FACILITY SOP ATTACHMENT

SOP NUMBER: IR-GCS-PCBs, Rev. 1 (09/21/12)	**CHANGE FORM ID: CF2**
SOP TITLE: Polychlorinated Biphenyls (PCBs) by GC EPA Methods 608 and 8082	
REASON FOR ADDITION OR CHANGE (Use additional sheets if necessary): To remove section 2) and 3) from CF1 due to Audit Finding	
CHANGE OR ADDITION (Use additional sheets if necessary): No change to section 9.2.3, 10.4.1, 10.5.3, 10.5.5, 12.2.1, 12.2.4	
Prepared By: L. Hoang	
***APPROVED BY:**	
Department Manager	Date 8/5/13
Quality Assurance Manager	Date 8-2-2013
Health and Safety Coordinator	Date 08/05/2013
Laboratory Director	Date 08/05/2013

Control Copy Number _____

FACILITY SOP ATTACHMENT

SOP NUMBER: IR-GCS-PCBs, Rev. 1 (09/21/12)	**CHANGE FORM ID:** CF1
SOP TITLE: Polychlorinated Biphenyls (PCBs) by GC EPA Methods 608 and 8082	

REASON FOR ADDITION OR CHANGE (Use additional sheets if necessary):

1) Specify extract holding time.
2) Removal of requirement for routine (daily) analysis of Aroclors 1221, 1232, 1242, 1248, 1254 and 1268. (Per 2013 Corporate audit recommendation.)
3) Specify retention time window studies are performed only on Aroclors 1016 and 1260.

CHANGE OR ADDITION (Use additional sheets if necessary):

Add to section 8.0:

The holding from completion of extraction to analysis is 40 days.

Change section 9.2.3 (first paragraph only):

Currently:

On a daily basis, verify calibration at the beginning of each 12-hour shift by injecting calibration verification standards (Aroclors 1016, 1260, 1221, 1232, 1242, 1248 and 1254) prior to conducting any sample analysis. A verification must also be checked at intervals of 10 to 20 samples – alternated between two different calibration levels (e.g. 1000 and 2500 ppb) – and at the end of the analysis sequence. CCV standards can be from either the primary or secondary source.

Revise to read:

On a daily basis, verify calibration at the beginning of each 12-hour shift by injecting calibration verification standards for Aroclors 1016 and 1260 only, prior to conducting any sample analysis. Verification must also be checked at intervals of 10 to 20 samples and at the end of the analysis sequence. CCV standards can be from either the primary or secondary source.

Change section 10.4.1

Currently:

Whenever a routine ICAL is performed for any Aroclor, single-points for all other Aroclors will be run at the same time.

Removed. (Full ICALs are analyzed, as needed, for all Aroclors.)

Change section 10.5.3

Currently:

Verify that the instrument blank (hexane) daily is free of contamination. Load and inject a series of CCV standards (1016/1260, 1221, 1232, 1242, 1248, 1254) and ensure that recoveries pass the acceptance limits (+15%). Note that 1221, 1232, 1242, 1248 and 1254 opening CCVs

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SOP NUMBER: IR-GCS-PCBs, Rev. 1 (09/21/12)	**CHANGE FORM ID:** **CF1**

are run on every sequence only as pattern recognition. If the pattern of these Aroclors is found in any samples, the samples must be re-run with passing bracketing CCVs.

Revise to read:

Verify that the instrument blank (hexane) daily is free of contamination. Load and inject a 1016/1260 standard and ensure that recoveries pass the acceptance limits (±15%). If the pattern of Aroclors other than 1016/1260 is found in any samples, the samples must be re-run with passing bracketing CCVs for those other Aroclors.

Change section 10.5.5

Currently

Load and inject a series of CCV standards (1016/1260, 1242, 1248, 1254) every 10 to 20 samples (or every 12 hours) and at the end of the sequence run. Alternate the CCV levels. Verify that they meet the acceptance limits

Revise to read:

Load and inject a 1016/1260 standard every 10 to 20 samples (or every 12 hours) and at the end of the sequence run. Verify that they meet the acceptance limits.

Change section 12.2.4

Currently:

If the standard deviation is less than 0.01 minutes, use a default RT window of 0.03 minutes for regular-level pesticides and an RL window of 0.01minutes for low-level pesticides.

Revise to read:

If the standard deviation is less than 0.01 minutes, use a default RT window of 0.03 minutes.

Change section 12.2.1

Currently:

Retention time window studies are to be performed as often as necessary, whenever a new column is installed, and at a minimum annually.

Revise to read:

Retention time window studies are to be performed as often as necessary, whenever a new column is installed and, at a minimum, annually. Studies are performed only on Aroclors 1016 and 1260.

Prepared By: Dave Dawes with Cornelia Nichols

APPROVED BY: CORNELIA NICHOLS for VALERIE SIERZCHULA

Cornelia Nichols — Technical Review Signature

7-25-13 — Date

FACILITY SOP ATTACHMENT

SOP NUMBER: IR-GCS-PCBs, Rev. 1 (09/21/12)	**CHANGE FORM ID: CF1**
Quality Assurance Manager	Date 7-25-2013
Environmental Health and Safety Coordinator	Date 07/25/2013
Laboratory Director	Date 07/25/2013

Control Copy Number _____

Title: POLYCHLORINATED BIPHENYS (PCBs) by GC EPA Methods 608 and 8082

Approvals (Signature/Date):

Paul Monroy 9/14/12 Date Technical Manager	William Nash 9/14/2012 Date Health & Safety Coordinator
David Dawes 9/13/12 Date Quality Assurance Manager	Fred Haley 9/17/12 Date Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 SCOPE AND APPLICATION

EPA Method 608 is used to determine the concentration of polychlorinated biphenyls (PCBs) in groundwater, surface water, or wastewater samples. EPA Method 8082 is used to determine the concentrations of PCBs as Aroclors (multi-component mixtures) in extracts from solid and aqueous matrices.

This standard operating procedure applies to both EPA Methods 608 and 8082 (for PCBs analysis only).

The common PCBs to be analyzed include:

Aroclor-1016
Aroclor -1221
Aroclor -1232
Aroclor -1242
Aroclor -1248
Aroclor –1254
Aroclor –1260
Aroclor –1268

These methods can be used to analyze generally down to 1.0 µg/L in liquids and 50 µg/Kg in solids. See attached analysis information for detailed reporting and QC limits. NOTE: See LIMS system for current MDL and Control Limit values.

