit in the Soxhlet apparatus called out in that section. If a larger thimble and extractor are required, use a 5-g layer of neutral baked silica and extract the sample with 600 mL of solvent, using a larger round-bottom flask on the Soxhlet extractor.*

11.4.7 Concentrate the extract by rotary evaporation (< 30 °C) to 1 mL (Section 13.1). (Other concentration equipment may be used, provided that the requirements in Section 9 of this method have been met.) Add 1 mL hexane and proceed to Section 12.3 for GPC cleanup.

12.0 Extract Cleanup

All sample types require some form of cleanup, but the specific cleanups required, and their order will differ among the sample types. Florisil® cleanup (Section 12.1) is required for all sample types. All tissue samples require gel permeation chromatography (Section 12.3) to remove lipids, followed by Florisil® column cleanup (Section 12.1). For all other matrices, the order is Florisil® column, followed by copper treatment (Section 12.2) to remove sulfur. If silica gel is employed, it is recommended to be performed prior to Florisil® cleanup and copper treatment. Optional cleanup procedures are listed in Section 12.5. Alumina cleanup (Section 12.5.2) may be required if the extract is still discolored after Florisil® cleanup, or if matrix interferences are observed during instrumental analysis. Before using a cleanup procedure, the laboratory must demonstrate that the requirements of Section 9.2 can be met using the cleanup procedure. Batch QC samples must be taken through the same clean-up procedures as performed for the samples.

The following cleanup schemes are suggested based on results observed during the multi-laboratory validation of this method. More than one round of cleanup might be required for difficult samples.

- Aqueous samples: acid (12.5.3), Florisil® (12.1) and copper (12.2)
- Soils/Biosolids: alumina (12.5.2), silica (12.4), acid (12.5.3), Florisil® (12.1) and copper (12.2)
- Tissue: GPC (12.3), silica (12.4), copper (12.2), alumina (12.5.2) and Florisil® (12.1)

12.1 Florisil® cleanup

The use of Florisil® cleanup is mandatory for all types of samples. Sample extracts are cleaned up on a Florisil® column for which column cutpoints are regularly determined. If the extract is not in hexane, a solvent exchange to hexane (Section 13.2.3) must be performed before proceeding to the Florisil® column cleanup procedures.

12.1.1 Immediately prior to use, fill a glass column (10 mm x 300 mm with a 300-mL reservoir, Section 6.8.3.2) with hexane and dry pack the Florisil® (8 g, Section 7.5.2). Cap the Florisil® with a 10-mm layer of anhydrous sodium sulfate (Section 7.2.1).

12.1.2 Begin to drain the hexane from the column. Adjust the flow rate of eluant to 4.5 - 5.0 mL/min.

12.1.3 When the hexane is within 1 mm of the sodium sulfate, apply the sample extract (in hexane) to the column. Rinse the sample container twice with 1-mL portions of hexane and apply to the column.

12.1.4 Elute the column with 10:90 dichloromethane:hexane and collect the eluate in a round-bottom flask. Use the elution volumes established during the Florisil® cutpoint determination for the particular batch of Florisil® (Section 7.5.2). A typical cutpoint when using the eluate above is 25 mL.

12.1.5 Concentrate the extract to a small volume (~1 mL) by rotary evaporation. (Other concentration equipment may be used, provided that the requirements in Section 9 of this method have been met.) Proceed to Section 12.2 for copper cleanup. If copper cleanup is not required, transfer the extract to a centrifuge tube (Section 6.7.3) with solvent rinses. The first rinse must be with either toluene or nonane; all subsequent rinses are with hexane. Concentrate the extract to ~300 µL under a stream of nitrogen.

12.1.6 If no additional cleanup is required, proceed to Section 13.2.4 for final extract concentration. The extract will then be ready for instrumental analysis (Section 14).

12.2 Copper treatment (to remove sulfur)

Copper treatment to remove sulfur is required for all aqueous and solid matrices other than tissues. This cleanup should typically be performed after Florisil® cleanup, but can be performed prior to, in addition to, or after, Florisil® cleanup.

12.2.1 Add 0.1 g or one 1 x 1 cm square of activated copper (Section 7.5.5) to the extract to remove sulfur. If the copper turns black, add another 0.1 g or one 1 x 1 cm square of activated copper. Repeat this step until the copper no longer turns black. Transfer the extract to a centrifuge tube (Section 6.7.3) with solvent rinses. The first rinse must be with either toluene or nonane; all subsequent rinses are with hexane.

12.2.2 Concentrate the extract to ~300 µL under a stream of nitrogen. If no additional cleanup is necessary, proceed to Section 13.2.4.

12.3 Gel permeation chromatography (GPC)

This cleanup procedure is mandatory for all tissue extracts, and optional for all other matrices. GPC requires that the column not be overloaded. The column specified in this method is designed to handle a maximum of 0.5 g of interferences from an aqueous, soil, or mixed-phase sample in a 5-mL extract and has been shown to handle 1.5 g of lipid from a tissue sample in a 5-mL extract. If the extract is known or expected to contain more than these amounts, the extract is split into two or more aliquots for GPC, and the aliquots are combined after elution from the column. The residue content of the extract may be obtained gravimetrically by evaporating the solvent from a 50-µL aliquot.

Note: *The procedures described below are for an automated GPC system, however, manual GPC cleanup can also be performed provided all method performance criteria can be met. In addition, the GPC system may be calibrated using an aliquot of the native standard solution diluted in (1:1 dichloromethane:hexane) at a concentration similar to those expected in samples, instead of the GPC calibration solution (Section 7.4). If the native standard solution is used, then multiple fractions of the eluant need to be collected, concentrated to 300 µL and analyzed to determine which fractions need to be collected when samples are put through GPC cleanup.*

12.3.1 Column packing - Place 70 to 75 g of S-X3 Bio-Beads® (Section 7.5.4) in a 400- to 500-mL beaker. Cover the beads with 1:1 dichloromethane/hexane and allow to swell for 24 hours prior to column preparation. Transfer the swelled beads to the column (Section 6.8.1.1) and pump solvent through the column, from bottom to top, at 4.5 to 5.5 mL/minute prior to connecting the column to the detector. After purging the column with solvent for 1 to 2 hours, adjust the column head pressure to 7 to 10 psig and purge for 4 to 5 hours to remove air. Maintain a head pressure of 7 to 10 psig. Connect the column to the detector (Section 6.8.1.4).

12.3.2 Column calibration - Load 5 mL of the GPC calibration solution (Section 7.4) into the sample loop. Inject the GPC calibration solution and record the signal from the detector. The elution pattern will be corn oil, BEHP, methoxychlor, perylene, and sulfur. Set times for the initial waste fraction to allow >85% removal of BEHP. Begin collection to allow >85% collection of methoxychlor and continue to the sulfur peak maximum. Verify calibration with the GPC calibration solution after every 20 extracts. Calibration is verified if the recovery of the methoxychlor is greater than 85%. If calibration is not verified, the system must be recalibrated using the GPC calibration solution and the previous sample batch must be re-extracted and cleaned up using the calibrated GPC system.

12.3.3 Extract cleanup – Filter the extract or load through the filter holder (Section 6.8.1.3) to remove particles. Load the sample extract onto the column with 1:1 dichloromethane: hexane. Elute the extract with 1:1 dichloromethane:hexane at 5 mL/min using the calibration data determined in Section 12.3.2. Collect the eluate in a clean 400- to 500-mL beaker. Rinse the sample loading tube thoroughly with dichloromethane (~10 minutes) between extracts to prepare for the next sample. If an extract is encountered that could overload the GPC column to the extent that carry-over could occur, a 5.0-mL dichloromethane blank must be run through the system to check for carry-over. Concentrate the eluate under a gentle stream of nitrogen to 1 - 2 mL as described in Section 13.2. The extract is ready for the Florisil® column cleanup procedure (Section 12.1).

12.4 Silica gel cleanup

Silica gel cleanup is recommended for soil and biosolid samples.

12.4.1 Place a glass-wool plug in a chromatography column (10 mm x 300 mm with a 300-mL reservoir, Section 6.8.3.2). Prepare the column by sequentially packing the following layers in hexane, in the order shown:

- 0.5 g of neutral silica (Section 7.5.1.1)
- 2 g of basic silica (28% NaOH, Section 7.5.1.4)
- 0.5 g of neutral silica (Section 7.5.1.1)
- 4 g of acidic silica (44% H_2SO_4, Section 7.5.1.3)
- 2 g of acidic silica (22% H_2SO_4, Section 7.5.1.2)
- 1 g of neutral silica (Section 7.5.1.1)

Tap the column to settle the adsorbents.

12.4.2 Pre-elute the column with 50 to 100 mL of hexane. Close the stopcock when the hexane is within 1 mm of the silica. Discard the eluate. Check the column for channeling. If channeling is present, discard the column and prepare another.

12.4.3 Apply the concentrated extract to the column. Open the stopcock until the extract is within 1 mm of the silica.

12.4.4 Rinse the round-bottom flask or centrifuge tube twice with 1-mL portions of hexane and apply separately to the column. Elute the PCBs with 100 mL of hexane and collect the eluate.

12.4.5 Concentrate the eluate under a gentle stream of nitrogen to 300 µL, as described in Section 13.2.3. Make the volume to 1 mL with hexane.

12.4.6 For extracts of samples known to contain large quantities of other organic compounds, it may be advisable to increase the capacity of the silica gel column. This may be accomplished by increasing the strengths of the acidic and basic silica gels. Prepare a layered silica chromatography column (20 mm x 300 mm with a 250-mL reservoir, Section 6.8.3.3) by sequentially packing the following layers in hexane, in the order shown:

- 1 g of neutral silica (Section 7.5.1.1)
- 4 g of basic silica (28% NaOH, Section 7.5.1.4)
- 1 g of neutral silica (Section 7.5.1.1)
- 8 g of acidic silica (44% H_2SO_4, Section 7.5.1.3)
- 4 g of acidic silica (22% H_2SO_4, Section 7.5.1.2)
- 2 g of neutral silica (Section 7.5.1.1)

Tap the column to settle the adsorbents.

12.4.7 If additional cleanup is required, proceed to follow the instructions in the appropriate section, otherwise the extract is ready for Florisil® cleanup (Section 12.1).

12.5 Optional cleanup procedures

The following cleanup procedures are optional and may be performed on extracts that are still discolored, or if matrix interferences are encountered during instrumental analysis after performing the cleanup procedures in Sections 12.1 through 12.4.

12.5.1 GPC column cleanup (*required* for tissue extracts, but optional for all other sample types)

12.5.1.1 Transfer the extract to a centrifuge tube (Section 6.7.3) with enough 1:1 dichloromethane:hexane to make the volume to 2 mL. Load the extract onto a GPC column and elute with 1:1 dichloromethane:hexane as described in Section 12.3.

12.5.1.2 Add 1 mL toluene or nonane as a "keeper" and concentrate to 1 mL by rotary evaporation or other concentration equipment, provided it meets the requirements in Section 9.0. Transfer the extract to a centrifuge tube with hexane rinses and concentrate to ~300 µL under a stream of nitrogen.

12.5.1.3 Proceed to the appropriate cleanup procedure if additional cleanup is necessary. If instrumental analysis has already been performed and no additional cleanup is required, proceed to Section 13.2.4 for final extract concentration. If cleanup is performed because matrix interferences were encountered during instrumental analysis, do **not** add additional non-extracted internal standard.

12.5.2 Alumina column cleanup

12.5.2.1 Pack alumina (baked, 1% deactivated 6 g, Section 7.5.3) into a glass chromatographic column (10 mm x 150 mm with a 150-mL reservoir, Section 6.8.3.1) filled with hexane. Cap the alumina with a 10-mm layer of anhydrous granular sodium sulfate (Section 7.2.1).

12.5.2.2 Quantitatively transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 300 µL under a gentle stream of nitrogen, then add enough hexane to make the volume to 1 mL (note that solvent exchange [Section 13.2.3] to 300 µL is also sufficient if this cleanup is done after Florisil®, but before instrumental analysis). Load the extract in hexane onto an alumina column (6 g, 1% deactivated). Use the elution volumes determined from the alumina cutpoint determination (Section 7.5.3) of the particular batch of alumina. Collect the eluate in a round-bottom flask.

12.5.2.3 Add 1 mL of toluene or nonane as a "keeper," and concentrate the eluate to 1 mL by rotary evaporation. Transfer the extract to a centrifuge tube with hexane rinses and concentrate to ~300 µL under a stream of nitrogen.

12.5.2.4 Proceed to the appropriate cleanup procedure if additional cleanup is necessary. If instrumental analysis has already been performed and no additional cleanup is required, proceed to Section 13.2.4 for final extract concentration. If cleanup is performed because matrix interferences were encountered during instrumental analysis, do **not** add additional non-extracted internal standard.

12.5.3 Acid wash (perform in centrifuge tube)

This cleanup may be useful in removing interferences from extracts that exhibit a strong discoloration.

12.5.3.1 Transfer the extract with hexane rinses to a 15-mL centrifuge tube (Section 6.7.3). Reduce the volume to ~1 - 2 mL in hexane. Add 1 mL concentrated sulfuric acid (Section 7.1.2) to the extract and vortex the mixture thoroughly. Let stand (do not exceed 15 minutes). Draw off the lower (aqueous) layer and discard. If the aqueous layer is strongly colored, that indicates that the acid treatment is removing organic material from the extract, so repeat the procedure until the color in the aqueous layer diminishes, using up to 3 more washes with sulfuric acid.

12.5.3.2 Wash the extract by adding 1 mL reagent water (Section 7.6.1) and mix with a vortex mixer. Draw off the lower (aqueous) layer and discard. Repeat the wash with a second 1-mL portion of reagent water.

12.5.3.3 Prepare a sodium sulfate drying column by placing a small amount of glass wool into the end of a clean Pasteur pipette (Section 6.8.2) and adding a few centimeters of anhydrous sodium sulfate. Pass the extract through the drying column with hexane rinses and collect in a centrifuge tube.

12.5.3.4 If no further cleanup is required, add 100 µL toluene or nonane as a "keeper" and concentrate to ~300 µL under a stream of nitrogen; otherwise, proceed to the appropriate cleanup procedure.

12.5.3.5 If instrumental analysis has already been performed and no additional cleanup is required, proceed to Section 13.2.4 for final extract concentration. If cleanup is performed because matrix interferences were encountered during instrumental analysis, do **not** add additional non-extracted internal standard.

12.5.4 Anthropogenic Isolation Column - Used for removal of lipids from tissue extracts

The following cleanup procedure may be useful for tissue extracts in addition to GPC. If used, it may be performed before or after GPC.

12.5.4.1 Prepare a layered chromatography column (20 mm x 300 mm with a 250-mL reservoir, Section 6.8.3.3) by sequentially packing the following layers in hexane, in the order shown:

- 2 g silica gel (Section 7.5.1.1)
- 2 g potassium silicate (Section 7.5.6)
- 2 g granular anhydrous sodium sulfate (Section 7.2.1)
- 10 g acid silica gel (Section 7.5.1.2)
- 2 g granular anhydrous sodium sulfate (Section 7.2.1)

Tap the column to settle the adsorbents.

12.5.4.2 Pre-elute the column with 100 mL of hexane. Drain the hexane layer to the top of the column, but do not expose the sodium sulfate.

12.5.4.3 Load the sample extract and rinses onto the column by draining each portion to the top of the bed. Elute the PCBs from the column into round-bottom flask using 200 mL of hexane.

12.5.4.4 Remove a small portion (e.g., 50 µL) of the extract for determination of residue content. Estimate the percent of the total that this portion represents. Concentrate the small portion to constant weight per Section 13.2. Calculate the total amount of residue in the extract. If more than 500 mg of material remains, repeat the cleanup using a fresh anthropogenic isolation column.

12.5.4.5 If necessary, exchange the extract to a solvent suitable for the additional cleanups to be used.

12.5.4.6 Additional sample extract cleanup using GPC (Section 12.3) and Florisil (Section 12.1) are recommended at a minimum.

12.5.4.7 If it is the last cleanup performed, then proceed to Section 13.2.4 for extract concentration.

13.0 Extract Concentration

13.1 Macro-concentration by rotary evaporator

Note: *In the concentration procedures below, the extract* ***must not*** *be allowed to concentrate to dryness because the mono- through tri-chlorobiphenyls may be totally or partially lost. Other concentration equipment may be used, provided that the requirements in Section 9 of this method have been met.*

13.1.1 Concentrate each extract in a separate round-bottom flask.

13.1.2 Assemble the rotary evaporator according to manufacturer's instructions and warm the water bath to < 30 ºC. On a daily basis, pre-clean the rotary evaporator by concentrating 100 mL of clean extraction solvent through the system. Archive both the concentrated solvent and the solvent in the catch flask for a contamination check if necessary. Between samples, three 2- to 3-mL aliquots of solvent should be rinsed down the feed tube into a waste beaker.

