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**
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**
µL	**microliter**

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 February 15, 2024, this revised CAM protocol, WSC-CAM-V A, replaces the previous version of the Polychlorinated Biphenyl GC CAM document, WSC-CAM-V A (effective date, July 1, 2010). 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 February 15, 2024 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on November 15, 2023.

This document provides Quality Control (QC) requirements and performance standards to be used in conjunction with the required analytical method SW-846 8082A (or the most current version), 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). Since the analytical techniques for EPA SW-846 8082A and EPA SW-846 8082 are substantially the same, use of either of these analytical methods (or a subsequent/more current version) meets the "Presumptive Certainty" requirement of WSC-CAM-V A.

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 or Lower Limits of Quantitation for WSC-CAM-V A

The reporting limit (RL) or lower limit of quantitation (LLOQ) 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/LLOQs 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/LLOQs may be expected using electrolytic conductivity detectors (ELCD). For "Presumptive Certainty" purposes, if the CAM RLs/LLOQs are not achieved, respond "NO" to Question G of the "MassDEP MCP Analytical Protocol Certification Form" and address the CAM RL/LLOQ exceedance in the laboratory narrative.

RLs/LLOQs lower than the above-referenced CAM RLs/LLOQs for WSC-CAM-V A target analytes may be required to satisfy project requirements. The RL/LLOQ (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/LLOQs 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 (QA) 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 extraction/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 (MD) 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 8000D. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 Method 8000D (Section 9.3) 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 8000D, Section 9.3
% Relative Standard Deviation	SW-846 Method 8000D, Section 9.3
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 8000D, Section 9.6. 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 GC with a narrow- or wide-bore fused silica capillary column. The GC column 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 GC.

Identification of PCB congeners is accomplished by comparing the retention time of the congener in samples with the retention time of the congener in 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/response 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/LLOQ.

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
3540C	Soil/Sediment	Soxhlet Extraction
3541	Soil/Sediment	Automated Soxhlet Extraction
3545A	Soil/Sediment	Pressurized Fluid Extraction
3546	Soil/Sediment	Microwave Extraction
3570	Soil/Sediment	Microscale Solvent Extraction
3550C	Contaminated Solids[1]	Ultrasonic Extraction
3580A	Non-aqueous Phase Liquid	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 8000D 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 (e.g., broad peaks) 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 Cleanup Method 3660B.

- Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized at laboratory drying oven temperatures and can 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 8000D 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) and 40.1057(2)(k) for Permanent and Temporary Solution 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 operated in the selective ion monitoring 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.

- Quantitation of PCB Aroclors and congeners must be performed using peak area. Use of peak height is only permissible if significant interference is present, precluding the use of the peak area. As a general rule, peak area is considered to be more reproducible since it represents an integrated response over the entire elution window of the analyte rather than an instantaneous (i.e., single point) maximum response within the elution window. Instantaneous response is inherently more variable than integrated response, and older GC/ECD instruments can be more prone to variable maximum/instantaneous response when GC conditions cause fluctuations in peak width vs height, as with temperature or pressure variations. Modern GC/ECD instruments are significantly less vulnerable to these types of fluctuations, but the inherent variability of instantaneous response can still be a factor. Complex matrices with multiple closely eluting peaks due to other analytes or interferences may make the use of peak area difficult; only in these cases can peak height be used for quantitation.

  In addition, a change in the peak shape due to matrix interference or slight variances in column resolution may occur, causing the peak to become shorter and wider, thus causing a decrease in the PCB concentration relative to the lower peak height. Although the peak height may change under these conditions, the same effect is not always seen on the peak area. When the peak shape changes, the height can be reduced, but the peak usually also gets wider resulting in the same peak area. This correlation often results in more consistent concentrations as peak shape changes. Therefore, as stated above, use of peak height may only be suitable with complex matrices.

- 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 9.3 of SW-846 8000D and Section 9.4 of SW-846 8082A.	No	NA	Refer to Section 9.3 of SW-846 8000D 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 11.6 of SW-846 8000D) (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/LLOQ must be	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.995, or r^2 <0.99) 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
		(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) Low standard must be ≤RL/LLOQ. (5) A minimum of 5 unique peaks must be evaluated for Aroclors 1016 and 1260. (6) %RSD ≤20 (average calibration/response factor), r >0.995 (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/LLOQ 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/LLOQ must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	If non-linear regression (e.g., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the congeners or PCB Aroclors affected.

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)

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 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 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.	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.
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) Percent difference or percent drift (%D) must be ≤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 ±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) Reanalyze "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. Note in the laboratory narrative if the %D indicates a low or high bias.