1.1 Differentiation between EPA 608 and EPA 8082

Although overlapping each other in many aspects, 608 method and 8082 method do differ in certain analysis requirements, notably:

- Method 8082 requires a higher number of calibration points (5 vs. 3 in 608).
- Method 608 requires a more stringent calibration factor RSD (10% vs. 20% in 8082).
- Method 8082, in conjunction with 8000B, does NOT allow the calibration curve to be forced through the point of origin.
- Method 608 requires a calibration verification (CCV) daily while Method 8082 specifies it at the beginning of each 12-hour shift.
- Method 608 does NOT allow averaging of calibration factor RSD and ICV/CCV recoveries.
- Method 8082 requires result confirmation from a secondary column.
- Surrogates are discussed only in method 8082.

The more stringent quality control will take precedent if the analysis is to be used for both methods. Additionally, this SOP will include the surrogates and the 12-hour CCV in all cases.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in “Validation of Methods” in the Quality Assurance Manual.

2.0 SUMMARY OF METHOD

Aqueous samples are extracted per Method 3510C. A measured volume of sample, nominally is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less.

Solid samples are extracted with hexane-acetone (1:1) using Method 3546 (microwave extraction). The extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less.

Non-aqueous samples (oils, products) are extracted in accordance with Method 3580A (waste dilution).

The extract is separated by gas chromatography and the parameters are then measured with an electron capture detector (ECD). Multi-component PCBs are identified using retention times and pattern recognition.

3.0 DEFINITIONS

3.1 The primary column is defined as the column that demonstrates the least interference throughout the sequence.

3.2 There are no additional specific definitions associated with this test. See the laboratory QA manual and EPA methods 608 and 8082 for general definitions.

4.0 INTERFERENCES

The following are three broad categories of sources of interferences in EPA Methods 608 and 8082:

4.1 Contaminated solvents, reagents, glassware, or other sample processing hardware. Cross-contamination of clean glassware can easily occur when plastics are handled during extraction, especially when solvent-wetted surfaces are handled. Flexible plastic used during sample preparation can introduce Phthalate esters.

4.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

4.3 Compounds extracted from the sample matrix to which the detector will respond.
For a detailed discussion on cleanup procedures, refer to Section 3.0 of Method 608, or sections 3.0 and 7.2 of Method 8082. Some recommended cleanups are as follows:

- Interferences from **phthalate esters** can be minimized by avoiding contact with any plastic material, and by checking all solvents and reagents for phthalate contamination.
- Phthalate esters can be removed prior to analysis by using Method 3665 (Sulfuric Acid Cleanup). See the EPA 3510 and EPA 3546 standard operating procedures regarding Sulfuric Acid cleanup.
- Elemental sulfur is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. It can be removed by the addition of activated copper powder (EPA method 3660B and EPA 608, section 11.3).

5.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples

and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Personal Protective Equipment required: Safety Glasses/Face Shield, Labcoat, and Nitrile gloves.

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Instrumentation

6.1.1 Gas Chromatograph - AT 5000 and 6000 Series, or equivalent
6.1.2 Column A: RTX-CLP (30m x 0.32mm x 0.5µm) or equivalent
6.1.3 Column B: RTX-CLP2 (30m x 0.32mm x 0.25µm) or equivalent
6.1.4 Column A: Zibron MT1 (30m x 0.25mm x 0.25µm) or equivalent
6.1.5 Column B: Zibron MR2 (30m x 0.25mm x 0.2µm) or equivalent
6.1.6 Electron capture detector (ECD)
6.1.7 Autosampler – AT 7673A, 7683, or equivalent
6.1.8 Injector liners

6.2 Supplies

6.2.1 Assorted volumetric flasks, class A
6.2.2 Assorted micro syringes – 5, 10, 50, 100, 500 and 1000 µl
6.2.3 Assorted glass and minert vials – 2, 10, 40 mL with Teflon-lined screw caps or crimp tops

7.0 REAGENTS AND STANDARDS

7.1 Reagents

All purchased and prepared reagents must be made from a traceable (NIST) source material, if available, and documentation of this traceability must be maintained by the laboratory.

7.1.1 Acetone, pesticide grade or equivalent
7.1.2 n-Hexane, pesticide grade or equivalent.
7.1.3 Methanol, pesticide grade or equivalent.
7.1.4 Copper granules, UCT, Part # ECCUOIK or equivalent

7.2 Standards

All purchased standards must be accompanied by a Certificate of Analysis (C of A) which is kept available at the laboratory in order to demonstrate traceability of the standard to certified (NIST-traceable, if available) source material.
All prepared standards must be made from a traceable (NIST) source material, if available, and documentation of this traceability must be maintained by the laboratory

7.2.1 Pesticides Surrogate Spike solution - 200 µg/ml (Restek or equivalent)
7.2.2 1000 µg/ml Aroclor 1016/1260 standard (Restek or equivalent)
7.2.3 1000 µg/ml Aroclor 1221 standard (Restek or equivalent)
7.2.4 1000 µg/ml Aroclor 1232 standard (Restek or equivalent)
7.2.5 1000 µg/ml Aroclor 1242 standard (Restek or equivalent)
7.2.6 1000 µg/ml Aroclor 1248 standard (Restek or equivalent)
7.2.7 1000 µg/ml Aroclor 1254 standard (Restek or equivalent)
7.2.8 1000 µg/ml Aroclor 1268 standard (Restek or equivalent)

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	1L amber glass	2 bottles	Cool >0 to 6°C*	7 Days	40 CFR Part 136.3 and SW846, Chapter 4
Soils/Oils	4 oz jar	100 g	Cool >0 to 6°C	14 Days	SW846, Chapter 4

* For 608 Only: Water samples shall be extracted within 72 hours of collection or 7 days if pH-adjusted to 5.0 – 9.0 using sulfuric acid or sodium hydroxide. Document the pH on the bench sheet.

9.0 QUALITY CONTROL

9.1 Sample QC

The following quality control samples are prepared with each batch of samples. Each of these QC samples may be re-analyzed once if it doesn't pass, in order to verify the failure wasn't due to a physical or mechanical problem

Note that if the sample extract (s) require any clean up procedure, the associated batch QC (i.e. MB, MS/MSD and LCS must also undergo the same clean up procedure with the sample extract (s).

9.1.1 Method Blank (MB)

Prepare and analyze a method blank (MB) for each matrix and with every batch of 20 samples, or less. Check that there are no analytes detected at or above the reporting limit. If the method blank shows contamination, re-prepare all samples in the batch unless:

- The samples are ND (flag the result accordingly).
- The sample result is > 20x the blank level (flag the result accordingly).