13.1.3 Attach the round-bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system and begin rotating the sample flask.

13.1.4 Lower the flask into the water bath and adjust the speed of rotation and the temperature as required to complete concentration in 15 to 20 minutes. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

Note: *If the rate of concentration is too fast, analyte loss may occur.*

13.1.5 When the liquid in the concentration flask has reached an apparent volume of approximately 2 mL, remove the flask from the water bath and stop the rotation. Slowly and carefully admit air into the system. Be sure not to open the valve so quickly that the sample is blown out of the flask. Rinse the feed tube with approximately 2 mL of solvent.

13.1.6 Transfer the extract to a centrifuge tube (Section 6.7.3) using 2 - 3 rinses of solvent. Proceed with micro-concentration and solvent exchange (Section 13.2).

13.2 Micro-concentration and solvent exchange

13.2.1 Extracts to be subjected to GPC cleanup are exchanged into dichloromethane. Extracts to be cleaned up using silica gel, alumina, acid wash, or Florisil® are exchanged into hexane (see Section 13.2.3).

13.2.2 Transfer the centrifuge tube containing the sample extract to a nitrogen evaporation device. Adjust the flow of nitrogen so that the surface of the solvent is just visibly disturbed and the temperature of the water bath to below the boiling point of the solvent being concentrated.

Note: *A large vortex in the solvent may cause analyte loss.*

13.2.3 If the sample is not a hexane extract, a solvent exchange to hexane must be done before proceeding to column cleanup procedures. To exchange the solvent, transfer the extract to a centrifuge tube with hexane. If extraction solvent is other than dichloromethane or hexane, add ~ 100 µL (10 drops) of toluene or nonane (unless already present). Evaporate to 300 µL (or 50 µL if toluene, acetone, or other solvents are present) under a stream of nitrogen. Add 1 mL hexane and proceed with cleanup procedures (Section 12).

13.2.4 If the extract is to be concentrated for injection into the GC/MS (Section 14), quantitatively transfer the extract to an autosampler vial for final concentration, rinsing the centrifuge tube with hexane and adding the rinse to the autosampler vial. Concentrate the extract to 200 - 250 µL under a gentle stream of nitrogen. Repeat the rinse and concentration sequence twice more, taking care not to reduce the volume in the autosampler vial to less than 200 µL until after the final rinse. Concentrate the extract to ~60 µL. Cap the autosampler vial. The extract is ready for instrumental analysis (Section 14). Store in the dark at room temperature until ready for GC/MS analysis. If GC/MS analysis will not be performed on the same day, store the vial at less than -20 ºC.

14.0 Instrumental Analysis

14.1 Analysis is performed on a low-resolution mass spectrometer (LRMS) equipped with a gas chromatograph (GC) operating on manufacturer's software. The MS is operated at a unit mass resolution in the electron ionization (EI) mode using SIM, acquiring two characteristic ions for each target analyte and labeled compound standard. A splitless/split injection sequence is used. The ions to be acquired are listed in Table 2.

14.2 Establish the operating conditions given in Section 10.1.

14.3 Add an aliquot (40 µL) of the NIS solution (Section 7.9) to the sample extract immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction. If an extract is to be reanalyzed and evaporation has occurred, **do not add** additional NIS. Rather, bring the extract back to its previous volume with pure hexane (e.g., 99 µL if a 1-µL injection used or 98 µL if a 2-µL injection used).

14.4 Inject 1.0 or 2.0 µL of the concentrated extract containing the NISs using on-column or splitless injection. The volume injected must be identical to the volume used for calibration (Section 10.5).

If necessary, extracts are diluted with solvent (Section 16.4) to bring all target responses within the calibration range.

14.5 After a successful initial calibration has been completed, including analyses of the retention time marker solutions, the analytical sequence for a batch of samples analyzed during the same 12-hour period is as follows:

1. Calibration verification
2. OPR
3. Solvent (toluene or nonane) blank
4. Method blank
5. Samples
6. Calibration verification

The closing calibration verification (CV) solution must be injected into the GC/MS within 12 hours of the injection of the opening calibration verification (CV) solution. If the results are acceptable, the closing calibration verification solution may be used as the opening solution for the next 12-hour period.

14.6 Analysis of Complex Samples

14.6.1 Some samples may contain high levels (> 10 ng/L for liquids, > 1000 ng/kg for solids) of the compounds of interest, interfering compounds, and/or polymeric materials. Some extracts may not concentrate to 60 µL (Section 13.2.4). Others may overload the GC column and/or mass spectrometer. Fragment ions from congeners at higher levels of chlorination may interfere with determination of congeners at lower levels of chlorination (Section 16.3).

14.6.2 Re-extract and analyze a smaller aliquot of the sample (Section 16.4) when the extract will not concentrate to 60 µL after all cleanup procedures have been exhausted. If a smaller aliquot of soils or mixed-phase samples is analyzed, attempt to ensure that the aliquot is representative of the bulk sample.

14.6.3 Perform integration of peak areas and calculate concentrations manually when interferences preclude computerized calculations.

15.0 Performance Tests

15.1 A mass resolution verification must be performed at the frequency stated in Section 10.6.1.

15.2 Mass accuracy calibration must be verified at the beginning of each 12-hour period as per Section 10.6.2. If analyses are performed on successive shifts, a closing mass accuracy verification check is required which serves as the start of the next shift. If the requirement in Section 10.3 cannot be met, the problem must be corrected before analyses can proceed. If any of the samples in the previous shift may be affected by poor mass resolution, those samples must be re-analyzed.

15.3 Calibration verification (CV)

After a passing the mass accuracy check (Section 15.2), the GC/MS system performance and calibration are verified for all native PCB congeners and labeled compounds by analyzing a CV standard (Section 10.6.4) at the beginning of each 12-hour period. Only after all performance

criteria are met may samples, blanks, MDLs, IPRs and OPRs be analyzed. A bracketing CV standard is analyzed at the end of the 12 hours.

15.3.1 CV calculations

Calculate an RR and RF for each native congener and RF_s for each label compound using the equations in Section 10.3.3.2.

15.3.2 CV criteria

The RRs and RFs for CVs must be within ± 20% of the mean RRs and RFs from the associated initial calibration. The RRs and RFs for the bracketing CVs must be within ± 20% of each other.

15.3.3 Instrument sensitivity check

The %D values for each of the three non-extracted internal standards in the calibration verification standard calculated in Section 10.6.4 must be within the range of -50% to +100% (e.g., a factor of 2) of their mean responses in the most recent initial calibration. Failure to meet this criteria indicates a substantive change in the sensitivity of the GC/MS over time. Extremely low area responses for all three non-extracted internal standards simultaneously may indicate a bad addition of the internal standards to the calibration verification standard.

15.3.4 CV corrective action

If the CV criteria in either Section 15.3.2 or 15.3.3 are not met, perform any necessary instrument maintenance, and reinject the CV. If the criteria are still not met, recalibrate the GC/MS instrument according to Section 10.2.

15.4 Retention times and GC resolution

15.4.1 The absolute retention times of each native congener and labeled analog must be within ± 3 seconds in the CV (Section 15.3).

15.4.2 The relative retention times (RRTs) of native PCBs and labeled compounds in the CV (Section 15.3) must be within their respective RRT limits established during calibration (Section 10.4.1).

15.5 Ongoing precision and recovery (OPR)

15.5.1 After verification, analyze the extract of the OPR (Sections 11.2.1.4, 11.2.2.3, 11.2.3.3, 11.2.4.3, 11.3.3, and 11.4.5) prior to analysis of samples from the same batch to ensure the analytical process is under control.

15.5.2 Compute the percent recovery of the native congeners by the appropriate quantification method depending on the congener (Section 10.5). Compute the percent recovery of each labeled compound by the non-extracted internal standard method (Sections 1.2 and 10.5).

$$Recovery\ (\%) = \frac{Concentration\ found\ (ng/mL)}{Concentration\ spiked\ (ng/mL)}\ x\ 100$$

15.5.3 For the spiked congeners and labeled compounds, compare the recovery to the OPR limits given in Table 8 and Table 9. If all compounds meet the acceptance criteria, system performance is acceptable, and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, the extraction/concentration processes are not being performed properly for that compound. In this event, correct the problem, re-prepare, extract, and clean up the sample batch and repeat the ongoing precision and recovery test.

15.5.4 If desired, add results that pass the specifications in Section 15.5.3 to initial and previous ongoing data for each compound in each matrix. Update QC charts to form a graphic representation of continued laboratory performance. Develop a statement of laboratory accuracy for each congener in each matrix type by calculating the average percent recovery (R) and the standard deviation of percent recovery (SR). Express the accuracy as a recovery interval from R - 2SR to R + 2SR. For example, if R = 95% and SR = 5%, the accuracy is 85 to 105%.

15.6 Labeled compound recoveries for field samples should fall within the acceptance criteria given in Table 10. Given that there are 29 labeled compounds being tested simultaneously, some failures of individual labeled compounds are not unexpected. The laboratory is allowed to have up to three labeled compounds in a single field sample that do not meet the acceptance criteria; however, the recoveries for those compounds must not be < 5%.

15.7 Solvent blank – After the analysis of the OPR, analyze a solvent blank (toluene only) to ensure no instrument contamination has occurred.

15.8 Method blank – After the analysis of the solvent blank and prior to the analysis of samples, analyze a method blank (Section 9.5).

15.9 Non-extracted internal standard areas

As described in Section 15.3.3, the overall sensitivity of the GC/MS system must be checked during each calibration verification to protect against gradual changes in instrument performance over time. In addition, *although not required*, it can be a useful QC practice to periodically examine the responses of the three non-extracted internal standards in field samples and QC samples, because such a check may be helpful in explaining other sample-specific performance issues.

16.0 Data Analysis and Calculations

16.1 Qualitative determination GC/LRMS Peak Identification

A native PCB or labeled compound is identified in a standard, blank, or sample when all of the criteria in Sections 16.1.1 through 16.1.5 are met.

16.1.1 Peak responses must be at least three times the background noise level (signal-to-noise ratio [S/N] ≥3:1). If the S/N ratio is not met due to high background noise, the laboratory must correct the issue (e.g., check and replace as needed transfer lines, filaments, liner, GC column, etc.). The instrument must be recalibrated (Section 10) after maintenance. If the S/N ratio is not met, but the background is low, then the analyte is to be considered a non-detect.

16.1.2 The RRTs must be within the window predicted from the retention time calibration runs (Section 10.1.2).

16.1.3 Peak maxima for quantification and confirmation ions must coincide within two seconds.

16.1.4 The relative ion abundance ratios must be within 20% of the theoretical (see Table 2). If the ion abundance ratio for an analyte in a sample (not a standard) falls outside of the 20% window, but it is within 10% of the ratio of the same analyte in the most recent CV standard, then the analyte identification is acceptable, but the issue must be noted by the laboratory in its data report.

16.1.5 Any response at the masses for PCB congeners containing one or two more chlorine atoms than the target PCB congener that occurs at the same retention time as the target PCB congener should be evaluated for possible effect. A fragment ion from a more highly chlorinated congener may enhance the response of the target congener. Where the target response may be enhanced, the concentration is reported as a maximum possible value.

16.1.6 If the field sample result does not all meet the criteria stated in Sections 16.1.2 through 16.1.5, and all sample preparation avenues (e.g. extract clean-up, sample dilution, re-extraction, etc.) have been exhausted, the result must be reported with a data qualifier alerting the data user that the result could not be confirmed because it did not meet the method required criteria and therefore should be considered an estimated value. If the criteria listed above are not met for the standards, the laboratory must stop analysis of samples immediately and correct the issue.

16.2 Quantitative determination/concentrations

Concentrations of the target analytes are determined with respect to a labeled compound, as shown in Table 2, and concentrations of the labeled compounds added to the sample before extraction are determined with respect to a NIS, as shown in Table 3, using the response ratios or response factors from the most recent multi-level initial calibration (Section 10.3), as follows. For the native analytes:

$$Concentration\ (ng/L\ or\ ng/g) = \frac{Area_n\ M_l}{Area_l\ (RR\ or\ RF)} \times \frac{1}{sample\ volume\ or\ weight\ (L\ or\ g)}$$

where:

$Area_n$ = The measured area of the Q1 *m/z* for the native (unlabeled) PCB
$Area_l$ = The measured area at the Q1 *m/z* for the labeled PCB
M_l = The mass of the labeled compound added to the sample prior to extraction (nominally 40 ng)
RR = Response ratio used to quantify target congeners by the isotope dilution method
RF = Response factor used to quantify target compounds by the extracted internal standard method

And for the labeled analytes:

$$Concentration\ (ng/L\ or\ ng/g) = \frac{Area_l\ M_{nis}}{Area_{nis}(RF_s)} \times \frac{1}{sample\ volume\ or\ weight\ (L\ or\ g)}$$

where:

$Area_l$ = The measured area at the Q1 *m/z* for the labeled PCB
$Area_{nis}$ = The measured area of the Q1 *m/z* for the non-extracted internal standard
M_{nis} = The mass of the non-extracted internal standard compound added to the sample extract (nominally 40 ng)
RF_s = Response factor used to quantify the labeled compound by the non-extracted internal standard method

Results for target compounds are recovery corrected by the method of quantification. Labeled compound recoveries are determined similarly against the non-extracted internal standard and are used as general indicators of overall analytical quality. Typical RF and RR seen during the multi-laboratory validation study ranged from 0.207 to 1.624. The range is given here only as guidance. Because each instrument has unique performance characteristics, acceptance limits for RF/RR are not part of this method; however, each laboratory is expected to establish in-house ranges.

16.3 Area subtraction of higher homolog interferences from PCBs 35, 77, 81, 123, 126, and 157

The loss of one or more chlorines from a more highly chlorinated congener will inflate the concentration or may produce a false positive result for a less-chlorinated congener that elutes at the same retention time. For example, PCB-110 (Cl_5) coelutes with PCB-77 (Cl_4) and has a primary quantification ion (Q1) of 326, but through the loss of one chlorine, it also produces an ion at 292, which is the Q1 for PCB-77. While the intensity of ion 292 is very small relative to ion 326 from PCB-110, it could represent a significant portion of the response of PCB-77, since PCB-77 is often found at low concentrations.

Based on the GC column and conditions specified in this method, the six congeners in Table 11 elute very closely with congeners from higher levels of chlorination (higher homologs) and thus are likely subject to higher homolog interferences. Using data from the injection of the higher homolog congener in Table 11 (in the absence of the lower homolog congener), it was possible to experimentally determine the proportion of the interfering ion from the higher homolog that is likely contributing to the signal for the congener in the lower homolog. As indicated by the correction factor in Table 11, the intensity of *m/z* 292 resulting from PCB-110 at the retention time of PCB-77 is just over 5% (e.g. 0.0567) of the intensity of *m/z* 326. Therefore, using the actual response in a sample for the Q1 of PCB-110, the response for PCB-77 can be corrected for the interference.

The quantification ion areas for the affected congeners are corrected by multiplying the area of the quantification ion (Q1) of the higher (interfering) homologue by an experimentally determined correction factor (see Table 11) and subtracting the product from the area of Q1 of the co-eluting lower homologue congener (see equation below).

When using the GC column and chromatographic conditions described in this method, the responses for the congeners in Table 11 can be corrected using the equation below and the correction factors in Table 11.

$$Q1_c = Q1_a - (Q1_i \times CF)$$

where:

$Q1_c$ = Area of corrected quantification ion for affected congener
$Q1_a$ = Area of uncorrected quantification ion for affected congener in the lower homolog
$Q1_i$ = Area of quantification ion for higher homolog (interfering) congener
CF = Correction factor (see Table 11)

Note 1: *Correction factors, interfering, and affected congeners will need to be re-established if a GC column or chromatographic conditions other than those specified in Sections 6.12 and 10.1 are used. That effort will require examination of the retention times for the congeners in each homolog and comparisons among the retention times with congeners in the two homologs immediately above (e.g., compare the tetrachlorinated congeners with the pentachlorinated and hexachlorinated congeners). Where coelutions occur, standards containing the higher homolog will need to be injected and the responses at the Q1 ions for the lower and higher homologs monitored at the retention times of concern. A minimum of three injections of each standard are recommended to obtain data on the relative contribution of the higher homolog to*

the Q1 ion of the lower homolog, such that new correction factors can be derived. Correction factors can be calculated using the following equation:

$$CF = \frac{Area\ at\ Q1\ of\ the\ lower\ homolog\ congener\ contributed\ by\ the\ higher\ homolog\ congener}{Q1_i}$$

Use the highest area of the higher homolog congener from the three (or more) injections of each standard as the area in the equation above.

