WSC – CAM – V A

Quality Control Requirements and Performance Standards for the ***Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

V. Gas Chromatography (GC) Methods

A. Quality Control Requirements and Performance Standards for WSC-CAM-V A (Polychlorinated Biphenyls by GC)

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CAM	**Compendium of Analytical Methods**
CASN	**Chemical Abstracts Service Number**
CCAL	**Continuing calibration**
%D	**Percent difference or percent drift**
DBOFB	**4,4'-Dibromooctafluorobiphenyl**
DCB	**Decachlorobiphenyl**
DDD	**Dichlorodipheyldichloroethane**
DDE	**Dichlorodiphenylethane**
DDT	**Dichlorodiphenyltrichloroethane**
DF	**Dilution factor**
ECD	**Electron capture detector**
ELCD	**Electrolytic conductivity detector**
GC	**Gas chromatograph**
ICV	**Initial calibration verification**
IRAs	**Immediate Response Actions**
LCS	**Laboratory control sample**
MassDEP	**Massachusetts Department of Environmental Protection**
MCP	**Massachusetts Contingency Plan**
MD	**Matrix duplicate**
MOHML	**Massachusetts Oil and Hazardous Materials List**
MS	**Matrix spike**
MSD	**Matrix spike duplicate**
NA	**Not applicable**
PCB	**Polychlorinated biphenyl**
PTFE	**Polytetrafluoroethylene**
QA	**Quality assurance**
QC	**Quality control**
r	**Correlation coefficient**
r^2	**Coefficient of determination**
RAO	**Response Action Outcome**
RCs	**Reportable Concentrations**
RL	**Reporting limit**
RPD	**Relative percent difference**
RQs	**Reportable Quantities**
%RSD	**Percent relative standard deviation**
TCMX	**Tetrachloro-m-xylene**
µg/kg	**micrograms per kilogram**
µg/L	**micrograms per liter**

1.0 Quality Control Requirements and Performance Standards for WSC-CAM-V A

1.1 Overview of WSC-CAM-V A

WSC-CAM-V A, *Quality Control Requirements and Performance Standards for the Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC) in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, is a component of MassDEP's Compendium of Analytical Methods (CAM). Effective July 1, 2010, this revised CAM protocol, WSC-CAM-V A, replaces the original Polychlorinated Biphenyl GC CAM document, WSC-CAM-V A (effective date, August 20, 2004). Refer to WSC-CAM-I A for an overview of the CAM process. Please note that while this protocol must be followed on and after the effective date of July 1, 2010 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on April 15, 2010.

This document provides Quality Control (QC) requirements and performance standards to be used in conjunction with the required analytical method SW-846 8082A, analysis for PCBs in aqueous and solid samples by GC preceded by conventional sample preparation methods via SW-846 Methods, as described in Section 1.3 of this protocol. The QC requirements and performance standards specified in this document in Table V A-1 together with the analytical procedures described in EPA SW-846 Method 8082A, *Polychlorinated Biphenyls (PCBs) by Gas Chromatography*, constitute the WSC-CAM-V A protocol. All protocols included in the CAM are considered "methods" published by the MassDEP pursuant to the provisions of 310 CMR 40.0017(2). Use of EPA SW-846 8082A is a "Presumptive Certainty" requirement of WSC-CAM-V A. However, it should be noted that if the laboratory utilizes the analytical procedures in SW-846 Method 8082 instead of 8082A, it is acceptable to answer "YES" to Question B on the MassDEP Analytical Protocol Certification Form since there are no analytical procedural differences between 8082 and 8082A. Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for PCBs analyzed in support of MCP decision-making are presented in Appendix V A-1 of this document and Appendix VII-A of WSC-CAM-VII A *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*. Data reporting requirements are also provided in WSC-CAM-VII A.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data quality objectives, regardless of "Presumptive Certainty" status. For more guidance on data usability, refer to MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.1.1 Reporting Limits for WSC-CAM-V A

The reporting limit (RL) for an individual PCB congener or PCB Aroclor using WSC-CAM-V A is dependent on the concentration of the lowest non-zero standard in the initial calibration, analyzed under identical conditions as the sample, with adjustments made for the sample size, extraction concentration factor, percent solids, dilution factors, etc., as required. The CAM RLs for WSC-CAM-V A target analytes are:

- 0.25 µg/L for aqueous samples (surface water, groundwater, and drinking water); and
- 100 µg/kg (wet weight) for soil/sediment samples (assuming 100% solids).

These values are readily achievable using electron capture detectors (ECDs). Somewhat higher RLs may be expected using electrolytic conductivity detectors (ELCD). For "Presumptive Certainty" purposes, if the CAM RLs are not achieved, respond "NO" to Question G of the "MassDEP MCP Analytical Protocol Certification Form" and address the CAM RL exceedance in the laboratory narrative.

Reporting limits lower than the above-referenced CAM RLs for WSC-CAM-V A target analytes may be required to satisfy project requirements. The RL (based on the concentration of the lowest calibration standard) for each contaminant of concern must be less than or equal to the MCP standards or criteria that the contaminant concentrations are being compared to (e.g., Method 1 Standards, benchmark values, background, etc.). Meeting MCP standards or criteria may require method modifications, such as reducing the volume of the final extract, to improve sensitivity. All such modifications must be described in the laboratory narrative. Regardless of the modification that is used, RLs for the WSC-CAM-V A target analytes will be proportionately higher for samples that require dilution, when a reduced sample size is used, or for an increased final extract volume.