9.1.2 Laboratory Control Sample (LCS)

Prepare and analyze a primary source laboratory control sample (LCS) for every batch of 20 samples or less. The LCS recovery must be within laboratory acceptance limits (see Attachment 1). If the LCS is outside of these limits, re-analyze the LCS once. If the second run of LCS is still outside the acceptance limits, then determine as follows:

- If the analyte(s) in both runs of the LCS is above the acceptance limits and the associated sample results are ND, report the data and flag the results accordingly to indicate the high LCS recovery.
- If the analyte(s) in both runs of the LCS is above the acceptance limits and the associated sample results are positive, re-extract and re-analyze the affected samples with acceptable QC criteria.
- If the analyte in both runs of the LCS is below the acceptance limits, re-extract and re-analyze the affected samples.
- Notify Project Manager immediately if there is insufficient sample left to re-extract. Flag samples results and fill out NCM if sample results are still to be reported with failed QC.

LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

For Oil and Product matrices, LCS/LCSD must be extracted by EPA 3580A.

9.1.3 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

The sample for MS/MSD is randomly selected, unless specifically requested by a client.
Prepare and analyze a matrix spike (MS) and a matrix spike (MSD) duplicate for each matrix and with every batch of 20 samples, or less. The recovery and relative percent difference must be within laboratory acceptance limits (see Attachment 1).

- If the MS/MSD are outside of the acceptance limits due to matrix effect, flag the results accordingly.
- If the MS/MSD are outside of the acceptance limits due to non-matrix related causes (instrument problems, analyst error, etc), re-analyze the samples after taking corrective action. If re-analyzing the MS/MSD is not possible, fill out a NCM with detailed explanation and notify the PM immediately.

9.2 Instrument QC

The following instrument QC samples are run with each analytical sequence. Each of these QC samples may be re-analyzed once if it does not pass, in order to verify the failure wasn't due to a physical or mechanical problem. Re-analysis must be performed before any batch QC or client samples are analyzed.

9.2.1 Instrument Blank

Analyze an instrument blank daily before analyzing samples and, if needed, after analyzing highly concentrated samples. Prepare the instrument blank with hexane. Verify that the instrument blank is free of target analytes before analyzing any samples.

9.2.2 Initial Calibration Verification (ICV)

Immediately after the initial calibration, analyze secondary source verification (ICV) near the mid-point. Verify that its recovery is within ± **15**% of spiked value and the retention times are within their respective accepted windows.

- If not, re-prepare the ICV standard.
- If the ICV is still out of control, re-calibrate the system

9.2.3 Continuing Calibration Verification (CCV)

On a daily basis, verify calibration at the beginning of each 12-hour shift by injecting calibration verification standards (Aroclors 1016, 1260, 1221, 1232, 1242, 1248 and 1254) prior to conducting any sample analysis. A verification must also be checked at intervals of 10 to 20 samples – alternated between two different calibration levels (e.g. 1000 and 2500 ppb) – and at the end of the analysis sequence. CCV standards can be from either the primary or secondary source.

NOTE: Even though EPA Method 608 does not require calibration verification more than once each working day, this SOP complies with the more stringent criteria of EPA Method 8082 regarding the CCV frequency.

Verify that the recovery of CCV is within ±15% of the initial calibration.

- If retention times have shifted slightly, perform the necessary instrument maintenance and/or reset the RT (but not the RT window).
- If the CCV fails the acceptance criteria, re-prepare and reanalyze a fresh standard once.
- If the second CCV also fails, end the analysis sequence. Perform any necessary maintenance (such as replacement of liner, etc.) and start a new sequence, reanalyzing those samples in the previous sequence that were not bracketed by passing CCVs.

If any sample has a detected analyte concentration above the reporting limit and the ICV/CCV percent recovery is outside of the acceptance limits (high or low):

- Reanalyze the sample one time with a passing opening QC.
- If the ending CCV is still out because of matrix effect, flag the results appropriately.

If the CCV result is > 115% of the expected value and all samples are ND for the compound then report the results and flag the data appropriately.

When a particular sample or project causes any CCV to fail the recovery verification two times, matrix interference is confirmed, the data is reported accordingly.

All of the criteria listed above for CCV also applies to the analysis sequence from the confirmation column if it is used to report data.

For Oil and Product matrices (extracted by EPA 3580A), LCS/LCSD may be used as CCVs.

9.2.4 Surrogates

DCBP is used as the surrogate. However TCMX may be reported as the substitute surrogate if, and only if, there is obvious chromatographic matrix interference with the DCBP surrogate (e.g. coelution). Flag the surrogate result with the appropriate NCM.

Calculate the surrogate recoveries for all samples, Blanks and Spikes. Verify that the surrogate recoveries are within in-house control limits. If the recoveries are not within limits, then:

- Verify that there are no errors in calibration, calculations, or surrogate standard solutions in use.
- Check instrument performance.
- Recalculate the data and/or re-analyze the extract if any of the above checks reveals a problem.
- Re-extract and re-analyze the sample if none of the above are a problem or flag the data and provide a NCM.

9.2.5 Calibration Acceptance Summary

Refer to the "Calibration Curves" SOP and the "Selection of Calibration Points" SOP for more information on calibrating the instrument.

- Prepare a calibration curve of a 1016/1260 mix by plotting the response (peak area) of at least a 3 standards against the corresponding concentrations (note that for 8082 the minimum number of standards is 5). The lowest standard must be at or below the reporting limit. Use calibration factors (CFs) to determine acceptability. NOTE that when a different Aroclor is identified in a sample above the reporting limit, perform a full calibration for that Aroclor as well
- For Method 608, the % RSD of the Calibration Factors (CFs) for each target analyte and surrogate must be ≤ 10% for both the primary and secondary columns in order to quantitate the compound using the average CF.
- For Method 8082, the requirement for the % RSD of the CFs is < 20%.
- The surrogates are calibrated at multiple levels at the same time as the PCB standards.
- If the %RSD of the CFs for any Aroclor is above the criteria limit, generate a calibration curve following these guidelines:
    - A first order (linear) or quadratic (non-linear) regression may be used for quantitation.
    - Examine the linearity and the accuracy in quantitating at the low calibration standard and at the point of origin in both options before selecting the better curve (1st order regression may be less accurate than 2nd order near the point of origin).

- For Method 608 only, the calibration curve is allowed to be forced through the origin.
- The Coefficient of Determination (r^2) must be $\geq$ 0.99 (Correlation Coefficient (r) $\geq$ 0.995) for the curve to be acceptable. If r^2 is < 0.99 then the instrument must be recalibrated for that compound.
- When the curve is not forced through the origin, inaccuracies may be present near the low end of the curve. If reporting down to the MDL is required or if the quantitation of a low response (< lowest calibration standard) from a calibration curve results in a negative or abnormally positive result (>RL) evaluate as follows:
  - When evaluating results near the RL review them carefully to ensure they make sense (i.e. no significantly negative values or false hits when the response area is below the lowest standard). If a result is questionable, the sample should be re-analyzed on another instrument or the result reported as estimated.