***Note 2**: The results for these six congeners should be considered to be maximum values, due to the potential for interferences. Laboratories should use a data qualifier to advise the data user when a correction factor has been applied to the results for a given congener.*

16.4 Sample dilutions

16.4.1 If the Q1 area for any congener exceeds the calibration range of the system, dilute the sample extract with hexane by the factor necessary to bring the concentration within the calibration range, adjust the amount of the NIS to 40 ng in the diluted extract, and analyze an aliquot of this diluted extract. If the PCB congener cannot be measured reliably by isotope dilution, dilute and analyze an aqueous sample, or analyze a smaller portion of a soil, tissue, or mixed-phase sample. Adjust the PCB congener concentrations, detection limits, and minimum levels to account for the dilution.

16.4.2 As noted in Section 9.3.3, up to three labeled compound recoveries may be outside the acceptance limits in Table 10; however, those recoveries must not be < 5%. If the recoveries for more than three labeled compound are outside of the normal range (Table 10), or any recoveries are < 5%, a diluted sample must be analyzed (Section 16.4.1). If the recovery of any labeled compound in the diluted sample is outside of the acceptance limits, the method does not apply to the sample being analyzed and the result may not be reported or used for permitting or regulatory compliance purposes. In this case, alternative extraction and cleanup procedures in this method or an alternative GC column should be employed to resolve the interference. If all cleanup procedures in this method and an alternative GC column have been employed and labeled compound recovery remains outside of the acceptance limits, extraction and/or cleanup procedures that are beyond this scope of this method will be required to analyze the sample.

16.5 Reporting of results

The data reporting practices described here are focused on wastewater compliance monitoring under the National Pollutant Discharge Elimination System (NPDES) and other Clean Water Act programs and may not be relevant to other uses of the method.

16.5.1 Report results for wastewater samples in ng/L. Report results for solid samples in ng/g, on a dry-weight basis, and report the percent solids for each sample separately. Report results for tissue samples in ng/g, on a wet-weight basis. If requested, determine the % lipid content of each tissue sample, and report it separately. (Other units may be used if required in a permit.) Report all QC data with the sample results.

16.5.2 Reporting level

Unless specified otherwise by a regulatory authority or in a discharge permit, results for analytes that meet the identification criteria are reported down to the concentration of the ML established by the laboratory through calibration of the instrument (see the glossary for

the derivation of the ML). EPA considers the terms "reporting limit," "quantitation limit," "limit of quantitation," and "minimum level" to be synonymous.

16.5.2.1 Report a result for each analyte in each field sample or QC standard at or above the ML to 3 significant figures. Report a result for each analyte found in each field sample or QC standard below the ML as "<ML," where ML is the concentration of the analyte at the ML, or as required by the regulatory/control authority or permit. Each laboratory must establish their own MDLs and MLs for all 209 congeners and for each matrix, as described in Section 9.2.2. The MDLs and MLs determined in a given laboratory will differ from the pooled values show in Table 12 but should generally be in a similar range.

16.5.2.2 Report a result for each analyte in a blank at or above the MDL to 2 significant figures. Report a result for each analyte not found in a blank above the MDL as "<MDL," where MDL is the concentration of the analyte at the MDL, or as required by the regulatory/control authority or permit. Blank subtraction is not allowed, unless explicitly requested or required by a regulatory authority or in a permit. In this case, both the sample result and the blank results must be reported together.

16.5.2.3 Report a result for an analyte found in a sample or extract that has been diluted at the least dilute level at which the area at the quantitation *m/z* is within the calibration range (i.e., above the ML for the analyte) and with labeled compound recoveries within their respective QC acceptance criteria. This may require reporting results for some analytes from different analyses.

16.5.3 Results from tests performed with an analytical system that is not in control (i.e., that does not meet acceptance criteria for any QC tests in this method) must be documented and reported (e.g., as a qualifier on results), unless the failure is not required to be reported as determined by the regulatory/control authority. Results associated with a QC failure cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results. If the holding time would be exceeded for a reanalysis of the sample, the regulatory/control authority should be consulted for disposition.

16.5.4 Coeluting congeners are reported as a sum of the congeners (e.g., PCB-66+80) and will not have CAS number assigned. Laboratories are encouraged to flag coeluting congeners when reporting results.

17.0 Method Performance

Initial method performance in each laboratory is assessed by performing the initial demonstration of capability (Section 9.2) and QC sample analyses. Ongoing method performance is monitored through QC samples analyzed alongside samples and through the use of labeled compounds added to every sample. The parameters monitored include percent recovery of labeled compounds, blank concentrations, native compound recoveries.

This method was validated, and interim performance specifications were developed using data from EPA's interlaboratory validation study (Reference 13). The interim quality control acceptance criteria developed from the interlaboratory study are located in Tables 8 thru 10.

Every laboratory performing analyses in support of Clean Water Act compliance monitoring must have an effective quality management system in place (see Section 9.1). Such systems must include assessment of all results against the various QC acceptance limits in a given analytical method, but also should include procedures for longer-term internal evaluations of laboratory performance. EPA expects that responsible laboratories will perform such evaluations and develop and apply in-house acceptance criteria, which by virtue of being from a single laboratory, will be narrower than the interim acceptance criteria listed in the tables in this method.

18.0 Pollution Prevention

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Many opportunities for pollution prevention exist in laboratory operations. EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address waste generation. When wastes cannot be reduced feasibly at the source, the Agency recommends recycling as the next best option.

18.2 The PCB congeners in this method are used in extremely small amounts and pose little threat to the environment when managed properly. Standards should be prepared in volumes consistent with laboratory use to minimize the disposal of excess volumes of expired standards.

18.3 For information about pollution prevention that may be applied to laboratories and research institutions, consult *Less is Better: Laboratory Chemical Management for Waste Reduction* (Reference 7).

19.0 Waste Management

19.1 The laboratory is responsible for complying with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance is also required with any sewage discharge permits and regulations. An overview of requirements can be found in *Environmental Management Guide for Small Laboratories* (Reference 8).

19.2 Samples at pH < 2 or pH > 12, are hazardous and must be handled and disposed of as hazardous waste or neutralized and disposed of in accordance with all federal, state, and local regulations. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions.

19.3 The PCBs decompose above 800 ºC. Low-level waste such as absorbent paper, tissues, animal remains, and plastic gloves may be burned in an appropriate incinerator. Gross quantities (milligrams) should be packaged securely and disposed of through commercial or governmental channels that are capable of handling extremely toxic wastes.

19.4 For further information on waste management, consult Less is Better: Guide to Minimizing Waste in Laboratories (Reference 7) and Prudent Practices in the Laboratory: Handling and Management of Chemical Hazards (Reference 9).

20.0 References

1. "NIOSH Pocket Guide to Chemical Hazards," Department of Health and Human Services, Centers for Disease Control, NIOSH, Publication 2005-149, September 2007, NTIS PB-2005-108099.

2. "OSHA Safety and Health Standards, General Industry," OSHA 2206, 29 CFR 1910.

3. "Safety in Academic Chemistry Laboratories," ACS Committee on Chemical Safety, 8th Edition, 2017.

4. "Standard Methods for the Examination of Water and Wastewater," 18th edition and later, American Public Health Association, 1015 15th St, NW, Washington, DC 20005, 1-35: Section 1090 (Safety), 1992 - 2017.

5. "Standard Practice for Sampling Water from Flowing Process Streams," ASTM D3370-18, ASTM, 1916 Race Street, Philadelphia, PA 19103-1187, 2018.

6. "Guidance for Developing Quality Systems for Environmental Programs," USEPA Office of Environmental Information, Washington DC, 20460, EPA QA/G-1, November 2002.

7. "Less is Better: Guide to Minimizing Waste in Laboratories," American Chemical Society, 2002. Available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington, DC 20036.

8. "Environmental Management Guide for Small Laboratories," USEPA, Small Business Division, Washington DC, EPA 233-B-00-001, May 2000.

9. "Prudent Practices in the Laboratory: Handling and Management of Chemical Hazards," National Research Council (US) Committee on Prudent Practices in the Laboratory, 2011. Available from the National Academies Press, 500 Fifth Street, NW, Washington, DC 20001

10. "Contaminant Trends in U.S. National Estuarine Research Reserves," NOAA Technical Memorandum NCCOS 156, National Status and Trends Program, Center for Coastal Monitoring and Assessment, National Centers for Coastal Ocean Science, National Ocean Service, National Oceanic and Atmospheric Administration, N/SCI 1, SSMC4, Room 9109, 1305 East-West Highway, Silver Spring, MD 20910, October 2002.

11. Van den Berg, Martin, Linda S. Birnbaum, Michael Denison, Mike De Vito, William Farland, Mark Feeley, Heidelore Fiedler, Helen Hakansson, Annika Hanberg, Laurie Haws, Martin Rose, Stephen Safe, Dieter Schrenk, Chiharu Tohyama, Angelika Tritscher, Jouko Tuomisto, Mats Tysklind, Nigel Walker, and Richard E. Peterson, 2006, "The 2005 World Health Organization Reevaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin-like Compounds," Toxicological Sciences 93(2): 223-241.

12. "Definition and Procedure for the Determination of the Method Detection Limit," USEPA, Office of Water, Engineering and Analysis Division, Washington DC, EPA 821-R-16-006, December 2016.

13. "Report on the Multi-laboratory Validation of Clean Water Act Method 1628 for PCB Congeners," USEPA, Office of Water, Engineering and Analysis Division, Washington DC, EPA 820-R-21-003, April 2021.

21.0 Tables

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native PCB Congeners

PCB congener[1]	Congener number	CAS Number
2-MoCB	PCB-1	2051-60-7
3-MoCB	PCB-2	2051-61-8
4-MoCB	PCB-3	2051-62-9
2,2'-DiCB	PCB-4	13029-08-8
2,3-DiCB	PCB-5	16605-91-7
2,3'-DiCB	PCB-6	25569-80-6
2,4-DiCB	PCB-7	33284-50-3
2,4'-DiCB[2]	PCB-8	34883-43-7
2,5-DiCB	PCB-9	34883-39-1
2,6-DiCB	PCB-10	33146-45-1
3,3'-DiCB	PCB-11	2050-67-1
3,4-DiCB	PCB-12	2974-92-7
3,4'-DiCB	PCB-13	2974-90-5
3,5-DiCB	PCB-14	34883-41-5
4,4'-DiCB	PCB-15	2050-68-2
2,2',3-TrCB	PCB-16	38444-78-9
2,2',4-TrCB	PCB-17	37680-66-3
2,2',5-TrCB[2]	PCB-18	37680-65-2
2,2',6-TrCB	PCB-19	38444-73-4
2,3,3'-TrCB	PCB-20	38444-84-7
2,3,4-TrCB	PCB-21	55702-46-0
2,3,4'-TrCB	PCB-22	38444-85-8
2,3,5-TrCB	PCB-23	55720-44-0
2,3,6-TrCB	PCB-24	55702-45-9
2,3',4-TrCB	PCB-25	55712-37-3
2,3',5-TrCB	PCB-26	38444-81-4
2,3',6-TrCB	PCB-27	38444-76-7
2,4,4'-TrCB[2]	PCB-28	7012-37-5
2,4,5-TrCB	PCB-29	15862-07-4
2,4,6-TrCB	PCB-30	35693-92-6
2,4',5-TrCB	PCB-31	16606-02-3
2,4',6-TrCB	PCB-32	38444-77-8
2',3,4-TrCB	PCB-33	38444-86-9
2',3,5-TrCB	PCB-34	37680-68-5
3,3',4-TrCB	PCB-35	37680-69-6
3,3',5-TrCB	PCB-36	38444-87-0
3,4,4'-TrCB	PCB-37	38444-90-5
3,4,5-TrCB	PCB-38	53555-66-1
3,4',5-TrCB	PCB-39	38444-88-1
2,2',3,3'-TeCB	PCB-40	38444-93-8
2,2',3,4-TeCB	PCB-41	52663-59-9
2,2',3,4'-TeCB	PCB-42	36559-22-5
2,2',3,5-TeCB	PCB-43	70362-46-8
2,2',3,5'-TeCB[2]	PCB-44	41464-39-5

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native PCB Congeners

PCB congener[1]	Congener number	CAS Number
2,2',3,6-TeCB	PCB-45	70362-45-7
2,2',3,6'-TeCB	PCB-46	41464-47-5
2,2',4,4'-TeCB	PCB-47	2437-79-8
2,2',4,5-TeCB	PCB-48	70362-47-9
2,2',4,5'-TeCB	PCB-49	41464-40-8
2,2',4,6-TeCB	PCB-50	62796-65-0
2,2',4,6'-TeCB	PCB-51	68194-04-7
2,2',5,5'-TeCB[2]	PCB-52	35693-99-3
2,2',5,6'-TeCB	PCB-53	41464-41-9
2,2',6,6'-TeCB	PCB-54	15968-05-5
2,3,3',4-TeCB	PCB-55	74338-24-2
2,3,3',4'-TeCB	PCB-56	41464-43-1
2,3,3',5-TeCB	PCB-57	70424-67-8
2,3,3',5'-TeCB	PCB-58	41464-49-7
2,3,3',6-TeCB	PCB-59	74472-33-6
2,3,4,4'-TeCB	PCB-60	33025-41-1
2,3,4,5-TeCB	PCB-61	33284-53-6
2,3,4,6-TeCB	PCB-62	54230-22-7
2,3,4',5-TeCB	PCB-63	74472-34-7
2,3,4',6-TeCB	PCB-64	52663-58-8
2,3,5,6-TeCB	PCB-65	33284-54-7
2,3',4,4'-TeCB[2]	PCB-66	32598-10-0
2,3',4,5-TeCB	PCB-67	73575-53-8
2,3',4,5'-TeCB	PCB-68	73575-52-7
2,3',4,6-TeCB	PCB-69	60233-24-1
2,3',4',5-TeCB	PCB-70	32598-11-1
2,3',4',6-TeCB	PCB-71	41464-46-4
2,3',5,5'-TeCB	PCB-72	41464-42-0
2,3',5',6-TeCB	PCB-73	74338-23-1
2,4,4',5-TeCB	PCB-74	32690-93-0
2,4,4',6-TeCB	PCB-75	32598-12-2
2',3,4,5-TeCB	PCB-76	70362-48-0
3,3',4,4'-TeCB[2,3]	PCB-77	32598-13-3
3,3',4,5-TeCB	PCB-78	70362-49-1
3,3',4,5'-TeCB	PCB-79	41464-48-6
3,3',5,5'-TeCB	PCB-80	33284-52-5
3,4,4',5-TeCB[3]	PCB-81	70362-50-4
2,2',3,3',4-PeCB	PCB-82	52663-62-4
2,2',3,3',5-PeCB	PCB-83	60145-20-2
2,2',3,3',6-PeCB	PCB-84	52663-60-2
2,2',3,4,4'-PeCB	PCB-85	65510-45-4
2,2',3,4,5-PeCB	PCB-86	55312-69-1
2,2',3,4,5'-PeCB	PCB-87	38380-02-8
2,2',3,4,6-PeCB	PCB-88	55215-17-3
2,2',3,4,6'-PeCB	PCB-89	73575-57-2
2,2',3,4',5-PeCB	PCB-90	68194-07-0