1.1.2 Initial Demonstration of Proficiency for WSC-CAM-V A

Each laboratory that uses the WSC-CAM-V A protocol is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, and the analysis of laboratory control samples (LCSs) and LCS duplicates to assess analytical accuracy and precision. Matrix spikes (MS), matrix spike duplicates (MSD) or matrix duplicates may also be used to evaluate accuracy and precision when such samples are analyzed either at the discretion of the laboratory or at the request of the data user.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Table V A-1 of this protocol and SW-846 Method 8000B. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 Method 8000B (Section 8.4) and SW-846 method 8082A (Section 9.4). The data associated with the Initial Demonstration of Proficiency must be kept on file at the laboratory and made available to potential data users on request. The data associated with the Initial Demonstration of Proficiency for WSC-CAM-V A must include the following information:

QC Element	Performance Criteria
Initial Calibration	WSC-CAM-V A, Table V A-1
Continuing Calibration	WSC-CAM-V A, Table V A-1
Method Blanks	WSC-CAM-V A, Table V A-1
Average Recovery	SW-846 Method 8000B, Section 8.4
% Relative Standard Deviation	SW-846 Method 8000B, Section 8.4
Surrogate Recovery	WSC-CAM-V A, Table V A-1
Internal Standards	WSC-CAM-V A, Table V A-1

NOTE: Because of the number of QC elements associated with the Initial Demonstration of Proficiency, it should be expected that one or more analytes may not meet the performance standard for one or more QC elements. Under these circumstances, the analyst should attempt to locate and correct the problem and repeat the analysis for all non-conforming analytes. All non-conforming analytes along with the laboratory-specific acceptance criteria should be noted in the Initial Demonstration of Proficiency documentation.

It is essential that laboratory-specific performance criteria for LCS, LCS duplicate and surrogate recoveries also be calculated and documented as described in SW-846 Method 8000B, Section 8.7. Experience indicates that the criteria recommended in specific methods are frequently not met for some analytes and/or matrices; the in-house performance criteria will be a means of documenting these repeated exceedances. Laboratories are encouraged to actively monitor pertinent QC performance standards described in Table V A-1 to assess analytical trends (i.e., systematic bias, etc) and improve overall method performance by preempting potential non-conformances.

For the WSC-CAM-V A protocol, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed in Table V A-1. It should be noted that the performance standards listed in Table V A-1 are based on multiple-laboratory data, which are in most cases expected to demonstrate more variability than performance standards developed by a single laboratory.

This protocol is restricted to use by, or under the supervision of, analysts experienced in the use of GC instrumentation as a quantitative tool and skilled in the interpretation of chromatograms for PCB Aroclors or congeners.

1.2 Summary of SW-846 Method 8082A

The samples are prepared for GC analysis using the appropriate sample preparation and, if necessary, sample cleanup procedures (refer to Section 1.3).

After cleanup, the extract is analyzed by injecting a 1 to 2-µL aliquot into a gas chromatograph with a narrow- or wide-bore fused silica capillary column. The GC oven is temperature-programmed to facilitate separation of the analytes of interest, which are then detected by an ECD or ECLD that is interfaced directly to the gas chromatograph.

Identification of PCB congeners is accomplished by comparing the sample retention time with the retention time of standards obtained under identical analytical conditions. Identification of PCB Aroclors is accomplished by comparing the sample's characteristic peaks that comprise the "fingerprint" of the mixture, using both the retention times and shapes of the indicator peaks with the same pattern of peaks in standards obtained under identical analytical conditions. Quantitation is accomplished by using the peak area and a calibration factor generated from a minimum five-point calibration curve.

Identification of PCBs on a single-column must be confirmed on a second column, or must be supported by at least one other independent qualitative technique. Although a dual-column option may satisfy this requirement, due caution should be exercised when highly contaminated samples are processed or during

times of high sample throughput. Dual column confirmation is not required for samples with concentrations of PCBs below their respective RL.

1.3 Sample Extraction/Cleanup Methods for WSC-CAM-V A

Samples for analysis by SW-846 Method 8082A must be extracted or diluted using one of the following methods.

SW-846 Extraction Method	Matrix	Description
3510C	Aqueous	Separatory Funnel Liquid-Liquid Extraction
3520C	Aqueous	Continuous Liquid-Liquid Extraction
3511	Aqueous	Organic Compounds in Water by Microextraction
3535A	Aqueous	Solid-phase Extraction (SPE)
3540C	Soil/Sediment	Soxhlet Extraction
3541	Soil/Sediment	Automated Soxhlet Extraction
3545A	Soil/Sediment	Pressurized Fluid Extraction (PFE)
3546	Soil/Sediment	Microwave Extraction
3570	Soil/Sediment	Microscale Solvent Extraction (MSE)
3550C	Contaminated Solids[1]	Ultrasonic Extraction
3580A	NAPL	Waste Dilution
[1]Sonication may only be used for the extraction of highly contaminated (free product) non-soil/sediments (debris). Any other use of ultrasonic extraction is not allowed.		