NOTE that Arizona follows method 8000C. EPA 8000 C does not allow the use of the grand mean RSD to evaluate calibration linearity.

10.0 PROCEDURE

10.1 Standard Preparation

10.1.1 Store standards at >0 °C in PTFE sealed containers, in the dark. Check the solutions frequently for signs of degradation or evaporation. Stock standard solutions must be replaced after one year from the date opened or according to the manufacturer's expiration date or sooner if routine QC indicates a problem. All other standards must be replaced 6 months or sooner if routine QC indicates a problem.

10.1.2 All working standards (surrogates, BS/BSD, MS/MSD spiking standards) used in water samples must be prepared using Acetone instead of Hexane.

10.1.3 Prepare all standards by using a calibrated syringe to deliver the required volume to a volumetric flask. Fill to the mark with the appropriate solvent.

10.1.4 Transfer each solution from the volumetric flasks into two or three 40 ml or 12 ml VOA vials with Teflon-lined screw caps. Store the solutions at 0 to < 6°C and protect from light.

NOTE: When PCBs are to be determined as Aroclors, decachlorobiphenyl (DCBP) is used as the surrogate compound. TCMX is present in the surrogate spiking solution, however, it is not normally utilized for quality control purposes. TCMX can be used as a quality control purposes only if the sample contains 1268 due to the coelution of one of the aroclors peaks and DCBP.

10.1.5 Enter the standard information into the LIMS system. All standard information must be reviewed for accuracy by a peer or department manager before the standard can be used.

10.1.6 Prepare new calibration standards every 6 months, or sooner, if comparison with check standards indicates a problem. Prepare the calibration standards by adding the volumes of spike solutions shown in the following tables (see attachments):

Table Number	Title
Table 1	Aroclor 1016/1260 Standards
Table 2	Aroclor 1254 Standards
Table 3	Aroclor 1248 Standards

Table Number	Title
Table 4	Aroclor 1242 Standards
Table 5	Aroclor 1232 Standards
Table 6	Aroclor 1221 Standards
Table 7	Aroclor 1268 Standards

10.2 Sample Preparation

Sample extracts are received from the Extractions department. Upon receipt:

- Store extracts in crimp-top vials at >0 to 6°C until analysis.
- Verify that the extract has not exceeded its 40 day holding time.
- Sulfur Clean-up. Method 3660 is a clean-up technique that will remove or destroy elemental sulfur from extracted soil or water sample that may cause chromatographic interference in the determination of PCBs.

If a sample is suspected to contain sulfur, add about 0.5 grams of copper metal granules (UCT, Part # ECCUOIK or equivalent) to both the extracted sample and all relevant QC in the batch. Shake the samples in a shaker for 15 min. Let the samples stand in the hood for 5 minutes and then transfer the organic layer into a new vial for analysis. Record in the sample worklist what samples needed the copper clean up and the LIMS standard number for the copper reagent used.

10.3 Instrument Initialization

The following are suggested instrument conditions. These may vary slightly between instruments, because of necessary instrument maintenance (e.g. column trimming) or because of column age.
Detectors: Dual Electron Capture Detector (ECD) or Dual Electron Capture Micro ECD.
Injection Volume: 1 µl
Refer to the following table for additional details:

	Option 2
Column A:	Restek CLP (30m x .32mm ID x 0.25um
Column B:	Restek CLP2 (30 m x .32 mm ID x 0.5um
Helium flow rate:	4 mL/min
ECD make-up Nitrogen:	35 mL/min
Electronic pressure control:	22 psia
Injector t °C	240 °C
Detector t °C	310 °C
Oven initial t °C	140 °C
Initial Hold time	1 minute
Rate 1	30 °C/min
Final Temp 1 °C:	240 °C
Hold time 2:	2 minutes
Rate 2	30 °C
Final temp 2 °C:	300 °C
Final Time 2:	2.67
Run Time:	11 minutes

10.3.1 Verify that the solvent bottles in the autosampler are full, and the solvent waste vials are empty.

10.4 Calibration

10.4.1 Whenever a routine ICAL is performed for any Aroclor, single-points for all other Aroclors will be run at the same time.

10.4.2 For a new calibration, load the calibration standards in ascending concentration order followed by the ICV. Verify all calibration criteria are met before proceeding.

10.4.3 For Method 608, analyze a minimum of three standard points for a working calibration curve.

10.4.4 For Method 8082, use a minimum of five standard points (or six if using a quadratic curve).

10.4.5 Calibrate the primary and secondary columns simultaneously.

10.4.6 Repeat for each different Aroclor.

10.4.7 The surrogates are calibrated in multiples levels at the same time as the PCBs standards.

10.4.8 To continue using a previous calibration, copy the worklist from the previous sequence, analyze an instrument blank and CCVRT standard (midpoint standard) . Verify acceptance criteria are met before proceeding.

10.5 Sample Analysis

10.5.1 Load one or two high level PCB standards to prime the column
10.5.2 Load and inject an instrument blank (IB) after priming.
10.5.3 Verify that the instrument blank (hexane) daily is free of contamination.
Load and inject a series of CCV standards (1016/1260, 1221, 1232, 1242, 1248, 1254) and ensure that recoveries pass the acceptance limits (±15%). Note that 1221, 1232, 1242, 1248 and 1254 opening CCVs are run on every sequence only as pattern recognition. If the pattern of these arochlors is found in any samples, the samples must be re-run with passing bracketing CCVs.
10.5.4 Analyze the method blank (MB) and verify that it is free of contamination (<RL).
10.5.5 Analyze the LCS and verify that it meets the QC limits.
10.5.6 Follow with 10-20 sample extracts.
10.5.7 Load an instrument blank after a dirty extract to prevent carry-over. Instrument blanks (and standards) do not count towards the 10-20 samples between CCVs.
10.5.8 Analyze the matrix spike (MS) and matrix spike duplicate (MSD) sometime during the sequence. The recoveries of the MS/MSD should be within in-house control limits.
10.5.9 Dilute and re-analyze an extract if its response exceeds the instrument calibration range.
10.5.10 Load and inject a series of CCV standards (1016/1260, 1242, 1248, 1254) every 10 to 20 samples (or every 12 hours) and at the end of the sequence run. Alternate the CCV levels. Verify that they meet the acceptance limits.