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native PCB Congeners

PCB congener[1]	Congener number	CAS Number
2,2',3,4',6-PeCB	PCB-91	68194-05-8
2,2',3,5,5'-PeCB	PCB-92	52663-61-3
2,2',3,5,6-PeCB	PCB-93	73575-56-1
2,2',3,5,6'-PeCB	PCB-94	73575-55-0
2,2',3,5',6-PeCB	PCB-95	38379-99-6
2,2',3,6,6'-PeCB	PCB-96	73575-54-9
2,2',3',4,5-PeCB	PCB-97	41464-51-1
2,2',3',4,6-PeCB	PCB-98	60233-25-2
2,2',4,4',5-PeCB	PCB-99	38380-01-7
2,2',4,4',6-PeCB	PCB-100	39485-83-1
2,2',4,5,5'-PeCB[2]	PCB-101	37680-73-2
2,2',4,5,6'-PeCB	PCB-102	68194-06-9
2,2',4,5,'6-PeCB	PCB-103	60145-21-3
2,2',4,6,6'-PeCB	PCB-104	56558-16-8
2,3,3',4,4'-PeCB[2,3]	PCB-105	32598-14-4
2,3,3',4,5-PeCB	PCB-106	70424-69-0
2,3,3',4',5-PeCB	PCB-107	70424-68-9
2,3,3',4,5'-PeCB	PCB-108	70362-41-3
2,3,3',4,6-PeCB	PCB-109	74472-35-8
2,3,3',4',6-PeCB	PCB-110	38380-03-9
2,3,3',5,5'-PeCB	PCB-111	39635-32-0
2,3,3',5,6-PeCB	PCB-112	74472-36-9
2,3,3',5',6-PeCB	PCB-113	68194-10-5
2,3,4,4',5-PeCB[3]	PCB-114	74472-37-0
2,3,4,4',6-PeCB	PCB-115	74472-38-1
2,3,4,5,6-PeCB	PCB-116	18259-05-7
2,3,4',5,6-PeCB	PCB-117	68194-11-6
2,3',4,4',5-PeCB[2,3]	PCB-118	31508-00-6
2,3',4,4',6-PeCB	PCB-119	56558-17-9
2,3',4,5,5'-PeCB	PCB-120	68194-12-7
2,3',4,5,'6-PeCB	PCB-121	56558-18-0
2',3,3',4,5-PeCB	PCB-122	76842-07-4
2',3,4,4',5-PeCB[3]	PCB-123	65510-44-3
2',3,4,5,5'-PeCB	PCB-124	70424-70-3
2',3,4,5,6'-PeCB	PCB-125	74472-39-2
3,3',4,4',5-PeCB[2,3]	PCB-126	57465-28-8
3,3',4,5,5'-PeCB	PCB-127	39635-33-1
2,2',3,3',4,4'-HxCB[2]	PCB-128	38380-07-3
2,2',3,3',4,5-HxCB	PCB-129	55215-18-4
2,2',3,3',4,5'-HxCB	PCB-130	52663-66-8
2,2',3,3',4,6-HxCB	PCB-131	61798-70-7
2,2',3,3',4,6'-HxCB	PCB-132	38380-05-1
2,2',3,3',5,5'-HxCB	PCB-133	35694-04-3
2,2',3,3',5,6-HxCB	PCB-134	52704-70-8
2,2',3,3',5,6'-HxCB	PCB-135	52744-13-5
2,2',3,3',6,6'-HxCB	PCB-136	38411-22-2

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native PCB Congeners

PCB congener[1]	Congener number	CAS Number
2,2',3,4,4',5-HxCB	PCB-137	35694-06-5
2,2',3,4,4',5'-HxCB[2]	PCB-138	35065-28-2
2,2',3,4,4',6-HxCB	PCB-139	56030-56-9
2,2',3,4,4',6'-HxCB	PCB-140	59291-64-4
2,2',3,4,5,5'-HxCB	PCB-141	52712-04-6
2,2',3,4,5,6-HxCB	PCB-142	41411-61-4
2,2',3,4,5,6'-HxCB	PCB-143	68194-15-0
2,2',3,4,5',6-HxCB	PCB-144	68194-14-9
2,2',3,4,6,6'-HxCB	PCB-145	74472-40-5
2,2',3,4',5,5'-HxCB	PCB-146	51908-16-8
2,2',3,4',5,6-HxCB	PCB-147	68194-13-8
2,2',3,4',5,6'-HxCB	PCB-148	74472-41-6
2,2',3,4',5',6-HxCB	PCB-149	38380-04-0
2,2',3,4',6,6'-HxCB	PCB-150	68194-08-1
2,2',3,5,5',6-HxCB	PCB-151	52663-63-5
2,2',3,5,6,6'-HxCB	PCB-152	68194-09-2
2,2',4,4',5,5'-HxCB[2]	PCB-153	35065-27-1
2,2',4,4',5,6’-HxCB	PCB-154	60145-22-4
2,2',4,4',6,6'-HxCB	PCB-155	33979-03-2
2,3,3',4,4',5-HxCB[3]	PCB-156	38380-08-4
2,3,3',4,4',5'-HxCB[3]	PCB-157	69782-90-7
2,3,3',4,4',6-HxCB	PCB-158	74472-42-7
2,3,3',4,5,5'-HxCB	PCB-159	39635-35-3
2,3,3',4,5,6-HxCB	PCB-160	41411-62-5
2,3,3',4,5',6-HxCB	PCB-161	74472-43-8
2,3,3',4',5,5'-HxCB	PCB-162	39635-34-2
2,3,3',4',5,6-HxCB	PCB-163	74472-44-9
2,3,3',4',5',6-HxCB	PCB-164	74472-45-0
2,3,3',5,5',6-HxCB	PCB-165	74472-46-1
2,3,4,4',5,6-HxCB	PCB-166	41411-63-6
2,3',4,4',5,5'-HxCB[3]	PCB-167	52663-72-6
2,3',4,4',5',6-HxCB	PCB-168	59291-65-5
3,3',4,4',5,5'-HxCB[2,3]	PCB-169	32774-16-6
2,2',3,3',4,4',5-HpCB[2]	PCB-170	35065-30-6
2,2'3,3',4,4',6-HpCB	PCB-171	52663-71-5
2,2',3,3',4,5,5'-HpCB	PCB-172	52663-74-8
2,2',3,3',4,5,6-HpCB	PCB-173	68194-16-1
2,2',3,3',4,5,6'-HpCB	PCB-174	38411-25-5
2,2',3,3',4,5',6-HpCB	PCB-175	40186-70-7
2,2',3,3',4,6,6'-HpCB	PCB-176	52663-65-7
2,2',3,3',4',5,6-HpCB	PCB-177	52663-70-4
2,2',3,3',5,5',6-HpCB	PCB-178	52663-67-9
2,2',3,3',5,6,6'-HpCB	PCB-179	52663-64-6
2,2',3,4,4',5,5'-HpCB[2]	PCB-180	35065-29-3
2,2',3,4,4',5,6-HpCB	PCB-181	74472-47-2
2,2',3,4,4',5,6'-HpCB	PCB-182	60145-23-5

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native PCB Congeners

PCB congener[1]	Congener number	CAS Number
2,2',3,4,4',5',6-HpCB	PCB-183	52663-69-1
2,2',3,4,4',6,6'-HpCB	PCB-184	74472-48-3
2,2',3,4,5,5',6-HpCB	PCB-185	52712-05-7
2,2',3,4,5,6,6'-HpCB	PCB-186	74472-49-4
2,2',3,4',5,5',6-HpCB[2]	PCB-187	52663-68-0
2,2',3,4',5,6,6'-HpCB	PCB-188	74487-85-7
2,3,3',4,4',5,5'-HpCB[3]	PCB-189	39635-31-9
2,3,3',4,4',5,6-HpCB	PCB-190	41411-64-7
2,3,3',4,4',5',6-HpCB	PCB-191	74472-50-7
2,3,3',4,5,5',6-HpCB	PCB-192	74472-51-8
2,3,3',4',5,5',6-HpCB	PCB-193	69782-91-8
2,2',3,3',4,4',5,5'-OcCB	PCB-194	35694-08-7
2,2',3,3',4,4',5,6-OcCB[2]	PCB-195	52663-78-2
2,2',3,3',4,4',5,6'-OcCB	PCB-196	42740-50-1
2,2',3,3',4,4',6,6'-OcCB	PCB-197	33091-17-7
2,2',3,3',4,5,5',6-OcCB	PCB-198	68194-17-2
2,2',3,3',4,5,5',6'-OcCB	PCB-199	52663-75-9
2,2',3,3',4,5,6,6'-OcCB	PCB-200	52663-73-7
2,2',3,3',4,5',6,6'-OcCB	PCB-201	40186-71-8
2,2',3,3',5,5',6,6'-OcCB	PCB-202	2136-99-4
2,2',3,4,4',5,5',6-OcCB	PCB-203	52663-76-0
2,2',3,4,4',5,6,6'-OcCB	PCB-204	74472-52-9
2,3,3',4,4',5,5',6-OcCB	PCB-205	74472-53-0
2,2',3,3',4,4',5,5',6-NoCB[2]	PCB-206	40186-72-9
2,2',3,3',4,4',5,6,6'-NoCB	PCB-207	52663-79-3
2,2',3,3',4,5,5',6,6'-NoCB	PCB-208	52663-77-1
DeCB[2]	PCB-209	2051-24-3

1. Abbreviations for chlorination levels (homologs)

MoCB	monochlorobiphenyl	HxCB	hexachlorobiphenyl
DiCB	dichlorobiphenyl	HpCB	heptachlorobiphenyl
TrCB	trichlorobiphenyl	OcCB	octachlorobiphenyl
TeCB	tetrachlorobiphenyl	NoCB	nonachlorobiphenyl
PeCB	pentachlorobiphenyl	DeCB	decachlorobiphenyl

2. National Oceanic and Atmospheric Administration (NOAA) congener of interest (Reference 10)

3. World Health Organization (WHO) toxic congener (Reference 11)

Table 2. Analyte Ions Monitored, Ion Ratios, and RR and RF values Used for Quantitation

Analyte	Typical RT (min)	Q1 (*m/z*)	Q2 (*m/z*)	Expected Ratio Q2/Q1 (%)	Acceptance Limits (%)	RR or RF Used
$^{13}C_{12}$-PCB-8 -NIS	17.46	234	236	65.6	52.5 - 78.7	
$^{13}C_{12}$-PCB-1	13.56	200	202	33.2	26.6 - 39.8	
PCB-1	13.57	188	190	33.2	26.6 - 39.8	
$^{13}C_{12}$-PCB-3	14.97	200	202	33.2	26.6 - 39.8	
PCB-2	14.83	188	190	33.2	26.6 - 39.8	PCB-3
PCB-3	14.98	188	190	33.2	26.6 - 39.8	
$^{13}C_{12}$-PCB-4	15.78	234	236	65.6	52.5 - 78.7	
PCB-4+10	15.79	222	224	65.6	52.5 - 78.7	
$^{13}C_{12}$-PCB-11	18.89	234	236	65.6	52.5 - 78.7	
PCB-7+9	16.77	222	224	65.6	52.5 - 78.7	PCB-11
PCB-6	17.21	222	224	65.6	52.5 - 78.7	PCB-11
PCB-8/5	17.46	222	224	65.6	52.5 - 78.7	
PCB-14	18.05	222	224	65.6	52.5 - 78.7	PCB-11
PCB-11	18.89	222	224	65.6	52.5 - 78.7	
PCB-12+13	19.16	222	224	65.6	52.5 - 78.7	PCB-11
$^{13}C_{12}$-PCB-15	19.41	234	236	65.6	52.5 - 78.7	
PCB-15	19.42	222	224	65.6	52.5 - 78.7	
$^{13}C_{12}$-PCB-19	18.31	268	270	98	78.4 - 117.6	
PCB-19	18.33	256	258	98	78.4 - 117.6	
$^{13}C_{12}$-PCB-28	21.49	268	270	98	78.4 - 117.6	
PCB-30	18.69	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-18	19.33	256	258	98	78.4 - 117.6	
PCB-17	19.41	256	258	98	78.4 - 117.6	PCB-18
PCB-24+27	19.80	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-16+32	20.19	256	258	98	78.4 - 117.6	PCB-18
PCB-34+23	20.65	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-29	20.81	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-26	21.04	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-25	21.15	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-31	21.43	256	258	98	78.4 - 117.6	
PCB-28	21.49	256	258	98	78.4 - 117.6	
PCB-33+20+21	21.95	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-22	22.32	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-36	22.60	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-39	23.02	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-38	23.49	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
PCB-35	23.98	256	258	98	78.4 - 117.6	Average PCBs 31 & 28
$^{13}C_{12}$-PCB-37	24.34	268	270	98	78.4 - 117.6	
PCB-37	24.35	256	258	98	78.4 - 117.6	
$^{13}C_{12}$-PCB-79 -NIS	27.91	304	302	76.7	61.4 - 92.0	
$^{13}C_{12}$-PCB-54	20.81	304	302	76.7	61.4 - 92.0	
PCB-54	20.82	292	290	76.7	61.4 - 92.0	
$^{13}C_{12}$-PCB-52	23.18	304	302	76.7	61.4 - 92.0	
PCB-50	21.44	292	290	76.7	61.4 - 92.0	PCB-52+73
PCB-53	22.01	292	290	76.7	61.4 - 92.0	PCB-52+73
PCB-51	22.24	292	290	76.7	61.4 - 92.0	PCB-52+73

Table 2. Analyte Ions Monitored, Ion Ratios, and RR and RF values Used for Quantitation

Analyte	Typical RT (min)	Q1 (*m/z*)	Q2 (*m/z*)	Expected Ratio Q2/Q1 (%)	Acceptance Limits (%)	RR or RF Used
PCB-45	22.57	292	290	76.7	61.4 - 92.0	PCB-44
PCB-46	22.96	292	290	76.7	61.4 - 92.0	PCB-44
PCB-52+73	23.20	292	290	76.7	61.4 - 92.0	
PCB-49+43	23.36	292	290	76.7	61.4 - 92.0	PCB-44
PCB-47+48+75	23.56	292	290	76.7	61.4 - 92.0	PCB-52+73
PCB-44	24.20	292	290	76.7	61.4 - 92.0	
PCB-42	24.37	292	290	76.7	61.4 - 92.0	PCB-44
PCB-40	25.22	292	290	76.7	61.4 - 92.0	PCB-44
$^{13}C_{12}$-PCB-70	26.18	304	302	76.7	61.4 - 92.0	
PCB-69	23.09	292	290	76.7	61.4 - 92.0	PCB-41+64
PCB-65	23.72	292	290	76.7	61.4 - 92.0	PCB-41+64
PCB-62	23.79	292	290	76.7	61.4 - 92.0	PCB-41+64
PCB-59	24.31	292	290	76.7	61.4 - 92.0	PCB-41+64
PCB-72	24.64	292	290	76.7	61.4 - 92.0	PCB-70
PCB-71	24.76	292	290	76.7	61.4 - 92.0	PCB-41+64
PCB-41+64	24.81	292	290	76.7	61.4 - 92.0	
PCB-68	24.88	292	290	76.7	61.4 - 92.0	PCB-70
PCB-57	25.30	292	290	76.7	61.4 - 92.0	PCB-70
PCB-67	25.52	292	290	76.7	61.4 - 92.0	PCB-70
PCB-58	25.68	292	290	76.7	61.4 - 92.0	PCB-70
PCB-63	25.81	292	290	76.7	61.4 - 92.0	PCB-70
PCB-74+61	26.01	292	290	76.7	61.4 - 92.0	
PCB-70	26.19	292	290	76.7	61.4 - 92.0	
PCB-76	26.25	292	290	76.7	61.4 - 92.0	PCB-70
PCB-66+80	26.35	292	290	76.7	61.4 - 92.0	
PCB-55	26.80	292	290	76.7	61.4 - 92.0	PCB-70
PCB-56+60	27.22	292	290	76.7	61.4 - 92.0	PCB-70
PCB-79	27.92	292	290	76.7	61.4 - 92.0	PCB-70
PCB-78	28.44	292	290	76.7	61.4 - 92.0	PCB-70
$^{13}C_{12}$-PCB-77	29.44	304	302	76.7	61.4 - 92.0	
PCB-81	28.95	292	290	76.7	61.4 - 92.0	PCB-77
PCB-77	29.45	292	290	76.7	61.4 - 92.0	
$^{13}C_{12}$-PCB-162 -NIS	34.59	372	374	81.5	65.2 - 97.8	
$^{13}C_{12}$-PCB-104	23.97	338	340	65.3	52.2 - 78.4	
PCB-104	23.99	326	328	65.3	52.2 - 78.4	
PCB-96	25.04	326	328	65.3	52.2 - 78.4	PCB-104
$^{13}C_{12}$-PCB-101	27.58	338	340	65.3	52.2 - 78.4	
PCB-103	25.25	326	328	65.3	52.2 - 78.4	PCB-95+93
PCB-100	25.53	326	328	65.3	52.2 - 78.4	PCB-95+93
PCB-94	25.94	326	328	65.3	52.2 - 78.4	PCB-95+93
PCB-98+102	26.29	326	328	65.3	52.2 - 78.4	PCB-95+93
PCB-95+93	26.40	326	328	65.3	52.2 - 78.4	
PCB-88+121	26.55	326	328	65.3	52.2 - 78.4	PCB-95+93
PCB-91	26.74	326	328	65.3	52.2 - 78.4	PCB-95+93
PCB-92	27.31	326	328	65.3	52.2 - 78.4	PCB-90+101+89
PCB-84	27.48	326	328	65.3	52.2 - 78.4	PCB-95+93