Extracts may be cleaned up, as required, by any of the following methods prior to GC analysis by SW-846 Method 8082A. The recommended cleanup methods for routine PCB analyses are SW-846 Methods 3660B and 3665A.

SW-846 Cleanup Methods	Cleanup Type
3600C	NA; General cleanup selection
3610B	Alumina column
3620C	Florisil column
3630C	Silica gel
3640A	Gel permeation chromatography
3660B	Sulfur
3665A	Sulfuric Acid/Permanganate Cleanup

1.4 Method Interferences

- Refer to SW-846 Methods 3500C (Section 4.0, in particular), 3600C, and 8000B for a detailed discussion of interferences. Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories.

  - Contaminated solvents, reagents, or sample processing hardware,

- Contaminated GC carrier gas, parts, column surfaces, or detector surfaces,
- Non-target compounds simultaneously extracted from the sample matrix which cause a detector response, and
- Co-elution of target analytes.

An in depth discussion of the causes and corrective actions for all of these interferences is beyond the scope of this guidance document. A brief discussion of the more prevalent interferences is presented below.

- Refer to SW-846 Method 8082A for a detailed description of chemical contaminants, cross-contamination, and corrective actions which may be taken to eliminate contamination. If a method blank contains a contaminant, data for samples associated with that blank must **not** undergo "blank correction" (i.e., if an associated sample also contains the contaminant, subtraction of the blank amount from the sample amount is not permitted).

- Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of PCBs. After the analysis of a sample containing high concentrations of PCBs, one or more blanks should be analyzed to check for potential cross-contamination/carryover. Concentrations of PCBs which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later sample analysis. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections.

- Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations by SW-846 Method 8082A. Common flexible plastics contain varying amounts of phthalate esters, as plasticizers, which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination. These materials can be removed through the use of SW-846 Cleanup Method 3665A (sulfuric acid/permanganate cleanup).

- Elemental sulfur (S) is readily extracted from soil/sediment samples and may cause chromatographic interferences in the determination of PCBs by SW-846 Method 8082A. Sulfur contamination should be expected with sediment samples. Sulfur contamination can be removed through the use of SW-846 Method 3660B.

- Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized at laboratory drying oven temperatures and spread to other glassware. Due caution should be exercised when drying glassware used for the analysis of samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

1.5 Quality Control Requirements for WSC-CAM-V A

1.5.1 General QC Requirements

Refer to SW-846 Method 8000B for general QC procedures for all chromatographic methods, which includes SW-846 method 8082A. Instrument QC and method performance requirements for the GC/ECD or GC/ELCD system may be found in SW-846 method 8082A, Sections 9.0 and 13.0, respectively.

1.5.2 Specific QC Requirements and Performance Standards for WSC-CAM-V A

Specific QC requirements and performance standards for the WSC-CAM-V A protocol are presented in Table V A-1. Refer to WSC-CAM-VII A for field QC requirements. Strict compliance with the QC requirements and performance standards, as well as satisfying the CAM's other analytical and reporting requirements will provide a data user with "Presumptive Certainty" in support of Response Actions under the MCP. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of WSC-CAM-VII A.

While optional, parties electing to utilize these protocols will be assured of "Presumptive Certainty" of data acceptance by agency reviewers. In order to achieve "Presumptive Certainty" for analytical data, parties must:

(a) Use the analytical method specified for the selected CAM protocol;
(b) Incorporate **all** required analytical QC elements specified for the selected CAM protocol;
(c) Implement, as necessary, required corrective actions and analytical response actions for **all** non-conforming analytical performance standards;
(d) Evaluate and narrate, as necessary, **all** identified CAM protocol non-compliances; and
(e) Comply with **all** the reporting requirements specified in WSC-CAM-VII A, including retention of reported and unreported analytical data and information for a period of ten (10) years.

In achieving "Presumptive Certainty" status, parties will be assured that analytical data sets:

- ✓ Satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data; and

- ✓ May be used in a data usability and representativeness assessment, as required in 310 CMR 40.1056(2)(k) for Response Action Outcome (RAO) submittals, consistent with the guidance described in MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.6 Special Analytical Considerations for WSC-CAM-V A

The following bullets highlight potential issues that may be encountered with the analysis of PCBs using this protocol.

- The identification of multi-component PCB Aroclors is not based on a single peak, but rather on the characteristic peaks that comprise the "fingerprint" of the mixture, using both the retention times and shapes of the indicator peaks. If, based on site history, specific PCB Aroclors are contaminants of

concern, it is the responsibility of the data user to request that these specific PCB Aroclor spikes be included in the LCSs and MS/MSDs. All PCB Aroclors are not routinely included in LCSs or MS/MSDs.

- It is highly recommended that extracts for PCB analysis be routinely subjected to a sulfuric acid cleanup using SW-846 Method 3665A. This cleanup technique will remove (destroy) most other organic compounds including many single component organochlorine or organophosphorus pesticides as well as phthalate contaminants which could potentially interfere with the quantitation of PCB Aroclors or congeners.