NOTE: If a sample contains any Aroclor other than 1016/1260, re-process the sequence run against the calibration method of the particular Aroclor. Verify that all QC requirements are met in the same fashion as 1016/1260 method.

1. Prime
2. Prime
3. Instrument Blank (Hexane)
4. CCV1 (1016/1260 & others)
5. Method Blank
6. LCS
7. 10 to 20 samples (icl. MS/MSD)
8. CCV2 (1016/1620 & others)
9. 10 samples
10. CCV1 (1016/1260 & others)

Figure 1 - A typical analysis sequence

10.6 Identification of PCBs

Refer to the "Reporting of multi-component organochlorine analytes" SOP for more Information on reporting multi-component analytes.

10.6.1 PCB identification is based on the agreement of retention times of the peaks present in the sample chromatogram and those of the suspected Aroclor standard and the use of pattern recognition.

10.6.2 Visually compare the sample extract chromatogram to the chromatogram of the most similar Aroclor standard. Overlay and expand the chromatograms using the software's "compare" feature. Check for retention time shifting by verifying that the surrogates retention times correspond. Expanding the baseline may help in the identification.

10.6.3 If a sample contains interferences as elemental sulfur and the identification is difficult, perform a copper clean-up using method 3660 and reanalyze the sample.

10.6.4 When samples contain more than one Aroclor and/or Aroclors in samples that have been subjected to weathering, a higher level of analytical expertise (Supervisor, Technical Director, etc.) is required to attain acceptable levels of qualitative and quantitative analysis.

10.6.5 Positive Aroclor results must be verified by a confirmation column.

- If the Aroclor is confirmed, verify that the result from the confirmation column agrees with the primary column (RPD $\leq$ 40%). Otherwise, check for co-elution or other error.
- If there is an explanation for the discrepancy in quantitated results, report the result from the unaffected column.
- If no discrepancy is found, re-run the sample on a different instrument or report the lower of the two results.
- **NOTE**: Confirmation analysis for detected analytes on a secondary column is only required by Method 8082, but strongly recommended for all Aroclor analyses.

10.7 Quantitation of PCBs

10.7.1 Routinely, 5 major characteristic peaks (4 peaks for 1221) are chosen to quantitate each Aroclor. Choose peaks that are at lease 25% of the height of the highest peak for a given Aroclor.

10.7.2 For 1016 and 1260 Aroclors, do not use shared peaks. Each Aroclor must be quantitated against a unique set of 5 peaks (4 peaks for 1221).

10.7.3 Calculate the calibration factor (CF) for each chosen peak in each of the initial calibration standards using the following equation:

$$CF = \frac{\text{Peak Area in the Standard}}{\text{Standard Concentration}}$$

10.7.4 Each calibration level will generate a set of 5 CFs (4 for 1221) corresponding to the chosen peaks. Therefore, ten sets of CFs will be generated for the 1016/1260 mixture

10.7.5 The single standard for each of the other Aroclors will generate 5 CFs and 4 CFs for 1221. The resulting peak concentrations are then averaged to determine the final PCB concentration.

10.7.6 Once a PCB (other than 1016 or 1260) has been identified in the sample, the sample is re-analyzed against a calibration curve of the identified Aroclor. The sample result is quantitated in the same way as Aroclors 1016 and 1260.

10.7.7 If multiple Aroclors are present in the sample, calculate all that can be positively identified on the chromatogram using at least three characteristic peaks that best fit with the PCB standard pattern.

10.7.8 If interfering peaks in the samples matrix necessitate the use of fewer than 5 peaks (4 for 1221) to quantitate an Aroclor, the data will be qualified to indicate "Due to matrix interference, the data was reprocessed with only X peaks in the primary and/or confirmation column."

10.7.9 Complex Chromatograms

- When there are numerous PCB peaks present but there are no good matches to any individual Aroclor, choose the Aroclor (or Aroclors) that most closely match the sample and quantify the peaks as that Aroclor. Multiple Aroclors should only be reported if their patterns are reasonably well separated. For example, 1232 and 1254 could be reported together, but not 1242 and 1248.
- When reporting and quantifying PCBs that do not closely match an Aroclor standard, it is absolutely essential and mandatory that this is explained in the report narrative.

10.8 Preventative Maintenance

10.8.1 To prevent carryover from a dirty extract, load one or two instrument blanks immediately after all dirty extracts.

10.8.2 If an instrument is unusable or has limitation to its use (bad signal, not for low level samples, etc), it must be tagged accordingly until such a time the problem has been corrected.

10.8.3 Record the problem, solution and verification of proper operation into the instrument maintenance logbook.

10.8.4 Record all performed maintenance in the instrument maintenance logbook.

11.0 CALCULATIONS / DATA REDUCTION

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Concentration

Water and Soil Samples:

$$C_f = C_i \times PF \times DF$$

where C_f = Final concentration in µg/L or µg/Kg
C_I = Concentration in µg/L from instrument
PF = Preparation Factor
DF = Any additional Dilution Factor (post-extraction)

11.4 Percent Difference

$$\% \text{ Difference} = \frac{[\text{Apparent conc}(\mu g/L) - \text{True conc } (\mu g/L)] \times 100}{\text{True conc } (\mu g/L)}$$

12.0 METHOD PERFORMANCE

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure as described in laboratory's SOP, IR-QA-MDL. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

Repeat the study after extensive instrument maintenance or a significant change in the method. Perform a method detection limit study by analyzing at least seven extracts preferably over two different days.

12.2 Retention Time Window Study

12.2.1 Retention time window studies are to be performed as often as necessary, whenever a new column is installed, and at a minimum annually.

12.2.2 Perform a retention time window study on each instrument and column by analyzing a triplicate run of a check standard (ICV/CCV) over a 72-hour period.

12.2.3 Calculate the mean and standard deviation of the retention time for each analyte and surrogate to three decimal places. The width of each retention time window is then calculated as three times the standard deviation.

12.2.4 If the standard deviation is less than 0.01 minutes, use a default RT window of 0.03 minutes for regular-level pesticides and an RL window of 0.01minutes for low-level pesticides.

12.2.5 NOTE: The experience of the analyst must be used in conjunction with the RT window calculations to ensure an appropriate window is established, one that minimizes the occurrence of both false positive and false negative results.

12.3 Demonstration of Capabilities

Every analyst must perform an Initial Demonstration of Capability (IDOC) before performing analyses on any client samples. An IDOC can be 1) 4 consecutive LCS samples (prepared from a source other than that used for the ICAL) with an average recovery and RSD within the in-house statistical limits, or 2) passing results on a blind or PT study.