Table 2. Analyte Ions Monitored, Ion Ratios, and RR and RF values Used for Quantitation

Analyte	Typical RT (min)	Q1 (*m/z*)	Q2 (*m/z*)	Expected Ratio Q2/Q1 (%)	Acceptance Limits (%)	RR or RF Used
PCB-90+101+89	27.60	326	328	65.3	52.2 - 78.4	
PCB-99	27.87	326	328	65.3	52.2 - 78.4	
PCB-83+109	28.43	326	328	65.3	52.2 - 78.4	PCB-90+101+89
PCB-97+86	28.75	326	328	65.3	52.2 - 78.4	PCB-90+101+89
PCB-87+115+116	28.98	326	328	65.3	52.2 - 78.4	PCB-90+101+89
PCB-82	30.04	326	328	65.3	52.2 - 78.4	PCB-90+101+89
$^{13}C_{12}$-PCB-118	30.81	338	340	65.3	52.2 - 78.4	
PCB-113	27.77	326	328	65.3	52.2 - 78.4	PCB-110
PCB-119	28.20	326	328	65.3	52.2 - 78.4	PCB-110
PCB-112	28.30	326	328	65.3	52.2 - 78.4	PCB-110
PCB-125	28.82	326	328	65.3	52.2 - 78.4	PCB-110
PCB-111+117	28.90	326	328	65.3	52.2 - 78.4	PCB-110
PCB-110	29.47	326	328	65.3	52.2 - 78.4	
PCB-124	30.39	326	328	65.3	52.2 - 78.4	PCB-118+106
PCB-107+108	30.57	326	328	65.3	52.2 - 78.4	PCB-118+106
PCB-123	30.70	326	328	65.3	52.2 - 78.4	PCB-118+106
PCB-118+106	30.83	326	328	65.3	52.2 - 78.4	
PCB-114	31.38	326	328	65.3	52.2 - 78.4	PCB-118+106
PCB-122	31.51	326	328	65.3	52.2 - 78.4	PCB-118+106
PCB-105+127	32.18	326	328	65.3	52.2 - 78.4	
$^{13}C_{12}$-PCB-85	29.17	338	340	65.3	52.2 - 78.4	
PCB-85+120	29.17	326	328	65.3	52.2 - 78.4	
$^{13}C_{12}$-PCB-126	33.85	338	340	65.3	52.2 - 78.4	
PCB-126	33.86	326	328	65.3	52.2 - 78.4	
$^{13}C_{12}$-PCB-155	27.11	372	374	81.5	65.2 - 97.8	
PCB-155	27.12	360	362	81.5	65.2 - 97.8	
$^{13}C_{12}$-PCB-153	32.00	372	374	81.5	65.2 - 97.8	
PCB-150	28.22	360	362	81.5	65.2 - 97.8	PCB-153
PCB-152	28.61	360	362	81.5	65.2 - 97.8	PCB-153
PCB-145	28.94	360	362	81.5	65.2 - 97.8	PCB-153
PCB-148	29.16	360	362	81.5	65.2 - 97.8	PCB-147
PCB-136	29.32	360	362	81.5	65.2 - 97.8	PCB-153
PCB-154	29.54	360	362	81.5	65.2 - 97.8	PCB-147
PCB-151	30.12	360	362	81.5	65.2 - 97.8	PCB-147
PCB-144+135	30.35	360	362	81.5	65.2 - 97.8	PCB-147
PCB-147	30.54	360	362	81.5	65.2 - 97.8	
PCB-149+139	30.74	360	362	81.5	65.2 - 97.8	
PCB-140	30.90	360	362	81.5	65.2 - 97.8	PCB-147
PCB-143	31.20	360	362	81.5	65.2 - 97.8	PCB-147
PCB-134	31.27	360	362	81.5	65.2 - 97.8	PCB-132+168
PCB-133	31.38	360	362	81.5	65.2 - 97.8	PCB-132+168
PCB-131+142	31.52	360	362	81.5	65.2 - 97.8	PCB-132+168
PCB-165	31.61	360	362	81.5	65.2 - 97.8	PCB-153
PCB-146	31.70	360	362	81.5	65.2 - 97.8	PCB-153
PCB-161	31.79	360	362	81.5	65.2 - 97.8	PCB-166
PCB-153	32.01	360	362	81.5	65.2 - 97.8	

Table 2. Analyte Ions Monitored, Ion Ratios, and RR and RF values Used for Quantitation

Analyte	Typical RT (min)	Q1 (*m/z*)	Q2 (*m/z*)	Expected Ratio Q2/Q1 (%)	Acceptance Limits (%)	RR or RF Used
PCB-132+168	32.14	360	362	81.5	65.2 - 97.8	
PCB-141	32.66	360	362	81.5	65.2 - 97.8	PCB-132+168
PCB-137	33.01	360	362	81.5	65.2 - 97.8	PCB-132+168
PCB-130	33.15	360	362	81.5	65.2 - 97.8	PCB-132+168
PCB-158+160	33.51	360	362	81.5	65.2 - 97.8	PCB-166
PCB-129	33.83	360	362	81.5	65.2 - 97.8	PCB-132+168
PCB-166	34.16	360	362	81.5	65.2 - 97.8	
PCB-159	34.31	360	362	81.5	65.2 - 97.8	PCB-156
PCB-162	34.60	360	362	81.5	65.2 - 97.8	PCB-156
PCB-128	34.82	360	362	81.5	65.2 - 97.8	PCB-132+168
PCB-167	34.90	360	362	81.5	65.2 - 97.8	PCB-156
PCB-156	36.05	360	362	81.5	65.2 - 97.8	
PCB-157	36.35	360	362	81.5	65.2 - 97.8	PCB-156
$^{13}C_{12}$-PCB-138	33.42	372	374	81.5	65.2 - 97.8	
PCB-138+163+164	33.41	360	362	81.5	65.2 - 97.8	
$^{13}C_{12}$-PCB-169	37.83	372	374	81.5	65.2 - 97.8	
PCB-169	37.83	360	362	81.5	65.2 - 97.8	
$^{13}C_{12}$-PCB-188	31.64	406	408	97.7	78.2 - 117.2	
PCB-188	31.66	394	396	97.7	78.2 - 117.2	
PCB-184	32.01	394	396	97.7	78.2 - 117.2	PCB-188
PCB-179	32.74	394	396	97.7	78.2 - 117.2	PCB-188
PCB-176	33.12	394	396	97.7	78.2 - 117.2	PCB-188
PCB-186	33.57	394	396	97.7	78.2 - 117.2	PCB-188
$^{13}C_{12}$-PCB-180	36.92	406	408	97.7	78.2 - 117.2	
PCB-178	33.89	394	396	97.7	78.2 - 117.2	PCB-177
PCB-175	34.16	394	396	97.7	78.2 - 117.2	PCB-180
PCB-187+182	34.31	394	396	97.7	78.2 - 117.2	
PCB-183	34.57	394	396	97.7	78.2 - 117.2	PCB-180
PCB-185	35.10	394	396	97.7	78.2 - 117.2	PCB-177
PCB-174	35.50	394	396	97.7	78.2 - 117.2	PCB-177
PCB-181	35.57	394	396	97.7	78.2 - 117.2	PCB-180
PCB-177	35.78	394	396	97.7	78.2 - 117.2	
PCB-171	36.01	394	396	97.7	78.2 - 117.2	PCB-177
PCB-173	36.30	394	396	97.7	78.2 - 117.2	PCB-177
PCB-172+192	36.63	394	396	97.7	78.2 - 117.2	PCB-180
PCB-180	36.94	394	396	97.7	78.2 - 117.2	
PCB-170+190	38.16	394	396	97.7	78.2 - 117.2	PCB-180
$^{13}C_{12}$-PCB-189	38.95	406	408	97.7	78.2 - 117.2	
PCB-193	37.08	394	396	97.7	78.2 - 117.2	PCB-189
PCB-191	37.28	394	396	97.7	78.2 - 117.2	PCB-189
PCB-189	38.95	394	396	97.7	78.2 - 117.2	
$^{13}C_{12}$-PCB-202	35.96	442	440	87.8	70.2 - 105.4	
PCB-202	35.97	430	428	87.8	70.2 - 105.4	
PCB-201	36.37	430	428	87.8	70.2 - 105.4	PCB-202
PCB-204	36.46	430	428	87.8	70.2 - 105.4	PCB-202
PCB-197	36.75	430	428	87.8	70.2 - 105.4	PCB-202

Table 2. Analyte Ions Monitored, Ion Ratios, and RR and RF values Used for Quantitation

Analyte	Typical RT (min)	Q1 (*m/z*)	Q2 (*m/z*)	Expected Ratio Q2/Q1 (%)	Acceptance Limits (%)	RR or RF Used
PCB-200	37.51	430	428	87.8	70.2 - 105.4	PCB-202
PCB-198	38.31	430	428	87.8	70.2 - 105.4	PCB-199
PCB-199	38.41	430	428	87.8	70.2 - 105.4	
PCB-196+203	38.56	430	428	87.8	70.2 - 105.4	PCB-199
PCB-195	39.36	430	428	87.8	70.2 - 105.4	PCB-199
PCB-194	39.86	430	428	87.8	70.2 - 105.4	PCB-199
$^{13}C_{12}$-PCB-205	39.99	442	440	87.8	70.2 - 105.4	
PCB-205	40.00	430	428	87.8	70.2 - 105.4	
$^{13}C_{12}$-PCB-208	39.32	476	474	76.9	61.5 - 92.3	
PCB-208	39.32	464	462	76.9	61.5 - 92.3	
PCB-207	39.52	464	462	76.9	61.5 - 92.3	PCB-208
$^{13}C_{12}$-PCB-206	40.77	476	474	76.9	61.5 - 92.3	
PCB-206	40.78	464	462	76.9	61.5 - 92.3	
$^{13}C_{12}$-PCB-209	41.47	510	512	86.7	69.4 - 104.0	
PCB-209	41.47	498	500	86.7	69.4 - 104.0	

Acceptance limits for the ion ratios are a ±20% range around the expected (e.g., theoretical) ratio, rounded to one decimal place.
Target compounds are listed below the labeled standard used for quantification.
No entry in the “RR or RF Used" column indicates that the RR or RF is derived from the compound itself.
RT = Retention Time
Q1 = Quantification ion
Q2 = Confirmation ion
m/z =Mass-to-charge ratio
NIS = Non-extracted internal standard

Table 3. NIS Assignments for Labeled Congener Quantitation

Labeled Congener	NIS
$^{13}C_{12}$-PCB-1	$^{13}C_{12}$-PCB-8
$^{13}C_{12}$-PCB-3	
$^{13}C_{12}$-PCB-4	
$^{13}C_{12}$-PCB-11	
$^{13}C_{12}$-PCB-15	
$^{13}C_{12}$-PCB-19	
$^{13}C_{12}$-PCB-28	
$^{13}C_{12}$-PCB-37	
$^{13}C_{12}$-PCB-52	
$^{13}C_{12}$-PCB-54	$^{13}C_{12}$-PCB-79
$^{13}C_{12}$-PCB-70	
$^{13}C_{12}$-PCB-77	
$^{13}C_{12}$-PCB-85	
$^{13}C_{12}$-PCB-101	
$^{13}C_{12}$-PCB-104	$^{13}C_{12}$-PCB-162
$^{13}C_{12}$-PCB-118	$^{13}C_{12}$-PCB-162
$^{13}C_{12}$-PCB-126	
$^{13}C_{12}$-PCB-138	
$^{13}C_{12}$-PCB-153	
$^{13}C_{12}$-PCB-155	
$^{13}C_{12}$-PCB-169	
$^{13}C_{12}$-PCB-180	
$^{13}C_{12}$-PCB-188	
$^{13}C_{12}$-PCB-189	
$^{13}C_{12}$-PCB-202	
$^{13}C_{12}$-PCB-205	
$^{13}C_{12}$-PCB-206	
$^{13}C_{12}$-PCB-208	
$^{13}C_{12}$-PCB-209	

Table 4. Nominal Concentrations of Standard Solutions (ng/mL)

Analyte	Nominal Conc. in Native Std Solution	Amt Added to Sample (ng)	Analyte	Nominal Conc. in Native Std Solution	Amt Added to Sample(ng)
Native Congeners					
PCB-1	80	16	PCB-105	80	16
PCB-3	80	16	PCB-110	80	16
PCB-4	80	16	PCB-118	80	16
PCB-8	80	16	PCB-126	80	16
PCB-11	80	16	PCB-132	80	16
PCB-15	80	16	PCB-138	80	16
PCB-18	80	16	PCB-147	80	16
PCB-19	80	16	PCB-149	80	16
PCB-28	80	16	PCB-153	80	16
PCB-31	80	16	PCB-155	80	16
PCB-37	80	16	PCB-156	80	16
PCB-44	80	16	PCB-166	80	16
PCB-52	80	16	PCB-169	80	16
PCB-54	80	16	PCB-177	80	16
PCB-64	80	16	PCB-180	80	16
PCB-66	80	16	PCB-187	80	16
PCB-70	80	16	PCB-188	80	16
PCB-74	80	16	PCB-189	80	16
PCB-77	80	16	PCB-199	80	16
PCB-85	80	16	PCB-202	80	16
PCB-95	80	16	PCB-205	80	16
PCB-99	80	16	PCB-206	80	16
PCB-101	80	16	PCB-208	80	16
PCB-104	80	16	PCB-209	80	16
Labeled Congeners					
$^{13}C_{12}$-PCB-1	1250	40	$^{13}C_{12}$-PCB-118	1250	40
$^{13}C_{12}$-PCB-3	1250	40	$^{13}C_{12}$-PCB-126	1250	40
$^{13}C_{12}$-PCB-4	1250	40	$^{13}C_{12}$-PCB-138	1250	40
$^{13}C_{12}$-PCB-11	1250	40	$^{13}C_{12}$-PCB-153	1250	40
$^{13}C_{12}$-PCB-15	1250	40	$^{13}C_{12}$-PCB-155	1250	40
$^{13}C_{12}$ PCB-19	1250	40	$^{13}C_{12}$-PCB-169	1250	40
$^{13}C_{12}$-PCB-28	1250	40	$^{13}C_{12}$-PCB-180	1250	40
$^{13}C_{12}$-PCB-37	1250	40	$^{13}C_{12}$-PCB-188	1250	40
$^{13}C_{12}$-PCB-52	1250	40	$^{13}C_{12}$-PCB-189	1250	40
$^{13}C_{12}$-PCB-54	1250	40	$^{13}C_{12}$-PCB-202	1250	40
$^{13}C_{12}$-PCB-70	1250	40	$^{13}C_{12}$-PCB-205	1250	40
$^{13}C_{12}$-PCB-77	1250	40	$^{13}C_{12}$-PCB-206	1250	40
$^{13}C_{12}$-PCB-85	1250	40	$^{13}C_{12}$-PCB-208	1250	40
$^{13}C_{12}$-PCB-101	1250	40	$^{13}C_{12}$-PCB-209	1250	40
$^{13}C_{12}$-PCB-104	1250	40			
Non-extracted Internal Standards					
$^{13}C_{12}$-PCB-8	1000	40			
$^{13}C_{12}$-PCB-79	1000	40			
$^{13}C_{12}$-PCB-162	1000	40			

Table 5. Composition of Native CB Congener Solutions or Retention Time Determination