- A sample may contain what appears to be PCBs but can still be reported as non-detect for the PCB Aroclors used in calibration. This can happen due to weathering, degradation, etc. of the PCBs causing an unclear match or "fingerprint" of the PCB Aroclor. When the match is unclear and reported as a nondetect, it is important to alert the data user of the potential presence of PCBs in their sample although PCB Aroclors are reported as nondetects. The laboratory will be required to note this in the laboratory narrative and provide the chromatogram of the affected sample in the laboratory report. The data user must be aware that additional sampling or other analyses (i.e., PCB congeners or PCB homologues) may be more appropriate in order to accurately quantitate total PCBs.

- PCBs are regulated under the MCP either as specific Aroclor mixtures (Aroclors 1016, 1221, 1232, 1242, 1248, 1254 and 1260) or as PCB-N.O.S. (Not Otherwise Specified, CAS Number 01336-36-3). The latter category includes ***all*** chlorinated Biphenyl derivatives (209 possible PCB congeners). The cumulative sum of all such congeners would be regulated under this CAS Number, PCB-N.O.S, as Total PCBs. At the discretion of the data user requesting the analysis, the use of PCB congeners rather than PCB Aroclors may be an appropriate analytical alternative under the following circumstances:

    - Samples containing multiple PCB Aroclors;
    - Samples containing PCB Aroclors that have been weathered by long exposure in the environment;
    - Process samples containing PCB Aroclors that have been subjected to degradation by destructive treatment technologies;
    - Evaluations requiring greater accuracy and specificity at sites with known PCB contamination;
    - Samples collected in support of comprehensive ecological risk assessments; and/or
    - To provide more specific and accurate total PCB contaminant concentrations in support of MCP Method 3 risk assessment evaluations.

- The appropriate list of congeners to be evaluated should be determined by the data user in consultation with the laboratory and other end users of the data (risk assessors, etc.) on a site-specific basis. Alternatively, EPA Method 680, *Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry* (November 1985) should be considered an option to resolve the aforementioned analytical complications. EPA Method 680 utilizes GC/mass spectrometry (MS) operated in the selective ion monitoring (SIM) mode to identify and quantify the various PCB homologues (same number of substituted chlorines). A summation of the individual PCB homologues may then be used to reliably determine Total PCBs.

- A linear or non-linear calibration model must not be used to compensate for detector saturation or to avoid proper instrument maintenance. As such, linear or non-linear regression must not be employed for initial calibration calculations that typically meet percent relative standard deviation (%RSD) requirements specified in Table V A-1.

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain Aroclors 1016/1260 for PCB Aroclor analysis and all target congeners for PCB congener analysis. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol.	NA
Retention Time Windows	Laboratory Analytical Accuracy	(1) Prior to initial calibration and when a new GC column is installed. (2) Calculated according to the method (Section 7.6 of SW-846 8000B) (3) If acid cleanup is not performed, also analyze DDT/DDE/DDD standard.	No	NA	(1) For PCB Aroclor analysis, if interference is present for any of the Aroclor peaks used for quantitation with DDT, DDE, or DDD, either adjust GC conditions to obtain better resolution or choose another peak for the Aroclor of interest that does not coelute with DDT, DDE, or DDD. (2) For PCB congener analysis, if interference is present for any of the target congeners with DDT, DDE, or DDD, adjust GC conditions to obtain better resolution.	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used).	No	NA	(1) Recalibrate as required by method. (2) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL limit must be	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.99, or r^2 <0.99) in laboratory narrative. If non-linear regression (e.g., quadratic equation) is used for calibration,

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(3) *PCB Aroclors:* 5-point calibration with 1016/1260 required; 5-point calibration for other Aroclors may be warranted based on site-specific conditions (i.e., if nature of PCB contamination known). *Congeners:* 5-point calibration must include all target PCB congeners. (4) A minimum of 5 unique peaks must be evaluated for Aroclors 1016 and 1260. (5) Low standard must be ≤RL. (6) %RSD ≤20, r ≥0.99 (linear regression), or r^2 ≥0.99 (non-linear regression) for each PCB Aroclor or each PCB congener. (7) If %RSD >20, linear or non-linear regression must be used. (8) PCB Aroclors: For Aroclors which are not calibrated with 5-points, laboratory must perform single analysis of these Aroclors at the midpoint of the calibration curve. (9) Calibration must be performed under the same conditions as the samples. (10) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.			reported as an estimated value[2], or * The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	this must be noted in the laboratory narrative along with the congeners or PCB Aroclors affected.
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial	No	NA	Locate source of problem; recalibrate if either PCB Aroclor 1016/1260 or >10% of all PCB congeners are outside of criteria.	If recovery is outside of 80-120% for any PCB Aroclor or congener, report non-conformances in laboratory narrative.