12.4 Training Requirements

The analyst must have documented training, including reading of the SOP and source methods, conducted by the department manager, senior chemist, or other analyst with training documentation and a passing DOC.

13.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in the "Waste Management and Pollution Prevention" section of the Corporate Environmental Health and Safety Manual (CW-E-M-001).

14.0 WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the laboratory's Waste Disposal SOP (IR-EHS-WASTE). The following waste streams are produced when this method is carried out:

- **Autosampler vials** – Once the analysts have reported the sample results, they store the autosampler vials in the appropriate refrigerator in the semivolatiles area. After 40 day minimum that the vials have been stored, the analysts remove the vials and disposed them into the Step-on waste container located in the extractions lab. The Step-on container is label "Autosampler vials". Waste bulked as autosampler vials.
- **Hexane/Acetone waste**. This waste is generated when analysts prepare sample and standards. The waste is store in a 4 L bottle, the bottle is placed inside the fume hood. Waste bulked as mixed flammable solvents.
- **PCBs waste** – After the 40 day of storage, the extracts with PCB concentrations of >50ppm are placed in plastic container label as "Hazardous waste". The container is taken by the analyst into the main waste storage area and placed in the appropriate shelf labeled "PCB haz waste".
- **Unused standards**. If hazardous standards cannot be collected with one of the waste streams generated in the method, then analyst and technicians take this standard and placed it on the shelves labeled "hazardous waste" in the main waste storage area. The standard will be lab packed (example: mercury standard). If the standard can be collected in the satellite waste container for one of the waste streams of the method, then pour the standard in the right satellite container, rinse the original container, and collect the rinsate in the satellite container. The original container can be placed in the regular trash.

15.0 REFERENCES / CROSS-REFERENCES

15.1 EPA Method 608, 40 CFR part 136, Appendix A
15.2 EPA Method 8082, EPA SW-846 Update III, December 1996
15.3 EPA Method 8000B, EPA SW-846 Update III, December 1996

15.4 EPA Method 8000C, EPA SW-846 Update IV, Revision 3, March 2003
15.5 EPA Method 3660B, Rev 2, December 1996
15.6 Arizona DHS Information Update #37, June 13, 1997
15.7 CA-Q-S-005, Calibration Curves (General)
15.8 CA-T-P-002, Selection Of Calibration Points
15.9 CA-Q-QM-003 Reporting of multi-component organochlorine analytes
15.10 CA-T-P-003_r1 Reporting results for methods that require second-column confirmation

16.0 METHOD MODIFICATIONS

Item	Method	Modification
1	EPA 608 Section 7.2.1	EPA Method 608 requires a calibration verification daily while EPA Method 8082 specifies it at the beginning of each 12-hour shift. The laboratory complies with the more stringent criteria specified in EPA method 8081A for this SOP.
2	No reference	Surrogate spikes are not discussed in EPA Method 608. The laboratory follows the criteria specified in EPA Method 8082 in this SOP
3	No reference	EPA Method 608 does not require the use of confirmation from a secondary column. The laboratory follows the criteria specified in EPA 8082 in this SOP
4	No reference	Multipoint components are calculated based on the area count of 3-5 peaks rather than the sum of the area of all peaks. This minimizes potential co-elutions adding to the areas and is consistent with EPA 8082.
5	EPA 8000B. Section 7.10.4	The reference method indicates to report the higher result in instances where the RPD between the results of the two columns is >40%. The laboratory follows the TestAmerica approach of reporting the lower result which is in alignment with 8000C.

17.0 ATTACHMENTS

17.1 **Attachment 1**: Analysis Information
17.2 **Attachment 2**: Datatypes
17.3 **Attachment 3**: Standard Prep Tables
17.4 **Attachment 4**: ICAL Review Checklist
17.5 **Attachment 5**: Data Review Checklist

18.0 REVISION HISTORY

18.1 **Revision 0, dated 29 January 2010**

- New Testamerica standardized format
- This revision supersedes PCBs.SOP, revision 0, 07/20/07
- Deleted the calibration requirement that RSDs > 50% cannot be used

18.2 Revision 1, dated 21 September 2012

- Added copper clean-up procedure
- Added datatypes
- Clarified ICAL frequency
- Clarified CCV Aroclor rotation
- Updated ICAL and daily checklists
- Remove references to criteria for combined methods
- Changed requirement of reporting the higher result to report the lower when the RPD between the two channels is greater than 40% and there is no evidence of chromatographic problems.
- Added reference to Calibration curves, Selection of calibration points and Reporting results for methods that require second-column confirmation corporate SOPs.
- Prepared by DD, CN and AS

Attachment 1a
Analysis Information

TestAmerica Irvine

8/24/2012

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8082/608 - PCBs in Water (EPA 8082 & EPA 608)								
Preservation: 4 C, Cool								
Container: 1 L Amber		**Amount Required:** 2000 ml				**Hold Time:** 7 days		
Aroclor 1016	0.25	1.0 ug/l			45 - 120	30	50 - 115	30
Aroclor 1221	0.25	1.0 ug/l						
Aroclor 1232	0.25	1.0 ug/l						
Aroclor 1242	0.25	1.0 ug/l						
Aroclor 1248	0.25	1.0 ug/l						
Aroclor 1254	0.25	1.0 ug/l						
Aroclor 1260	0.25	1.0 ug/l			55 - 125	25	60 - 120	25
surr: Decachlorobiphenyl			45 - 120					

TestAmerica Irvine

8/24/2010

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8082 - PCBs in Soil (EPA 8082)								
Preservation: 4 C, Cool								
Container: 4 oz Jar		**Amount Required:** 100 grams				**Hold Time:** 14 days		
Aroclor 1016	6.7	50 ug/kg			50 - 120	30	65 - 115	30
Aroclor 1221	6.7	50 ug/kg						
Aroclor 1232	6.7	50 ug/kg						
Aroclor 1242	6.7	50 ug/kg						
Aroclor 1248	6.7	50 ug/kg						
Aroclor 1254	6.7	50 ug/kg						
Aroclor 1260	6.7	50 ug/kg			50 - 125	30	65 - 115	30
surr: Decachlorobiphenyl			45 - 120					