Recommended Mixtures[1]								
#1	**#2**	**#3**	**#4**	**#5**	**#6**	**#7**	**#8**	**#9**
AccuStandard Part Numbers								
C-CS-01	**C-CS-02**	**C-CS-03**	**C-CS-04**	**C-CS-05**	**C-CS-06**	**C-CS-07**	**C-CS-08**	**C-CS-09**
PCB-1	PCB-5	PCB-15	PCB-13	PCB-12	PCB-11	PCB-36	PCB-30	PCB-23
PCB-2	PCB-7	PCB-20	PCB-14	PCB-33	PCB-21	PCB-72	PCB-43	PCB-39
PCB-3	PCB-10	PCB-27	PCB-35	PCB-49	PCB-38	PCB-78	PCB-55	PCB-62
PCB-4	PCB-17	PCB-29	PCB-51	PCB-59	PCB-50	PCB-79	PCB-58	PCB-68
PCB-6	PCB-24	PCB-34	PCB-53	PCB-63	PCB-57	PCB-89	PCB-76	PCB-80
PCB-8	PCB-26	PCB-40	PCB-54	PCB-64	PCB-61	PCB-96	PCB-109	PCB-88
PCB-9	PCB-31	PCB-42	PCB-73	PCB-77	PCB-65	PCB-98	PCB-112	PCB-94
PCB-16	PCB-32	PCB-47	PCB-75	PCB-85	PCB-86	PCB-106	PCB-120	PCB-111
PCB-18	PCB-37	PCB-69	PCB-81	PCB-91	PCB-102	PCB-108	PCB-159	PCB-116
PCB-19	PCB-41	PCB-92	PCB-90	PCB-97	PCB-113	PCB-152	PCB-186	PCB-121
PCB-22	PCB-45	PCB-93	PCB-100	PCB-104	PCB-126	PCB-166	PCB-192	PCB-125
PCB-25	PCB-46	PCB-101	PCB-117	PCB-114	PCB-127	PCB-182	PCB-198	PCB-140
PCB-28	PCB-48	PCB-105	PCB-122	PCB-123	PCB-133	PCB-184		PCB-142
PCB-44	PCB-60	PCB-118	PCB-124	PCB-129	PCB-139	PCB-204		PCB-143
PCB-52	PCB-70	PCB-119	PCB-130	PCB-137	PCB-145			PCB-148
PCB-56	PCB-83	PCB-128	PCB-154	PCB-156	PCB-161			PCB-150
PCB-66	PCB-84	PCB-134	PCB-163	PCB-167	PCB-169			PCB-155
PCB-67	PCB-95	PCB-136	PCB-165	PCB-176	PCB-181			PCB-160
PCB-71	PCB-103	PCB-144	PCB-175	PCB-185				PCB-162
PCB-74	PCB-107	PCB-151	PCB-200	PCB-189				PCB-168
PCB-82	PCB-115	PCB-157	PCB-201					PCB-188
PCB-87	PCB-131	PCB-158	PCB-202					
PCB-99	PCB-132	PCB-190						
PCB-110	PCB-135	PCB-191						
PCB-138	PCB-141	PCB-207						
PCB-146	PCB-149	PCB-208						
PCB-147	PCB-164	PCB-209						
PCB-153	PCB-170							
PCB-173	PCB-171							
PCB-174	PCB-172							
PCB-177	PCB-178							
PCB-179	PCB-183							
PCB-180	PCB-193							
PCB-187	PCB-196							
PCB-194	PCB-197							
PCB-195	PCB-205							
PCB-199								
PCB-203								
PCB-206								

[1] See Section 7.12

Table 6. Quantification Reference and Calibration Approach for the 65 Calibrated Congeners

Target Congener	LOC	Quantification Reference	Calibration Approach
PCB-1	Mono	$^{13}C_{12}$-PCB-1	tID
PCB-3		$^{13}C_{12}$-PCB-3	tID
PCB-4 + 10	Di	$^{13}C_{12}$-PCB-4	mID
PCB-5 + 8		$^{13}C_{12}$-PCB-11	EIS
PCB-11		$^{13}C_{12}$-PCB-11	tID
PCB-15		$^{13}C_{12}$-PCB-15	tID
PCB-18	Tri	$^{13}C_{12}$-PCB-28	EIS
PCB-19		$^{13}C_{12}$-PCB-19	tID
PCB-28		$^{13}C_{12}$-PCB-28	tID
PCB-31		$^{13}C_{12}$-PCB-28	EIS
PCB-37		$^{13}C_{12}$-PCB-37	tID
PCB-41 + 64	Tetra	$^{13}C_{12}$-PCB-70	EIS
PCB-44		$^{13}C_{12}$-PCB-52	EIS
PCB-52 + 73		$^{13}C_{12}$-PCB-52	mID
PCB-54		$^{13}C_{12}$-PCB-54	tID
PCB-61 + 74		$^{13}C_{12}$-PCB-70	EIS
PCB-66 + 80		$^{13}C_{12}$-PCB-70	EIS
PCB-70		$^{13}C_{12}$-PCB-70	tID
PCB-77		$^{13}C_{12}$-PCB-77	tID
PCB-85 + 120	Penta	$^{13}C_{12}$-PCB-85	mID
PCB-89 + 90 + 101		$^{13}C_{12}$-PCB-101	mID
PCB-93 + 95		$^{13}C_{12}$-PCB-101	EIS
PCB-99		$^{13}C_{12}$-PCB-101	EIS
PCB-104		$^{13}C_{12}$-PCB-104	tID
PCB-105 + 127		$^{13}C_{12}$-PCB-118	EIS
PCB-106 + 118		$^{13}C_{12}$-PCB-118	mID
PCB-110		$^{13}C_{12}$-PCB-118	EIS
PCB-126		$^{13}C_{12}$-PCB-126	tID
PCB-132 + 168	Hexa	$^{13}C_{12}$-PCB-153	EIS
PCB-138 + 163 + 164		$^{13}C_{12}$-PCB-138	mID
PCB-139 + 149		$^{13}C_{12}$-PCB-153	EIS
PCB-147		$^{13}C_{12}$-PCB-153	EIS
PCB-153		$^{13}C_{12}$-PCB-153	tID
PCB-155		$^{13}C_{12}$-PCB-155	tID
PCB-156		$^{13}C_{12}$-PCB-153	EIS
PCB-166		$^{13}C_{12}$-PCB-153	EIS
PCB-169		$^{13}C_{12}$-PCB-169	tID
PCB-177	Hepta	$^{13}C_{12}$-PCB-180	EIS
PCB-180		$^{13}C_{12}$-PCB-180	tID
PCB-182 + 187		$^{13}C_{12}$-PCB-180	EIS
PCB-188		$^{13}C_{12}$-PCB-188	tID
PCB-189		$^{13}C_{12}$-PCB-189	tID
PCB-199	Octa	$^{13}C_{12}$-PCB-202	EIS
PCB-202		$^{13}C_{12}$-PCB-202	tID

Table 6. Quantification Reference and Calibration Approach for the 65 Calibrated Congeners

Target Congener	LOC	Quantification Reference	Calibration Approach
PCB-205	Octa	$^{13}C_{12}$-PCB-205	tID
PCB-206	Nona	$^{13}C_{12}$-PCB-206	tID
PCB-208		$^{13}C_{12}$-PCB-208	tID
PCB-209	Deca	$^{13}C_{12}$-PCB-209	tID

tID = True isotope dilution quantitation
mID = Modified isotope dilution quantitation
EIS = Extracted internal standard quantitation

Table 7. Calibration Standard Solutions

Analyte	Calibration Standards (ng/mL)						Coeluting Congeners	Calibration Approach
	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6		
Target Congeners								
PCB-1	10	20	40	160	400	2000		tID
PCB-3	10	20	40	160	400	2000		tID
PCB-4	10	20	40	160	400	2000	PCB-10 [1]	mID
PCB-8	10	20	40	160	400	2000	PCB-5 [2]	EIS
PCB-11	10	20	40	160	400	2000		tID
PCB-15	10	20	40	160	400	2000		tID
PCB-18	10	20	40	160	400	2000		EIS
PCB-19	10	20	40	160	400	2000		tID
PCB-28	10	20	40	160	400	2000		tID
PCB-31	10	20	40	160	400	2000		EIS
PCB-37	10	20	40	160	400	2000		tID
PCB-44	10	20	40	160	400	2000		EIS
PCB-52	10	20	40	160	400	2000	PCB-73 [1]	mID
PCB-54	10	20	40	160	400	2000		tID
PCB-64	10	20	40	160	400	2000	PCB-41 [2]	EIS
PCB-66	10	20	40	160	400	2000	PCB-80 [2]	EIS
PCB-70	10	20	40	160	400	2000		tID
PCB-74	10	20	40	160	400	2000	PCB-61 [2]	EIS
PCB-77	10	20	40	160	400	2000		tID
PCB-85	10	20	40	160	400	2000	PCB-120 [1]	mID
PCB-95	10	20	40	160	400	2000	PCB-93 [2]	EIS
PCB-99	10	20	40	160	400	2000		EIS
PCB-101	10	20	40	160	400	2000	PCB-89 [1] + PCB-90 [1]	mID
PCB-104	10	20	40	160	400	2000		tID
PCB-105	10	20	40	160	400	2000	PCB-127 [2]	EIS
PCB-110	10	20	40	160	400	2000		EIS
PCB-118	10	20	40	160	400	2000	PCB-106 [1]	mID
PCB-126	10	20	40	160	400	2000		tID
PCB-132	10	20	40	160	400	2000	PCB-168 [2]	EIS
PCB-138	10	20	40	160	400	2000	PCB-163 [1] + PCB-164 [1]	mID
PCB-147	10	20	40	160	400	2000		EIS
PCB-149	10	20	40	160	400	2000	PCB-139 [2]	EIS
PCB-153	10	20	40	160	400	2000		tID
PCB-155	10	20	40	160	400	2000		tID
PCB-156	10	20	40	160	400	2000		EIS
PCB-166	10	20	40	160	400	2000		EIS
PCB-169	10	20	40	160	400	2000		tID
PCB-177	10	20	40	160	400	2000		EIS
PCB-180	10	20	40	160	400	2000		tID
PCB-187	10	20	40	160	400	2000	PCB-182 [2]	EIS
PCB-188	10	20	40	160	400	2000		tID
PCB-189	10	20	40	160	400	2000		tID
PCB-199	10	20	40	160	400	2000		EIS
PCB-202	10	20	40	160	400	2000		tID
PCB-205	10	20	40	160	400	2000		tID
PCB-206	10	20	40	160	400	2000		tID
PCB-208	10	20	40	160	400	2000		tID
PCB-209	10	20	40	160	400	2000		tID

Table 7. Calibration Standard Solutions

Analyte	Calibration Standards (ng/mL)						Coeluting Congeners	Calibration Approach
	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6		
Labeled Congeners								
$^{13}C_{12}$-PCB-1	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-3	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-4	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-11	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-15	400	400	400	400	400	400		NIS
$^{13}C_{12}$ PCB-19	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-28	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-37	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-52	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-54	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-70	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-77	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-85	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-101	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-104	400	400	400	400	400	400	NA	NIS
$^{13}C_{12}$-PCB-118	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-126	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-138	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-153	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-155	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-169	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-180	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-188	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-189	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-202	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-205	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-206	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-208	400	400	400	400	400	400		NIS
$^{13}C_{12}$-PCB-209	400	400	400	400	400	400		NIS
Non-extracted Internal Standards								
$^{13}C_{12}$-PCB-8	400	400	400	400	400	400		NA
$^{13}C_{12}$-PCB-79	400	400	400	400	400	400		NA
$^{13}C_{12}$-PCB-162	400	400	400	400	400	400		NA

[1] These coeluting congeners are not included in the calibration standard, but the responses in the samples for all of the congeners that elute together at a given retention time are quantified by modified isotope dilution, based on the response ratio derived for the single congener in the calibration standard and its corresponding labeled analog.

[2] These coeluting congeners are not included in the calibration standard, but the responses in the samples for all of the congeners that elute together at a given retention time are quantified by extracted internal standard, based on the response factor derived for the single congener in the calibration standard and the labeled analog for another congener in the same level of chlorination.

tID = True isotope dilution quantitation
mID = Modified isotope dilution quantitation
EIS = Extracted internal standard quantitation
NIS = Non-extracted internal standard quantitation
NA = Not applicable - these congeners are not quantified

Table 8. IPR and OPR QC Interim Acceptance Criteria for Target Analytes by Matrix Type

	Aqueous Matrix			Solid Matrix			Tissue Matrix		
Congener	IPR Mean Rec (%)	RSD (%)	OPR Rec (%)	IPR Mean Rec (%)	RSD (%)	OPR Rec (%)	IPR Mean Rec (%)	RSD (%)	OPR Rec (%)
PCB-1	78- 130	18	71 - 136	61 - 154	59	19 - 196	25 - 150	25	25 - 150
PCB-3	74- 117	14	71 - 120	40 - 156	42	29 - 167	25 - 150	25	25 - 150
PCB-4	77- 112	14	72 - 117	48 - 144	61	13 - 179	25 - 150	25	25 - 150
PCB-8	42- 120	18	43 - 119	35 - 150	40	25 - 160	25 - 150	25	25 - 150
PCB-11	62- 125	9	63 - 124	35 - 150	40	25 - 160	25 - 150	25	25 - 150
PCB-15	70- 111	10	69 - 111	36 - 150	44	25 - 162	25 - 150	25	25 - 150
PCB-18	60- 107	17	57 - 111	20 - 148	40	18 - 149	25 - 150	25	25 - 150
PCB-19	77- 107	12	73 - 111	26 - 157	32	28 - 156	25 - 150	25	25 - 150
PCB-28	18- 184	17	21 - 180	25 - 150	35	30 - 150	25 - 150	25	25 - 150
PCB-31	46- 129	19	46 - 129	38 - 147	37	32 - 153	25 - 150	25	25 - 150
PCB-37	67- 112	9	68 - 111	38 - 147	38	31 - 155	25 - 150	25	25 - 150
PCB-44	44- 131	13	46 - 130	23 - 153	34	24 - 151	25 - 150	25	25 - 150
PCB-52	61- 128	8	62 - 127	57 - 138	42	36 - 159	25 - 150	25	25 - 150
PCB-54	67- 112	8	68 - 111	56 - 132	56	21 - 167	25 - 150	25	25 - 150
PCB-64	74- 108	10	73 - 110	29 - 153	29	31 - 150	25 - 150	25	25 - 150
PCB-66	64- 118	8	65 - 117	50 - 138	25	49 - 140	25 - 150	25	25 - 150
PCB-70	55- 127	8	57 - 126	43 - 144	27	42 - 144	25 - 150	25	25 - 150
PCB-74	74- 102	8	73 - 103	41 - 135	30	38 - 138	25 - 150	25	25 - 150
PCB-77	58- 118	9	59 - 116	42 - 134	40	30 - 145	25 - 150	25	25 - 150
PCB-85	68- 106	7	69 - 105	57 - 121	27	50 - 128	25 - 150	25	25 - 150
PCB-95	63- 117	12	63 - 117	55 - 125	29	47 - 133	25 - 150	25	25 - 150
PCB-99	66- 107	10	66 - 107	33 - 140	34	30 - 143	25 - 150	25	25 - 150
PCB-101	64- 118	9	65 - 117	57 - 125	26	51 - 132	25 - 150	25	25 - 150
PCB-104	64- 117	8	65 - 116	52 - 128	44	32 - 148	25 - 150	25	25 - 150
PCB-105	64- 120	10	65 - 119	65 - 122	17	63 - 124	25 - 150	25	25 - 150
PCB-118	61- 119	10	62 - 118	48 - 133	19	50 - 131	25 - 150	25	25 - 150
PCB-110	63- 106	12	62 - 107	31 - 142	20	36 - 137	25 - 150	25	38 - 138
PCB-126	63- 113	7	64 - 112	48 - 129	14	52 - 124	25 - 150	25	38 - 139
PCB-132	51- 133	11	53 - 131	42 - 146	18	47 - 141	32 - 156	25	48 - 140
PCB-138	61- 117	11	61 - 116	60 - 123	19	58 - 125	27 - 150	25	43 - 134
PCB-147	61- 117	12	62 - 117	58 - 126	25	53 - 132	25 - 150	25	25 - 150
PCB-149	57- 120	11	58 - 119	51 - 129	28	46 - 134	25 - 150	25	25 - 150
PCB-153	46- 134	16	48 - 132	76 - 109	25	61 - 124	33 - 142	25	47 - 127
PCB-155	64- 116	10	65 - 115	60 - 122	37	41 - 140	25 - 150	25	25 - 150
PCB-156	46- 149	23	45 - 150	76 - 119	25	62 - 133	25 - 150	25	25 - 150
PCB-166	34- 157	9	36 - 156	71 - 122	21	64 - 128	25 - 150	25	25 - 150
PCB-169	50- 122	10	52 - 121	56 - 130	55	23 - 164	25 - 150	25	25 - 150
PCB-177	47- 130	10	49 - 128	71 - 114	29	55 - 130	25 - 150	25	25 - 150
PCB-180	52- 124	11	53 - 123	72 - 112	25	58 - 125	42 - 137	25	52 - 127
PCB-187	36- 138	17	38 - 136	64 - 114	23	56 - 122	39 - 137	25	49 - 126
PCB-188	57- 122	11	58 - 121	61 - 118	27	52 - 128	25 - 150	25	25 - 150
PCB-189	56- 119	11	58 - 118	67 - 117	24	58 - 126	25 - 150	25	25 - 150
PCB-199	42- 164	57	14 - 193	62 - 126	22	58 - 130	34 - 153	25	45 - 142
PCB-202	55- 121	12	56 - 120	51 - 127	24	49 - 129	33 - 145	25	47 - 131
PCB-205	52- 118	18	51 - 119	54 - 116	31	44 - 126	25 - 150	25	37 - 144
PCB-206	35- 135	17	37 - 133	52 - 129	49	27 - 154	25 - 150	25	25 - 150
PCB-208	44- 125	15	45 - 124	45 - 131	21	47 - 129	25 - 150	25	25 - 150
PCB-209	31- 130	27	30 - 131	67 - 111	19	62 - 117	25 - 150	25	25 - 150