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		calibration. (4) Must contain Aroclors 1016/1260 for PCB Aroclor analysis and all target congeners for PCB congener analysis. (5) Percent recoveries must be between 80-120% for each PCB Aroclor or congener.				
Continuing Calibration	Laboratory Analytical Accuracy	(1) Prior to samples, every 12 hours or every 20 samples, whichever is more frequent, and at the end of the analytical sequence. (NOTE: if internal standard calibration used, the continuing calibration at the end of the analytical sequence is not required). (2) Concentration level near midpoint of curve. (3) *PCB Aroclors:* Must contain Aroclors 1016/1260. Aroclors other than 1016/1260 must be verified with a one-point standard within 12 hours of being detected in a sample. *Congeners:* Must include all target PCB congeners. (4) %D must be $\leq$20 for each PCB Aroclor or PCB congener. (5) Verify that all analytes fall within retention time windows. (6) Area count of internal standard in continuing calibration must be within $\pm$50% of the average area count in the associated initial calibration.	No	NA	(1) Perform instrument maintenance, reanalyze continuing calibration and/or recalibrate as required by method. (2) Renalyze "associated samples" if beginning or ending continuing calibration exhibited low response. (3) Reanalyze "associated samples" if beginning or ending continuing calibration exhibited high response and associated PCB Aroclors or congeners were detected in the "associated samples." NOTE: "Associated samples" refers to all samples analyzed since the last acceptable continuing calibration.	Report non-conforming compounds (%D >20) and associated samples in laboratory narrative.
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil).	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of	(1) If sample re-extraction is not possible, report non-conformance in

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Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(3) Target analytes must be <RL.			contamination; correct problem; re-extract and re-analyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) *PCB Aroclors:* 1016/1260 required. Optionally, LCSs may be spiked with other Aroclors which have been fully calibrated, based on site-specific conditions (i.e., if specific Aroclors are known to be present or expected in samples). *Congeners:* Must include all target PCB congeners. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected PCB Aroclor or PCB congener in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if either Aroclor 1016/1260 or >10% of all PCB congeners are outside of criteria. (2) If ≤10% of PCB congeners are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of PCB congeners or either Aroclor 1016/1260 are above the acceptance criteria (>140), reextraction is not required if the affected congeners or all PCB Aroclors were not detected in associated samples.	(1) If sample re-extraction is not possible, report nonconformance in laboratory narrative. (2) If recovery is outside of 40-140% for any PCB Aroclor or congener, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) *PCB Aroclors:* 1016/1260 required. Optionally, LCS Duplicates may be spiked with other Aroclors which have been fully calibrated, based on site-specific conditions (i.e., if specific Aroclors are known to be present or expected in samples).	Yes	Recovery <10%; affects nondetect results for affected PCB Aroclor or PCB congener in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if either Aroclor 1016/1260 or >10% of all PCB congeners are outside of recovery acceptance criteria. (2) If ≤10% of PCB congeners are outside of the recovery acceptance	(1) If sample re-extraction is not possible, report nonconformance in laboratory narrative. (2) If recovery is outside of 40-140% for any PCB Aroclor or congener or if RPD is outside of criteria, report non-conforming compounds in laboratory

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		Congeners: Must include all target PCB congeners. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) RPDs must be ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).			criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of PCB congeners or either Aroclor 1016/1260 are above the recovery acceptance criteria (>140%), reextraction is not required if the affected congeners or all PCB Aroclors were not detected in associated samples.	narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) *PCB Aroclors:* 1016/1260 required. Optionally, MS/MSDs may be spiked with other PCB Aroclors which have been fully calibrated, based on site-specific conditions (i.e., if specific Aroclors known to be present or expected in samples). *Congeners:* Must include all target PCB congeners. (5) Percent recoveries between 40 – 140%. (6) RPDs ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected PCB Aroclor or PCB congener in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 2 surrogates, one that elutes at beginning of GC run and one that elutes at end of GC run. Recommended surrogates: *PCB Aroclor analysis:* TCMX and DCB *PCB Congener analysis:* TCMX or DBOFB and BZ198 (2) Percent recoveries must be between 30-150% for both surrogates on both columns.	Yes (report surrogate recoveries from both columns)	Recovery <10%; affects all nondetect results in affected sample.	If the same surrogate is outside limits on both columns: (1) Re-extract the sample if surrogate recoveries are low and there is no chromatographic interference. (2) Re-extract the sample if surrogate recoveries are high and PCB Aroclors or PCB congeners were detected in the sample. NOTES: (a) If surrogate recoveries are high and target analytes are not detected in sample, re-extraction is not required. (b) If chromatographic interference is present and surrogate recovery would cause rejection of data (i.e., <10%), reanalyze sample on dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, reextraction and/or reanalysis is not required.	(1) Report recoveries outside of 30-150% in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both the initial extraction and re-extraction. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of the holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial extraction and re-extraction. (5) If sample is not re-extracted due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Internal Standards (Congeners only)	Laboratory Analytical Accuracy and Method Accuracy in	(1) Minimum of 1. Recommended internal standard: DCB (2) Area counts in samples must be between 50 – 200% of the area counts	No	Recovery <20%; affects all nondetect results quantitated using	If internal standard is outside of limits, reanalyze sample unless chromatographic	(1) Report non-conformances in laboratory narrative. Include actual recovery

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
	Sample Matrix	in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.		affected internal standard in associated sample.	interference present. NOTE: If chromatographic interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution.	of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Identification and Quantitation	NA	(1) Peak area is the expected default to be used for quantitation of PCB Aroclors and congeners under most circumstances. Regardless if peak area or peak height is used, the same method used for quantitation of	NA	If RPD >100 for PCB congener, reject positive result for affected PCB congener. If RPD >500 for	If the RPD between the dual column results is >100 for PCB congeners or >500 for PCB Aroclors, reanalyze the sample on dilution. Both analyses	If the RPD between the dual column results exceeds 40, the laboratory must qualify the sample results and/or note the