Attachment 1b
Analysis Information

TestAmerica Irvine

8/24/2012

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8082/608-PCB, Low in Water (EPA 8082 & 608)								
Preservation: 4 C, Cool								
Container: 1 L Amber		**Amount Required:** 2000 ml			**Hold Time:** 7 days			
Aroclor 1016	0.25	0.50 ug/l			45 - 120	30	50 - 115	30
Aroclor 1221	0.25	0.50 ug/l						
Aroclor 1232	0.25	0.50 ug/l						
Aroclor 1242	0.25	0.50 ug/l						
Aroclor 1248	0.25	0.50 ug/l						
Aroclor 1254	0.25	0.50 ug/l						
Aroclor 1260	0.25	0.50 ug/l			55 - 125	25	60 - 120	25
surr: Decachlorobiphenyl			45 - 120					

Attachment 2
Datatypes

Method Code	Level	Datatype Description	Value to Enter	Units
8082	**BATCH**	**Analysis comment**	**If needed**	**NONE**
8082	ANALYSIS	Injection volume	n/a	uL
8082	**ANALYSIS**	**Batch Comment**	**If needed**	**NONE**
8082	ANALYSIS	Final weight/volume of sample	n/a	N/A
8082	ANALYSIS	Initial weight/volume of sample	n/a	N/A
8082	BATCH	Perform Calculation (0=No, 1=Yes)	n/a	NONE
608_PCB	**ANALYSIS**	**Analysis comment**	**If needed**	**N/A**
608_PCB	**ANALYSIS**	**Batch Comment**	**If needed**	**mL**
608_PCB	BATCH	Final weight/volume of sample	n/a	N/A

Attachment 3a
Table 1: Aroclor 1016/1260 Standards

Ar 1016-1260 Mother Cal **1st source**

PCB AR 1016/1260 1000 µg/mL (mL)	Volume of surr stock 200 µg/mL (mL)	Bring to Volume	Final Conc (ppb)
1.25	0.625	25mL	50K/5K

PCB calibration

Calibration	Volume of Mother soln (mL)	Bring to Volume (mL)
125/12.5ppb	0.0625	25
250/25ppb	0.1250	25
500/50ppb	0.2500	25
1K/100ppb	0.5000	25
2.5K/250ppb	1.2500	25
4K/400ppb	2.0000	25
6K/600ppb	3.0000	25

PCB Cal ICV STD 2nd source

Volume of PCB AR1016/1260 1000 µg/mL (mL)	Volume of surr. Stock 200 µg/mL (mL)	Bring to Volume (mL)	Final Conc (ppb)
0.05	0.025	50	1K/100

PCB Cal CCV1 STD 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc (ppb)
2	100ml	1K/100

PCB Cal CCV2 STD 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc (ppb)
5	100	2.5K/250

PCB LCS/SPIKE Soln 1st source

Volume of PCB AR1016/1260 1000 µg/mL (mL)	Bring to Volume (mL)	Final Conc (ppb)
2	500	4K

PCB MDL Spike 1st Source

Volume of PCB AR1016/1260 1000 µg/mL (mL)	Bring to Volume (mL)	Final Conc (ppb)
0.025	50	500

Attachment 3b
Table 2: Aroclor 1254 Standards

AR 1254 Mother Cal std 1st source

Vol 1254 stock (1000 µg/mL)	Volume 8080 Surr stock (mL)	Bring to Volume (mL)	Final Conc.
1.25 mL	0.625 mL	25	50K/5K

AR 1254 Calibrations 1st source

Calibration	Volume Mother soln (ml)	Bring to Volume (mL)
125/12.5ppb	0.0625 mL	25 mL
#1 - 250/25ppb	0.125 mL	25 mL
#2 - 500/50ppb	0.25 mL	25mL
#3 - 1K/100ppb	0.5 mL	25mL
#4 - 2.5K/250ppb	1.25 mL	25mL
#5 - 4K/400ppb	2 mL	25mL
#6 - 6K/600ppb	3 mL	25mL

AR 1254 Cal ICV std 2nd source

Volume 1254 stock (200 µg/mL)	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
0.125 mL	0.0125 mL	25	1K/100PPB

AR 1254 Cal CCV1 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
1	50	1K/100PPB

AR 1254 Cal CCV2 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
2.5	50	2.5K/250PPB

AR 1254 MDL Spike 1st source

Volume 1254 stock (mL)	Bring to Volume (mL)	Final Conc (ppb)
0.025	50	500

Attachment 3c
Table 3: Aroclor 1248 Standards

AR 1248 Mother Cal std 1st source

PCB AR 1248 1000 µg/mL	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
1.25 mL	0.625 mL	25	50K/5K

AR 1248 Calibrations 1st source

Calibration	Volume Mother soln (mL)	Bring to Volume (mL)
125/12.5ppb	0.0625 mL	25 mL
#1 - 250/25ppb	0.125 mL	25 mL
#2 - 500/50ppb	0.25 mL	25 mL
#3 - 1K/100ppb	0.5 mL	25 mL
#4 - 2.5K/250ppb	1.25 mL	25 mL
#5 - 4K/400ppb	2 mL	25 mL
#6 - 6K/600ppb	3 mL	25 mL

AR 1248 Cal ICV std 2nd source - Supelco

Vol 1248 stock (200 µg/mL)	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
0.125mL	0.0125mL	25	1K/100PPB

AR 1248 Cal CCV1 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
1	50	1K/100PPB

AR 1248 Cal CCV2 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
2.5	50	2.5K/250PPB

AR 1248 MDL Spike 1st source

Volume 1248 stock (mL)	Bring to Volume (mL)	Final Conc (ppb)
0.025	50	500

Attachment 3d
Table 4: Aroclor 1242 Standards

AR 1242 Mother Cal std 1st source

PCB AR 1242 1000 µg/mL	V0lume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
1.25 mL	0.625ml	25	50K/5K

AR 1242 Calibrations 1st source

Calibration	Volume Mother soln (ml)	Bring to Volume (ml)
125/12.5ppb	0.0625 mL	25 mL
#1 - 250/25ppb	0.125 mL	25 mL
#2 - 500/50ppb	0.25 mL	25 mL
#3 - 1K/100ppb	0.5 mL	25 mL
#4 - 2.5K/250ppb	1.25 mL	25 mL
#5 - 4K/400ppb	2 mL	25 mL
#6 - 6K/600ppb	3 mL	25 mL

AR 1242 Cal ICV std 2nd source - Supelco

Volume 1242 stock (200 µg/mL)	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
0.125 mL	0.0125 mL	25	1K/100PPB

AR 1242 Cal CCV1 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
1	50	1K/100PPB

AR 1242 Cal CCV2 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
2.5	50	2.5K/250PPB

AR 1242 MDL Spike 1st source

Volume 1242 stock (mL)	Bring to Volume (mL)	Final Conc (ppb)
0.025	50	500

Attachment 3e
Table 5: Aroclor 1232 Standards

AR 1232 Mother Cal std 1st source

PCB AR 1232 1000 µg/ml	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
1.25 mL	0.625 mL	25	50K/5K