Table 9. IPR and OPR QC Interim Acceptance Criteria for Labeled Congeners by Matrix Type

	Aqueous Matrix			Solid Matrix			Tissue Matrix		
Congener	**IPR Aliquot Rec (%)**	**RSD (%)**	**OPR Rec (%)**	**IPR Aliquot Rec (%)**	**RSD (%)**	**OPR Rec (%)**	**IPR Aliquot Rec (%)**	**RSD (%)**	**OPR Rec (%)**
$^{13}C_{12}$-PCB-1	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-3	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-4	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-11	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-15	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-19	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-28	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-37	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-52	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-54	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-70	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-77	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-85	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-101	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-104	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-118	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-126	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-138	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-153	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-155	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-169	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-180	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-188	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-189	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-202	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-205	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-206	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-208	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130
$^{13}C_{12}$-PCB-209	15 - 130	40	15 - 130	15 - 130	60	15 -130	15 - 130	60	15 -130

[1] The recovery limits are applied to all samples, method blanks, IPR, OPR samples for all matrix types. Up to three labeled compounds may fail the acceptance recovery limits; however, the recoveries of the failing compounds should not be < 5% (see Section 9.3.3)

Table 10. QC Acceptance Criteria for Recovery of Labeled Congeners in Field Samples

Congener	Interim QC Acceptance Criteria (%)[1]
$^{13}C_{12}$-PCB-1	15 - 130
$^{13}C_{12}$-PCB-3	15 - 130
$^{13}C_{12}$-PCB-4	15 - 130
$^{13}C_{12}$-PCB-11	15 - 130
$^{13}C_{12}$-PCB-15	15 - 130
$^{13}C_{12}$-PCB-19	15 - 130
$^{13}C_{12}$-PCB-28	15 - 130
$^{13}C_{12}$-PCB-37	15 - 130
$^{13}C_{12}$-PCB-52	15 - 130
$^{13}C_{12}$-PCB-54	15 - 130
$^{13}C_{12}$-PCB-70	15 - 130
$^{13}C_{12}$-PCB-77	15 - 130
$^{13}C_{12}$-PCB-85	15 - 130
$^{13}C_{12}$-PCB-101	15 - 130
$^{13}C_{12}$-PCB-104	15 - 130
$^{13}C_{12}$-PCB-118	15 - 130
$^{13}C_{12}$-PCB-126	15 - 130
$^{13}C_{12}$-PCB-138	15 - 130
$^{13}C_{12}$-PCB-153	15 - 130
$^{13}C_{12}$-PCB-155	15 - 130
$^{13}C_{12}$-PCB-169	15 - 130
$^{13}C_{12}$-PCB-180	15 - 130
$^{13}C_{12}$-PCB-188	15 - 130
$^{13}C_{12}$-PCB-189	15 - 130
$^{13}C_{12}$-PCB-202	15 - 130
$^{13}C_{12}$-PCB-205	15 - 130
$^{13}C_{12}$-PCB-206	15 - 130
$^{13}C_{12}$-PCB-208	15 - 130
$^{13}C_{12}$-PCB-209	15 - 130

[1] The recovery limits are applied to all field samples for all matrix types. Up to three labeled compounds may fail the acceptance recovery limits; however, the recoveries of the failing compounds should not be < 5% (see Section 9.3.3).

Table 11. Ions Monitored for Correcting for Interferences from Higher Homologues

Congener	Q1 of Congener	HH Interference	Q1 of HH interference	Correction Factor[1]
PCB-35	256	PCB-104	326	0.497
PCB-77	292	PCB-110	326	0.057
PCB-81	292	PCB-87+115+116	326	0.014
PCB 123	326	PCB149+139	360	0.046
PCB-126	326	PCB-178	394	0.512
PCB-157	360	PCB-201	430	0.451

[1] These correction factors were experimentally derived using the method-specified GC column and conditions, and as noted in Section 16.3, use of another GC column or other chromatographic conditions will require that the laboratory determine correction factors specific to that other column and conditions. See Section 16.3 for details of their derivation. These factors are applied to the responses for the quantification ions (e.g., the area counts) and they are presented to 3 decimal places here, recognizing that not all of those digits will be "significant" in the final calculation of the analyte concentration present in a sample.

Table 12. Pooled MDL_s and ML values from the Validation Study by Matrix[1]

	Aqueous (ng/L)		Solid (ng/g)		Tissue (ng/g)	
Congener	**MDL_s**	**ML**	**MDL_s**	**ML**	**MDL_s**	**ML**
PCB-1	1.75	5	0.63	2	0.11	0.2
PCB-2	0.71	2	0.06	0.2	0.13	0.5
PCB-3	0.69	2	0.10	0.2	0.11	0.2
PCB-4+10	1.90	5	0.15	0.5	0.23	0.5
PCB-8+5	1.00	2	0.22	0.5	0.18	0.5
PCB-6	0.57	2	0.09	0.2	0.10	0.2
PCB-7+9	1.17	5	0.24	1	0.22	0.5
PCB-11	0.72	2	0.42	1	0.06	0.2
PCB-12+13	1.11	5	0.21	0.5	0.13	0.5
PCB-14	0.64	2	0.11	0.2	0.07	0.2
PCB-15	0.44	1	0.09	0.2	0.06	0.2
PCB-16+32	0.80	2	0.14	0.5	0.18	0.5
PCB-17	0.49	2	0.07	0.2	0.08	0.2
PCB-18	0.46	1	0.07	0.2	0.09	0.2
PCB-19	0.63	2	0.08	0.2	0.07	0.2
PCB-33+20+21	1.11	5	0.30	1	0.20	0.5
PCB-22	0.39	1	0.08	0.2	0.10	0.2
PCB-34+23	1.00	2	0.11	0.2	0.13	0.5
PCB-24+27	0.64	2	0.09	0.2	0.11	0.5
PCB-25	0.46	1	0.08	0.2	0.08	0.2
PCB-26	0.43	1	0.09	0.2	0.07	0.2
PCB-28	0.69	2	0.15	0.5	0.14	0.5
PCB-29	0.49	2	0.06	0.2	0.08	0.2
PCB-30	0.61	2	0.08	0.2	0.08	0.2
PCB-31	0.50	2	0.07	0.2	0.09	0.2
PCB-35	0.89	2	0.21	0.5	0.14	0.5
PCB-36	0.54	2	0.17	0.5	0.10	0.2
PCB-37	0.44	1	0.18	0.5	0.12	0.5
PCB-38	1.66	5	0.14	0.5	0.13	0.5
PCB-39	0.53	2	0.10	0.2	0.06	0.2
PCB-40	1.12	5	0.16	0.5	0.13	0.5
PCB-41+64	0.97	2	0.17	0.5	0.15	0.5
PCB-42	0.73	2	0.10	0.2	0.09	0.2
PCB-49+43	1.06	2	0.24	1	0.22	0.5
PCB-44	0.40	1	0.11	0.5	0.09	0.2
PCB-45	0.31	1	0.09	0.2	0.07	0.2
PCB-46	0.36	1	0.06	0.2	0.07	0.2
PCB-47+48+75	1.71	5	0.24	1	0.23	0.5
PCB-50	0.58	2	0.07	0.2	0.07	0.2
PCB-51	0.48	2	0.06	0.2	0.07	0.2
PCB-52+73	0.97	2	0.17	0.5	0.24	1.0
PCB-53	0.33	1	0.05	0.2	0.05	0.2
PCB-54	0.58	2	0.06	0.2	0.06	0.2
PCB-55	0.39	1	0.08	0.2	0.10	0.2
PCB-56+60	0.74	2	0.13	0.5	0.09	0.2
PCB-57	0.47	1	0.10	0.2	0.07	0.2

Table 12. Pooled MDL_s and ML values from the Validation Study by Matrix[1]

	Aqueous (ng/L)		Solid (ng/g)		Tissue (ng/g)	
Congener	**MDL_s**	**ML**	**MDL_s**	**ML**	**MDL_s**	**ML**
PCB-58	0.46	1	0.11	0.5	0.09	0.2
PCB-59	0.60	2	0.07	0.2	0.08	0.2
PCB-74+61	0.96	2	0.14	0.5	0.12	0.5
PCB-62	0.49	2	0.11	0.5	0.06	0.2
PCB-63	0.38	1	0.08	0.2	0.08	0.2
PCB-65	0.57	2	0.10	0.2	0.07	0.2
PCB-66+80	0.91	2	0.19	0.5	0.16	0.5
PCB-67	0.45	1	0.11	0.2	0.07	0.2
PCB-68	0.66	2	0.16	0.5	0.10	0.2
PCB-69	0.53	2	0.12	0.5	0.06	0.2
PCB-70	1.32	5	0.08	0.2	0.09	0.2
PCB-71	1.09	2	0.14	0.5	0.07	0.2
PCB-72	0.50	2	0.10	0.2	0.11	0.2
PCB-76	0.53	2	0.11	0.2	0.08	0.2
PCB-77	0.50	2	0.07	0.2	0.09	0.2
PCB-78	0.51	2	0.10	0.2	0.11	0.5
PCB-79	0.48	2	0.08	0.2	0.06	0.2
PCB-81	0.49	2	0.09	0.2	0.07	0.2
PCB-82	0.61	2	0.06	0.2	0.08	0.2
PCB-83+109	0.76	2	0.14	0.5	0.10	0.2
PCB-84	2.53	10	0.07	0.2	0.06	0.2
PCB-85+120	1.19	5	0.15	0.5	0.17	0.5
PCB-97+86	1.67	5	0.11	0.2	0.07	0.2
PCB-87+115+116	2.23	5	0.37	1	0.22	0.5
PCB-88+121	0.93	2	0.12	0.5	0.13	0.5
PCB-90+101+89	3.36	10	0.24	1	0.10	0.2
PCB-91	0.39	1	0.05	0.2	0.05	0.2
PCB-92	0.53	2	0.06	0.2	0.05	0.2
PCB-95+93	2.01	5	0.12	0.5	0.10	0.2
PCB-94	0.32	1	0.06	0.2	0.03	0.1
PCB-96	0.37	1	0.06	0.2	0.05	0.1
PCB-98+102	0.77	2	0.12	0.5	0.12	0.5
PCB-99	1.30	5	0.10	0.2	0.06	0.2
PCB-100	0.50	2	0.17	0.5	0.06	0.2
PCB-103	0.48	2	0.15	0.5	0.06	0.2
PCB-104	0.51	2	0.05	0.2	0.05	0.2
PCB-105+127	1.23	5	0.19	0.5	0.14	0.5
PCB-118+106	3.21	10	0.39	1	0.12	0.5
PCB-107+108	0.86	2	0.16	0.5	0.13	0.5
PCB-110	3.94	10	0.31	1	0.06	0.2
PCB-111+117	1.33	5	0.21	0.5	0.16	0.5
PCB-112	0.34	1	0.09	0.2	0.06	0.2
PCB-113	0.34	1	0.08	0.2	0.04	0.1
PCB-114	0.28	1	0.06	0.2	0.07	0.2
PCB-119	0.42	1	0.08	0.2	0.09	0.2
PCB-122	0.19	0.5	0.07	0.2	0.05	0.2

Table 12. Pooled MDL_s and ML values from the Validation Study by Matrix[1]

	Aqueous (ng/L)		Solid (ng/g)		Tissue (ng/g)	
Congener	**MDL_s**	**ML**	**MDL_s**	**ML**	**MDL_s**	**ML**
PCB-123	0.31	1	0.09	0.2	0.06	0.2
PCB-124	0.35	1	0.08	0.2	0.06	0.2
PCB-125	0.81	2	0.07	0.2	0.05	0.2
PCB-126	0.42	1	0.07	0.2	0.10	0.2
PCB-128	1.27	5	0.08	0.2	0.08	0.2
PCB-129	0.33	1	0.07	0.2	0.08	0.2
PCB-130	0.35	1	0.07	0.2	0.06	0.2
PCB-131+142	1.46	5	0.10	0.2	0.19	0.5
PCB-132+168	1.91	5	0.18	0.5	0.14	0.5
PCB-133	0.39	1	0.07	0.2	0.07	0.2
PCB-134	0.75	2	0.08	0.2	0.06	0.2
PCB-144+135	1.26	5	0.19	0.5	0.11	0.5
PCB-136	1.39	5	0.06	0.2	0.05	0.1
PCB-137	0.38	1	0.08	0.2	0.06	0.2
PCB-138+163+164	3.95	10	0.34	1	0.17	0.5
PCB-149+139	4.98	20	0.20	0.5	0.12	0.5
PCB-140	4.00	10	0.06	0.2	0.06	0.2
PCB-141	1.35	5	0.09	0.2	0.07	0.2
PCB-143	0.40	1	0.07	0.2	0.07	0.2
PCB-145	0.43	1	0.08	0.2	0.06	0.2
PCB-146	0.57	2	0.07	0.2	0.04	0.1
PCB-147	0.30	1	0.08	0.2	0.07	0.2
PCB-148	0.44	1	0.07	0.2	0.06	0.2
PCB-150	0.46	1	0.07	0.2	0.11	0.2
PCB-151	1.97	5	0.08	0.2	0.05	0.2
PCB-152	0.50	2	0.07	0.2	0.06	0.2
PCB-153	3.90	10	0.20	0.5	0.09	0.2
PCB-154	0.42	1	0.08	0.2	0.06	0.2
PCB-155	0.43	1	0.05	0.1	0.05	0.2
PCB-156	0.37	1	0.06	0.2	0.07	0.2
PCB-157	0.60	2	0.07	0.2	0.08	0.2
PCB-158+160	0.73	2	0.12	0.5	0.13	0.5
PCB-159	0.51	2	0.06	0.2	0.06	0.2
PCB-161	0.43	1	0.07	0.2	0.07	0.2
PCB-162	0.60	2	0.06	0.2	0.05	0.2
PCB-165	1.51	5	0.07	0.2	0.04	0.1
PCB-166	0.37	1	0.08	0.2	0.09	0.2
PCB-167	0.94	2	0.06	0.2	0.06	0.2
PCB-169	0.34	1	0.10	0.2	0.06	0.2
PCB-170+190	1.95	5	0.14	0.5	0.15	0.5
PCB-171	0.60	2	0.07	0.2	0.13	0.5
PCB-172+192	0.59	2	0.13	0.5	0.12	0.5
PCB-173	0.33	1	0.07	0.2	0.21	0.5
PCB-174	3.12	10	0.09	0.2	0.08	0.2
PCB-175	0.33	1	0.07	0.2	0.08	0.2
PCB-176	0.56	2	0.06	0.2	0.07	0.2

Table 12. Pooled MDL_s and ML values from the Validation Study by Matrix[1]

	Aqueous (ng/L)		Solid (ng/g)		Tissue (ng/g)	
Congener	**MDL_s**	**ML**	**MDL_s**	**ML**	**MDL_s**	**ML**
PCB-177	1.57	5	0.07	0.2	0.06	0.2
PCB-178	0.50	2	0.09	0.2	0.09	0.2
PCB-179	1.54	5	0.05	0.2	0.05	0.2
PCB-180	0.37	1	0.07	0.2	0.19	0.5
PCB-181	3.10	10	0.07	0.2	0.08	0.2
PCB-187+182	2.28	5	0.15	0.5	0.13	0.5
PCB-183	0.92	2	0.08	0.2	0.07	0.2
PCB-184	0.49	2	0.05	0.2	0.06	0.2
PCB-185	0.36	1	0.07	0.2	0.06	0.2
PCB-186	0.35	1	0.07	0.2	0.05	0.2
PCB-188	0.39	1	0.06	0.2	0.05	0.2
PCB-189	0.26	1	0.06	0.2	0.07	0.2
PCB-191	0.22	0.5	0.07	0.2	0.05	0.2
PCB-193	0.39	1	0.07	0.2	0.06	0.2
PCB-194	3.16	10	0.18	0.5	0.10	0.2
PCB-195	0.43	1	0.07	0.2	0.08	0.2
PCB-196+203	1.13	5	0.15	0.5	0.17	0.5
PCB-197	0.43	1	0.06	0.2	0.04	0.1
PCB-198	0.80	2	0.10	0.2	0.06	0.2
PCB-199	0.88	2	0.08	0.2	0.10	0.2
PCB-200	0.44	1	0.06	0.2	0.05	0.2
PCB-201	0.59	2	0.06	0.2	0.14	0.5
PCB-202	0.26	1	0.05	0.2	0.05	0.2
PCB-204	0.65	2	0.07	0.2	0.09	0.2
PCB-205	0.75	2	0.06	0.2	0.11	0.5
PCB-206	0.64	2	0.06	0.2	0.06	0.2
PCB-207	0.62	2	0.06	0.2	0.06	0.2
PCB-208	0.90	2	0.05	0.2	0.05	0.2
PCB-209	0.50	2	0.26	1	0.09	0.2

[1] MDL_s and ML values are the pooled results generated during the interlaboratory method validation study (see Reference 13). Pooled MDL values do not include MDL_b results.