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		samples must also be used for calibration standards. (2) *PCB Aroclors:* The laboratory must quantitate all Aroclors with the same five peaks used for calibration. If interference exists with select peaks, these peaks do not have to be included in the quantitation of the Aroclor; however, a minimum of three peaks is required. All peaks must be ≥25% of the height of the largest PCB Aroclor peak. At least one peak must be unique to the PCB Aroclor. (3) *PCB Congeners:* The laboratory must use the average calibration factor, response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each PCB congener. *PCB Aroclors:* Laboratory should use the average calibration factor, linear or non-linear regression curve for each of three to five peaks from each concentration level to quantitate Aroclors 1016 and 1260. Laboratory should use the average calibration factor for each of three to five peaks from single point standard to quantitate remaining Aroclors (when only single-point standard analyzed). If 5-point calibration is performed for other Aroclors, follow procedure for 1016 and 1260. Calculate concentration of Aroclor using each individual peak and calculate the average concentration of the three to five results to obtain the final Aroclor concentration.		PCB Aroclor, reject positive result for affected PCB Aroclor.	must be reported. Alternatively, additional sample cleanup techniques may be warranted.	exceedance in the laboratory narrative. If the RPD exceedance is due to interference, the lower of the dual column values can be reported; this must be noted in the laboratory narrative.

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(4) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results. The laboratory must report the higher of the two results. All required QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well. (5) Results must be reported with 2 or more "significant figures" if ≥ RL. If reporting values below the RL, report with 1 or more "significant figures".[3]				
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. **NOTE: Laboratories shall not perform dilutions on samples due to sulfur interference. Laboratories must employ a cleanup technique to reduce the presence of sulfur interference.** **It is highly recommended that acid cleanup be performed on all sample extracts prior to analysis.** (3) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (4) Refer to Appendix V A-1 for chain-of-custody requirements regarding preservation, cooler temperature, and	NA	NA	NA	(1) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (2) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (3) If PCB Aroclors are not detected but chromatogram shows evidence of weathered Aroclors or potential presence of PCBs, this must be noted in the laboratory narrative and a copy of the

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		holding times.				chromatogram must be provided in the data report. (4) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative. (5) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.

[1]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.
[2]If the RL is estimated due to unacceptable recovery of the lowest standard, the CAM RL has not been achieved; Question G of the "MassDEP MCP Analytical Protocol Certification Form" must be answered "NO" and this must be addressed in the laboratory narrative.
[3]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

1.7 Analyte List for WSC-CAM-V A

The MCP analyte list for WSC-CAM-V A is presented in Table V A-2. The list is comprised of nine (9) PCB Aroclor mixtures that are readily-analyzable by WSC-CAM-V A.

This method also provides procedures for the determination of a subset of the possible 209 PCB congeners. Nineteen (19) of 209 possible PCB congeners, listed in Section 1 of SW-846 Method 8082A, have been tested by this method. These congeners were chosen for testing by EPA because many of them are present in the most common PCB Aroclor formulations, and ***not because of their toxicological significance***. Most, ***but not all***, of the remaining 209 potential PCB congeners can be identified/resolved and quantified using the GC columns and chromatographic conditions described in this method after an initial demonstration of proficiency. Congeners are mentioned in this guidance for informational purposes only and ***need not be evaluated*** in support of routine MCP decision-making. Refer to Section 1.6 to determine when the analysis of PCB congeners may be appropriate.

It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL for the PCB Aroclors or congeners. Sources of various MassDEP standards and criteria are as follows:

- Reportable Quantities (RQs) and Concentrations (RCs) as described in 310 CMR 40.1600, The Massachusetts Oil and Hazardous Materials List (MOHML), in Subpart P of the MCP may be found at the following URL: https://www.mass.gov/site-cleanup-regulations-policies-forms-more.
- An online searchable Oil & Hazardous Materials List of RQs and RCs values may be found at the following URL: https://www.mass.gov/service-details/oil-hazardous-material-list.
- An updated list of MCP Method 1 Standards may be found at the following URL: https://www.mass.gov/site-cleanup-regulations-policies-forms-more.

All of the PCB Aroclors listed in Table V A-2 have a promulgated MCP Method 1 groundwater/soil standard.

1.7.1 Analyte List Reporting Requirements for WSC-CAM-V A

While it is not necessary to request and report all the WSC-CAM-V A PCB Aroclors listed in Table V A-2 to obtain “Presumptive Certainty” status, it is necessary to document use and reporting of a reduced analyte list, for site characterization and data representativeness considerations. MassDEP strongly recommends use of the full analyte list during the initial stages of site investigations, and/or at sites with an unknown or complicated history of uses of oil or hazardous materials. These assessment activities may include but are not limited to:

- ✓ Immediate Response Actions (IRAs) performed in accordance with 310 CMR 40.0410;
- ✓ Initial Site Investigation Activities performed in accordance with 310 CMR 40.0405(1);
- ✓ Phase I Initial Site Investigation Activities performed in accordance with 310 CMR 40.0480 through 40.0483; and
- ✓ Phase II Comprehensive Site Investigation Activities performed in accordance with 310 CMR 40.0830

In a limited number of cases, the use of the full analyte list for a chosen analytical method may not be necessary, with respect to data representativeness concerns, including:

- ✓ Sites where substantial site/use history information is available to rule-out all but a limited number of contaminants of concern, and where use of the full analyte list would significantly increase investigative costs; or

- ✓ Well-characterized sites where initial full-analyte list testing efforts have sufficiently narrowed the list of contaminants of concern.