AR 1232 Calibrations 1st source

Calibration	Volume Mother soln (ml)	Bring to Volume (ml)
125/12.5ppb	0.0625 mL	25 mL
#1 - 250/25ppb	0.125 mL	25 mL
#2 - 500/50ppb	0.25 mL	25 mL
#3 - 1K/100ppb	0.5 mL	25 mL
#4 - 2.5K/250ppb	1.25 mL	25 mL
#5 - 4K/400ppb	2 mL	25 mL
#6 - 6K/600ppb	3 mL	25 mL

AR 1232 Cal ICV std 2nd source - Supelco

Volume 1232 stock (200 µg/mL)	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
0.125 mL	0.0125 mL	25	1K/100PPB

AR 1232 Cal CCV1 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
1	50	1K/100PPB

AR 1232 Cal CCV2 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
2.5	50	2.5K/250PPB

AR 1232 MDL Spike 1st source

Volume 1232 stock (mL)	Bring to Volume (mL)	Final Conc (ppb)
0.025	50	500

Attachment 3f
Table 6: Aroclor 1221 Standards

AR 1221 Mother Cal std 1st source

PCB AR 1221 1000 µg/mL	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
1.25 mL	0.625 mL	25	50K/5K

AR 1221 Calibrations 1st source

Calibration	Volume Mother soln (mL)	Bring to Volume (mL)
125/12.5ppb	0.0625 mL	25 mL
#1 - 250/25ppb	0.125 mL	25 mL
#2 - 500/50ppb	0.25 mL	25 mL
#3 - 1K/100ppb	0.5 mL	25 mL
#4 - 2.5K/250ppb	1.25 mL	25 mL
#5 - 4K/400ppb	2 mL	25 mL
#6 - 6K/600ppb	3 mL	25 mL

AR 1221Cal ICV std 2nd source - Supelco

Volume 1221 stock (200 µg/mL)	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
0.125 mL	0.0125 mL	25	1K/100PPB

AR 1221 Cal CCV1 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
1	50	1K/100PPB

AR 1221 Cal CCV2 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
2.5	50	2.5K/250PPB

AR 1221 MDL Spike 1st source

Volume 1221 stock (mL)	Bring to Volume (mL)	Final Conc (ppb)
0.025	50	500

Attachment 3g
Table 7: Aroclor 1268 Standards

AR 1268 Mother Cal std 1st source

PCB AR 1268 1000 µg/mL	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
0.5 mL	0.25	10	50K/5K

AR 1268 Calibrations 1st source

Calibration	Volume Mother soln (mL)	Bring to Volume (mL)
125/12.5ppb	0.025 mL	10
#1 - 250/25ppb	0.05 mL	10
#2 - 500/50ppb	0.1 mL	10
#3 - 1K/100ppb	0.2 mL	10
#4 - 2.5K/250ppb	0.5 mL	10
#5 - 4K/400ppb	0.8 mL	10
#6 - 6K/600ppb	1.2 mL	10

AR 1268 Cal ICV std **2nd Source (o2si)**

Volume 1268 stock (1000 µg/mL)	Volume of surr. Stock 200 µg/mL	Bring to Volume (mL)	Final Conc.
0.01	0.005	10	1K/100PPB

AR 1268 Cal CCV1 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
1	50	1K/100PPB

AR 1268 Cal CCV2 std 1st source

Volume of Mother soln (mL)	Bring to Volume (mL)	Final Conc.
2.5	50	2.5K/250PPB

AR 1268 MDL Spike 1st source

Volume 1242 stock (mL)	Bring to Volume (mL)	Final Conc (ppb)
0.025	50	500

Attachment 4
ICAL Review Checklist

GC INITIAL CALIBRATION CHECK LIST
EPA 8082 & EPA 608 – Polychlorinated Biphenyls (PCBs)

Analyst:	_____	2nd Level Review:	_____
Calibration Date:	_____	Date:	_____
Calibration Worklist #:	_____	GC #:	_____

Analyst Rev	2nd Level Rev	
_____	_____	Minimum 5-point calibration – lowest standard at Reporting Limit
		Calibration:
_____	_____	• For 8082, average RSD ≤ 20% for each Aroclor
_____	_____	• For 608, average RSDs≤ 10% for each Aroclor
		If average RSD does not meet method requirements, then generate a linear curve, $r^2 \geq 0.99$
_____	_____	**2nd Source ICV:**
		• Immediately after initial calibration
_____	_____	• %Recovery: 85 – 115
		Check and verify calibration data for:
_____	_____	• Correct retention times
_____	_____	• All peaks are identified
_____	_____	• Saturated chromatographic peaks
_____	_____	• Manual integration
_____	_____	• All graphics uploaded
_____	_____	• P flags checked

Comments: _____

G:\DATA_REV\CHCKLIST\GC_SV\PCB/8082_Cal_R3
Rev 8/20/2012

Attachment 5
Data Review Checklist

DAILY DATA CHECKLIST
EPA 8082 & EPA 608 – Polychlorinated Biphenyls (PCBs)

Analyst:	_____	2nd Level Review:	_____
Analysis Date:	_____	Review Date:	_____
GC #:	_____	Primary Channel (A/B):	_____
Prep Batches:	_____	Confirm. Channel (A/B):	_____
Analytical Batches:	_____		

Analyst Rev	2nd Level Rev	
		ICV/CCV (1rst or 2nd source)
_____	_____	• Beginning of every 12-hour shift, every 10-20 samples and at the end of analysis
_____	_____	• Two different levels during the daily analysis
_____	_____	• %Recovery = 85 – 115
_____	_____	Instrument blank before sample analysis : < Reporting limit
_____	_____	Method blank every extraction batch: < Reporting Limit
_____	_____	MS/MSD every extraction batch of 20 samples or less (refer to in-house limits in LIMS system)
_____	_____	LCS every extraction batch of 20 samples or less (refer to in-house limits in LIMS system)
		All samples checked for:
_____	_____	• Dilution Factor
_____	_____	• Manual integration
_____	_____	• Surrogates within limits (refer to in-house limits in LIMS system)
_____	_____	• Precision between columns: <= 40% (or otherwise justified)
_____	_____	• Frequency of 10 (recommended) to 20 between compliant ICV/CCV
_____	_____	• All graphics uploaded
		Documentation:
_____	_____	• All standards used are uniquely identified and are not expired
_____	_____	• All data flags correctly applied and NCMs written, as required
_____	_____	• Run logs printed

Comments: _____

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Rev 8/20/2012