22.0 Figures

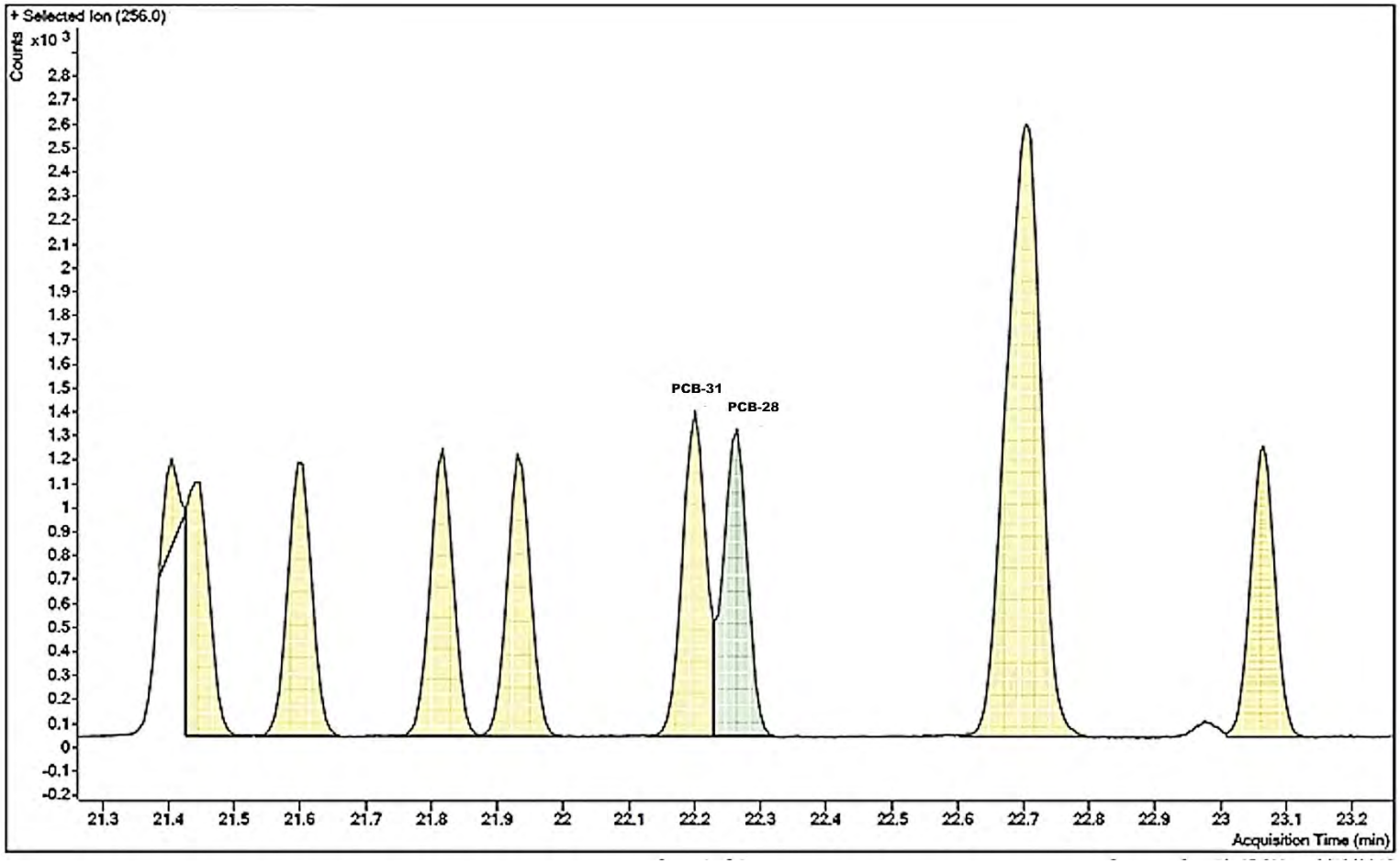


Figure 1. Example of GC Resolution for PCB-28 and PCB-31 using DB-5 Chromatographic Column and the Instrument Conditions in Section 10.1.3

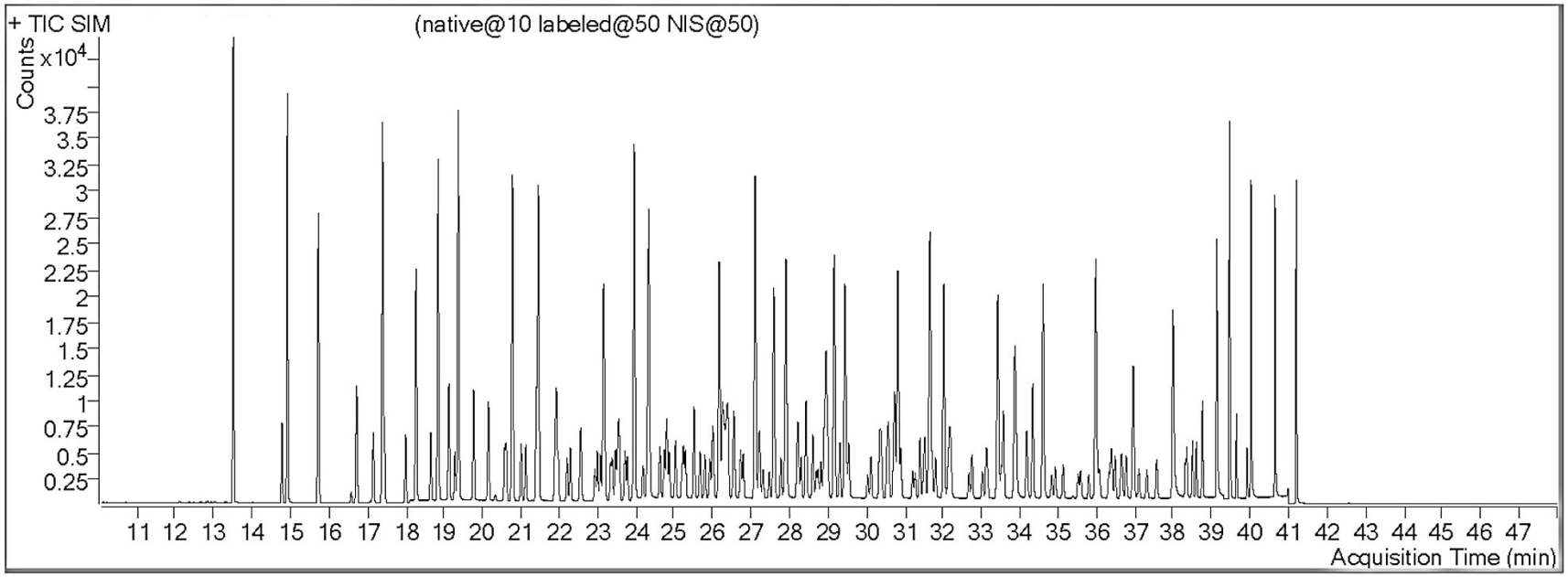


Figure 2. Example Total Ion Chromatogram of 209 PCB Congeners using DB-5 Chromatographic Column and the Instrument Conditions in Section 10.1.3

23.0 Glossary

The following definitions and purposes are specific to this method but have been conformed to common usage to the extent possible.

23.1 Units of weight and measure and their abbreviations

23.1.1 Symbols

Å	Angstrom (1×10^{-10} m)
ºC	degrees Celsius
Da	Dalton (same as atomic mass unit)
µg	microgram
µL	microliter
µm	micrometer
<	less than
≤	less than or equal
>	greater than
≥	greater than or equal
%	percent
±	plus or minus

23.1.2 Alphabetical abbreviations

amu	atomic mass units
cm	centimeter
eV	electron Volt
g	gram
h	hour
ID	inside diameter
L	liter
M	molar
m	meter
m^2	square meter(s)
mg	milligram
min	minute
mL	milliliter
mm	millimeter
m/z	mass-to-charge ratio
OD	outside diameter
ng	nanogram
psig	pound-per-square-inch gauge
Q1	quantitation ion
Q2	confirmation ion
rpm	revolutions per minute

23.2 Definitions and acronyms (in alphabetical order)

Analyte – A PCB congener, or group of coeluting congeners, tested for by this method. The analytes are listed in Table 1.

Calibration standard (CS) – A solution prepared from a secondary standard and/or stock solutions and used to calibrate the response of the GC/MS instrument.
Calibration verification standard (CV) – The mid-point calibration standard (CS-4) that is used to verify calibration. See Table 7.

CFR – Code of Federal Regulations

Congener - One of many variants or configurations of a common chemical structure. Individual chlorobiphenyl congeners are identified by the number and position of the chlorine atoms around the biphenyl ring.

Class A glassware – Volumetric glassware that provides the highest accuracy. Class A volumetric glassware complies with the Class A tolerances defined in ASTM E694, must be permanently labeled as Class A, and is supplied with a serialized certificate of precision.

CWA – Clean Water Act

DeCB – Decachlorobiphenyl (PCB 209)

DiCB – Dichlorobiphenyl

DVB – Divinylbenzene

Extracted internal standard (EIS) quantification – The response of the target congener is compared to the response of the $^{13}C_{12}$-labeled analog of another congener in the same LOC.

GC – Gas chromatograph or gas chromatography

GPC – Gel permeation chromatograph or gel permeation chromatography

Homolog – A group of PCB congeners that contain the same number of chlorine atoms. Synonymous with level of chlorination, or LOC

HpCB – Heptachlorobiphenyl

HxCB – Hexachlorobiphenyl

Interim QC acceptance criteria – For the purposes of this method, these are performance criteria developed from the multi-laboratory validation study using an approach that strikes a balance between statistically based QC acceptance criteria that are sometimes seen as overly wide, and common consensus-style criteria that may lead to high failure rates when applied across the participant laboratories. That approach examined the failure rates at various upper and lower acceptance limits and established the interim criteria wide enough to yield reasonable failure rates for the observed results from the number of laboratories in the validation study. The interim criteria are to be used by laboratories until such time as they have collected enough results to support development of in-house performance criteria, which by virtue of being from a single laboratory, will be narrower than the interim acceptance criteria listed in this method.

Internal standard – A labeled compound used as a reference for quantitation of other labeled compounds and for quantitation of native PCB congeners other than the congener of which it is a labeled analog. See Internal standard quantitation.

Internal standard quantitation – A means of determining the concentration of (1) a naturally occurring (native) compound by reference to a compound other than its labeled analog and (2) a labeled compound by reference to another labeled compound

IPR – Initial precision and recovery; four aliquots of a reference matrix spiked with the analytes of interest and labeled compounds and analyzed to establish the ability of the laboratory to generate acceptable precision and recovery. An IPR is performed prior to the first time this method is used and any time the method or instrumentation is modified.

Isotope dilution quantitation - See true isotope dilution quantitation.

Keeper – A solvent with a high boiling point, added in small quantities during macro-concentration of a sample extract. The keeper is used to minimize the risk that the extract will be taken to dryness and to retain the analytes of interest.

LOC – Level of chlorination; indicates the number of chlorine atoms that are substituted on the biphenyl ring of the PCB. Synonymous with homolog.

LRMS – Low resolution mass spectrometry

Mass accuracy – A metric describing the difference between the measured mass/charge of an ion and the real or exact mass/charge of that ion.

Mass resolution – The ability of separating two narrow mass spectral peaks. This is also referred to as "tuning" or the "tune check" in this method

May – This action, activity, or procedural step is neither required nor prohibited.

May not – This action, activity, or procedural step is prohibited.

Method blank – An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and labeled compounds that are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

Method Detection Limit (MDL) – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is distinguishable from method blank results (40 CFR 136, Appendix B).

MESA – Mining Enforcement and Safety Administration

Minimum level of quantitation (ML) – The term "minimum level" refers to either the sample concentration equivalent to the lowest calibration point in a method or a multiple of the method detection limit (MDL), whichever is higher. Minimum levels may be obtained in several ways: They may be published in a method; they may be based on the lowest acceptable calibration point used by a laboratory; or they may be calculated by multiplying the MDL in a method, or the MDL determined by a laboratory, by a factor of 3. For the purposes of NPDES compliance monitoring, EPA considers the following terms to be synonymous: "quantitation limit," "reporting limit," and "minimum level."

MoCB – Monochlorobiphenyl

Modified isotope dilution (mID) quantification –- Based on the response ratio derived for the single congener (of a coeluting congener) in the calibration standard and its corresponding labeled analog.

MS – Mass spectrometer or mass spectrometry

Multiple ion monitoring (MIM) – Also known as selected ion monitoring (SIM). A type of mass spectrometry where the intensities of multiple specific ion beams are recorded rather than the entire mass spectrum.

Must – This action, activity, or procedural step is required.

NIOSH – The National Institute of Occupational Safety and Health

NoCB – Nonachlorobiphenyl

Non-extracted internal standard (NIS) – Three $^{13}C_{12}$-labeled PCB congeners spiked into the concentrated extract immediately prior to injection of an aliquot of the extract into the GC/MS. The three non-extracted internal standards in this method are PCBs with congener numbers 8L, 79L, and 162L.

OcCB – Octachlorobiphenyl

OPR – Ongoing precision and recovery standard (OPR); a method blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

PCB – Polychlorinated biphenyl – One of the 209 individual chlorinated biphenyl congeners determined using this method. The 209 CBs are listed in Table 1.

PeCB – Pentachlorobiphenyl

Perfluorotributylamine (PFTBA) – Calibrant compound used to calibrate and tune the MS instrument.

PTFE – Polytetrafluoroethylene

Reagent water – Water demonstrated to be free from the analytes of interest and potentially interfering substances at the method detection limit for the analyte.

Relative standard deviation (RSD) – The standard deviation multiplied by a 100 and divided by the mean. Also termed "coefficient of variation."

Relative standard error (RSE) – The standard error multiplied by a 100 and divided by the mean. RSE may be used as an alternative approach to evaluating the calibration model.

Retention time calibration – process by which, using a mixture of known target analytes, the retention times, relative retention times, and retention time windows of those analytes are determined for later use in identifying the analytes.

RF – Response factor. See Section 10.5.3.2.

RR – Relative response. See Section 10.5.3.2.

RRT – Relative retention time; ratio calculated by dividing the RT of the analyte with the RT of the labeled compound used for quantification

RT – Retention time**;** the time it takes for an analyte or labeled compound to elute from the GC column. Usually measured from the point of injection to the apex of the peak. For asymmetric peaks, it is measured to the center of the mass of the peak.

Selected ion monitoring (SIM) – See definition for multiple ion monitoring (MIM).

Should – This action, activity, or procedural step is suggested but not required.

Signal-to-noise ratio (S/N) – The height of the signal as measured from the mean (average) of the noise to the peak maximum divided by the width of the noise.

SFE – Separatory funnel extraction; an extraction technique in which an analyte is extracted from an aqueous sample into an organic solvent using a separatory funnel

SPE – Solid-phase extraction; an extraction technique in which an analyte is extracted from an aqueous sample by passage over or through a material capable of reversibly adsorbing the analyte. Also termed liquid-solid extraction.

Stock solution – A solution containing an analyte that is prepared using a reference material traceable to EPA, NIST, or a source that will attest to the purity and authenticity of the reference material.

TeCB – Tetrachlorobiphenyl

TrCB – Trichlorobiphenyl

True Isotope dilution quantitation (tID) – A means of determining the concentration of a target analyte by reference to the same compound in which one or more atoms has been replaced with a stable isotope. In this method, all 12 carbon atoms in the biphenyl molecule are replaced with carbon-13 atoms to produce $^{13}C_{12}$- labeled analogs of the chlorinated biphenyls. The $^{13}C_{12}$-labeled PCBs are spiked into each sample and allow identification and correction of the concentration of the native compounds in the analytical process.

Tuning – see Mass resolution

WHO – World Health Organization