Note: a data user who avoids the detection and quantitation of a contaminant that is present or likely present at a site above background levels by limiting an analyte list could be found in criminal violation of MGL c. 21E or any regulations or orders adopted or issued thereunder.

In cases where a reduced list of analytes is requested, laboratories must still employ the specified QC requirements and performance standards in WSC-CAM-V A to obtain “Presumptive Certainty” status.

Table V A-2: PCB Aroclor Analyte List for WSC-CAM-V A (SW-846 8082A)

PCB Aroclor	CASN
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262[1]	37324-23-5
Aroclor 1268[1]	11100-14-4

1. Not specifically listed in Subpart P, MOHML but regulated as PCB-N.O.S. (not otherwise specified, CAS Number 01336-36-3)

NOTE: PCB congeners may also be analyzed using the WSC-CAM-V A Protocol but are not considered part of the CAM target analyte list.

CASN – Chemical Abstracts Service Numbers

2.0 Data Usability Assessment

Specific guidance applicable to all Class A, B or C RAO Statements, including partial RAOs, for preparation of Representativeness Evaluations and Data Usability Assessments pursuant to 310 CMR 40.1056(2)(k) of the MCP is provided in *MCP Representativeness Evaluations and Data Usability Assessments* (Policy #WSC-07-350). This document provides general information regarding the purpose and content of these required evaluations as a component of and in support of an RAO submittal. The most current version of this document may be found at the following URL: https://www.mass.gov/site-cleanup-regulations-policies-forms-more.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data objectives using MassDEP Policy #WSC-07-350, regardless of "Presumptive Certainty" status.

3.0 Reporting Requirements for WSC-CAM-V A

3.1 General Reporting Requirements for WSC-CAM-V A

General environmental laboratory reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in WSC-CAM-VII A, Section 2.4. This guidance document provides limited recommendations for field QC, as well as the required content of the laboratory report, which includes:

- Laboratory identification information,
- Analytical results and supporting information,
- Sample- and batch-specific QC information,
- Laboratory Report Certification Statement,
- Copy of the Analytical Protocol Certification Form,
- Laboratory narrative contents, and
- Chain-of-custody form requirements.

3.2 Specific Reporting Requirements for WSC-CAM-V A

Specific QC requirements and performance standards for WSC-CAM-V A are presented in Table V A-1. Specific reporting requirements for WSC-CAM-V A are summarized below in Table V A-3 as "Required Analytical Deliverables (**YES**)". These routine reporting requirements must always be included as part of the laboratory deliverable for this method. It should be noted that although certain items are not specified as "Required Analytical Deliverables (**NO**)", these data must be available for review during an audit and may also be requested on a client-specific basis.

Soil and sediment results must be reported on a dry-weight basis. Refer to ASTM Method D2216, Determination of Moisture Content of Soils and Sediments, for more detailed analytical and equipment specifications.

Table V A-3 Routine Reporting Requirements for WSC-CAM-V A (SW-846 8082A)

Parameter	Required Analytical Deliverable
Retention Time Windows	***NO***
Initial Calibration	***NO***
Initial Calibration Verification	***NO***
Continuing Calibration (CCAL)	***NO***
Method Blank	***YES***
Laboratory Control Samples (LCSs)	***YES***
LCS Duplicates	***YES***
Matrix Spike (MS)	***YES (if requested by data user)***
Matrix Spike Duplicate (MSD)	***YES (if requested by data user)***
Matrix Duplicate (MD)	***YES (if requested by data user)***
Surrogates	***YES***
Internal Standards	***NO***
Identification and Quantitation	***NO***
General Reporting Issues	***YES***

3.2.2 Sample Dilution

Under circumstances that sample dilution is required because either the concentration of one or more of the target analytes exceed the concentration of their respective highest calibration standard or any non-target peak exceeds the dynamic range of the detector (i.e., "off scale"), the RL for each PCB Aroclor or congener must be adjusted (increased) in direct proportion to the Dilution Factor (DF).

The revised RL for the diluted sample, RL_d:

$$RL_d = DF \times \text{Lowest Calibration Standard for Target Analyte}$$

It should be understood that samples with elevated RLs as a result of a dilution may not be able to satisfy MCP standards/criteria in some cases if the RL_d is greater than the applicable MCP standard or criterion to which the concentration is being compared. Such increases in RLs are the unavoidable but acceptable consequence of sample dilution that enable quantification of target analytes which exceed the calibration range. All dilutions must be fully documented in the laboratory narrative.

NOTE: **Over dilution is an unacceptable laboratory practice.** The post-dilution concentration of the target analyte with the highest concentration must be at least 60 to 80% of its associated highest calibration standard. This will avoid unnecessarily high RLs for other target analytes which did not require dilution.