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
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). (3) Target analytes must be <RL/LLOQ.	Yes	NA	(1) If concentration of contaminant in sample is ≤10x concentration in blank, locate source of 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.	(1) If sample re-extraction is not possible, report non-conformance in 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.
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	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	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any PCB Aroclor or congener, report non-conforming

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
		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%. (6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).			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), re-extraction is not required if the affected congeners or all PCB Aroclors were not detected in associated samples.	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). *Congeners:* Must include all target PCB congeners. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140%. (6) RPDs must be ≤20 for waters and ≤30 for solids. (7) 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 recovery acceptance criteria. (2) If ≤10% of PCB congeners are outside of the recovery 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 recovery acceptance criteria (>140%), re-extraction is not required if the	(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 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

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
					affected congeners or all PCB Aroclors were not detected in associated samples.	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 (e.g., water, soil). (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 $\leq$20 for waters and $\leq$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 non-conformances in laboratory narrative.
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	(1) Report recoveries outside of acceptance limits in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both - extractions. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, 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
					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. (3) If a surrogate is diluted to a concentration below that of the lowest calibration standard, re-extraction and/or re-analysis is not required.	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 extractions. (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 Sample Matrix	(1) Minimum of 1. Recommended internal standard: DCB (2) Area counts in samples must be between 50 – 200% of the area counts 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.	No	Recovery <20%; affects all nondetect results quantitated using affected internal standard in associated sample.	If internal standard is outside of limits, re-analyze sample unless chromatographic interference present. NOTE: If chromatographic interference is present and internal standard area would cause rejection of data (i.e., <20%), re-analyze sample on dilution.	(1) Report non-conformances in laboratory narrative. Include actual recovery of internal standard and provide summary of analytes quantitated using the internal standard. (2) If re-analysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the re-analysis only.

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) If re-analysis 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 re-analyzed due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Identification and Quantitation	NA	(1) Peak area is required to be used for quantitation of PCB Aroclors and congeners unless significant interference precludes the use of peak area, in which case peak height can be used (See Section 1.6). If peak height is used due to the significant interference, a separate calibration curve using peak height would need to be generated for quantitation. (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. (4) *PCB Aroclors:* Laboratory should use the	NA	If RPD >100 for PCB congener, reject positive result for affected PCB congener. If RPD >500 for PCB Aroclor, reject positive result for affected PCB Aroclor.	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 must be reported. Alternatively, additional sample cleanup techniques may be warranted.	(1) If the RPD between the dual column results exceeds 40, the laboratory must qualify the sample results and/or note the exceedance in the laboratory narrative. (2) 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
		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. (5) 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. (6) Results must be reported with 2 or more "significant figures" if > RL/LLOQ. If reporting values below the RL/LLOQ, report with 1 or more "significant figures".[3]				
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific RL/LLOQ. (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**	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

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
		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 holding times.				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 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*, 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/LLOQ is estimated due to unacceptable recovery of the lowest standard, the CAM RL/LLOQ 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/LLOQ for the PCB Aroclors or congeners. Sources of various MassDEP standards and criteria are as follows:

- Reportable Quantities (RQs) and Reportable 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: http://www.mass.gov/dep/cleanup/laws/regulati.htm#mcp
- An online searchable Oil & Hazardous Materials List of RQs and RCs values may be found at the following URL: http://eeaonline.eea.state.ma.us/DEP/MOMHL/hazmat.aspx
- An updated list of MCP Method 1 Standards may be found at the following URL: https://www.mass.gov/regulations/310-CMR-4000-massachusetts-contingency-plan

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 Permanent and Temporary Solutions, including Permanent and Temporary Solutions on a portion of a disposal site, for preparation of Representativeness Evaluations and Data Usability Assessments pursuant to 310 CMR 40.1056(2)(k) and 40.1057(2)(k), respectively, 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 a Permanent or Temporary Solution submittal. The most current version of this document may be found at the following URL: http://www.mass.gov/dep/cleanup/laws/policies.htm#finpol.

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**)". Requirements listed as "YES" must always be included as part of the laboratory deliverable for this method. It should be noted that data for those items listed as "NO" under "Required Analytical Deliverables" must be available for review during an audit and may also be requested for inclusion in the analytical deliverable 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/LLOQ for each PCB Aroclor or congener must be adjusted (increased) in direct proportion to the Dilution Factor (DF).

The revised RL/LLOQ for the diluted sample, $RL/LLOQ_d$:

$$RL/LLOQ_d = DF \times \text{Lowest Calibration Standard for Target Analyte}$$

It should be understood that samples with elevated RLs/LLOQs as a result of a dilution may not be able to satisfy MCP standards/criteria in some cases if the $RL/LLOQ_d$ is greater than the applicable MCP standard or criterion to which the concentration is being compared. Such increases in RLs/LLOQs 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 50% of its associated highest calibration standard. This will avoid unnecessarily high RLs/LLOQs 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 ≤ 6ºC 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 ≤ 6ºC 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 ≤ 6ºC or freeze at <-10 ºC [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]<u>If frozen, sampling container should only be filled to 2/3 of capacity to avoid breakage caused by expansion during freezing</u>. Temperature must never be allowed to go below – 20°C to avoid damage to seals, etc. Once the thawing process begins, samples must be kept at 0-6◦C until extraction.

[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 4ºC, 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*, 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 6ºC 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: SW-846 8082A)	
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/response factors for all standards in initial calibration; average calibration/response 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/response 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, re-analyses, and dilutions Quantitation reports for all sample analyses, re-analyses, and dilutions Percent solids results Summary of results, including RLs/LLOQs 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 RLs/LLOQs 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.	