Appendix V A-1

Sample Collection, Preservation, and Handling Procedures for Polychlorinated Biphenyl Analyses

Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for PCBs analyzed in support of MCP decision-making are summarized below and presented in Appendix VII A-1 of WSC-CAM-VII A, *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data Conducted in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*.

Matrix	Container[1]	Preservation[7]	Holding Time[3,6]
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Cool to ≤ 6°C but not frozen	1 year to extraction; 40 days from extraction to analysis[5]
Aqueous Samples, with Residual Chlorine[4]	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%)[4]. Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to ≤ 6°C but not frozen.	1 year to extraction; 40 days from extraction to analysis[5]
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap[2]	Cool to ≤ 6°C[2]	1 year to extraction; 40 days from extraction to analysis[5]
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a teflon-lined screw cap.	No special preservation required	1 year to extraction; 40 days from extraction to analysis[5]

[1]The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised during shipping and/or analysis.

[2]Alternatively, soil/sediment samples for PCB analyses may be held for up to one (1) year if frozen within 24 hours of collection at <-10°C. Sampling container should only be filled to 2/3 of capacity to avoid breakage caused by expansion during freezing. Preparation or extraction must be commenced within 24 hours of thawing. Temperature must never be allowed to go below – 20°C to avoid damage to seals, etc.

[3]Holding time begins from time of sample collection or date thawed (see note #2 above).

[4]Presence of chlorine residual is usually associated with drinking water samples. Confirm dechlorination. If residual chlorine >5 mg/L, additional dechlorination agent may be required.

[5]PCB sample extracts must be stored at 4°C, protected from light, and stored in sealed vials (e .g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa.

[6]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if the holding time is exceeded by >2x the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

[7]If samples were received by the laboratory on the same day of collection and were stored and transported to the laboratory on ice, cooler temperatures above 6°C are acceptable.

Appendix V A-2

Data Deliverable Requirements for Data Audits

If requested by MassDEP, submission of the information listed below may be required to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a "full data package" including all sample documentation from receipt through preparation, analysis, and data reporting. The laboratory must ensure that these deliverables are available, in the event a data audit is performed. The laboratory is required to retain these deliverables for a period of 10 years from the date generated.

DELIVERABLE REQUIREMENTS FOR DATA AUDITS WSC-CAM-V A (PCBs by GC/ECD)	
Laboratory Narrative	Must comply with the required laboratory narrative contents as described in WSC-CAM-VII A
Sample Handling Information	Chains-of-custody (external and internal), sample receipt logs (cooler temperatures and sample pH), correspondences
Miscellaneous Logs	Dry weight logs Injection logs Soil/sediment sample weight logs Freezer logs Sample preparation/cleanup logs[1]
Initial Calibration Data (both columns)	Summary of calibration factors for all standards in initial calibration; average calibration factors, %RSDs, correlation coefficients, and coefficients of determination for all target compounds Chromatograms for all standards used in initial calibration (multi-point and single-point calibrations) Quantitation reports for all standards used in initial calibration (multi-point and single-point calibrations) Concentrations of standards used must be clearly presented
Initial Calibration Verification Data (both columns)	Summary of percent recoveries for all target compounds Chromatograms for all ICVs Quantitation reports for all ICVs
DDT/DDD/DDE Standards (if acid cleanup not performed) (both columns)	Chromatograms for all standards Quantitation reports for all standards
Continuing Calibration Data (both columns)	Summary of %Ds and calibration factors Chromatograms for all continuing calibration standards Quantitation reports for all continuing calibration standards Concentrations of standards used must be clearly presented

DELIVERABLE REQUIREMENTS FOR DATA AUDITS **WSC-CAM-V A (PCBs by GC/ECD)**	
Sample Results (both columns)	Chromatograms for all sample analyses, reanalyses, and dilutions Quantitation reports for all sample analyses, reanalyses, and dilutions Percent solids results Summary of results, including reporting limits for each sample Date of analysis
Method Blank Results (both columns)	Chromatograms for all method blanks Quantitation reports for all method blanks Summary of results, including reporting limits Summary of how method blank was prepared in solid and aqueous matrices, as appropriate
LCS/LCS Duplicate Results (both columns)	Chromatograms for all LCS and LCS Duplicates Quantitation reports for all LCS and LCS Duplicates Summary of results, including concentrations detected, concentrations spiked, percent recoveries and RPDs Summary of how LCS/LCS Duplicates were prepared in solid and aqueous matrices, as appropriate
MS/MSD Results (if performed) (both columns)	Chromatograms for all MS/MSDs Quantitation reports for all MS/MSDs Summary of results, including unspiked sample concentrations, concentrations detected, concentrations spiked, percent recoveries and RPDs Summary of how MS/MSDs were prepared in solid and aqueous matrices, as appropriate
QC Summaries (both columns)	Surrogate recoveries Internal standard performance Retention time windows Dual column RPDs
Other Information	Demonstration that ICV prepared from second source standard

Quantitation reports must exhibit peak area counts or peak heights, as appropriate, of target compounds, internal standards, and surrogates.

[1]Must clearly indicate sample weights or volumes, final extract volumes, extraction method used, extraction times where appropriate for the method, etc.