LIST OF TABLES

APPENDICES

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APPENDICES

APPENDIX 5

STANDARD OPERATING PROCEDURE (SOP) GEHR8082

1.0 Title: General Electric (GE) Hudson River Design Support Sediment Sampling and Analysis Program Standard Operating Procedure for the analysis of Polychlorinated Biphenyls (PCBs) by SW-846 Method 8082

Capillary Column

Standard operating procedure for the analysis of Polychlorinated Biphenyls by Gas Chromatography with Electron Capture Detection and Total Aroclor Quantification.

(Acknowledgment: This SOP is based substantially on internal method SOPs provided by Northeast Analytical, Inc. of Schenectady, N.Y.)

2.0 Purpose

The purpose of this SOP is to provide a detailed written document for measurement of Polychlorinated Biphenyls (PCBs) according to SW-846 Method 8082 specifications.

3.0 Scope

3.1 This SOP is applicable to the determination and quantification of PCBs as outlined in EPA SW-846 Method 8082 for the GE Hudson River Design Support Sediment Sampling and Analysis Program. It is applicable to the sediment/solid samples.

3.2 The following compounds can be determined by this method:

Compound	CAS Number
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

3.3 In general, samples are extracted, with a pesticide-grade solvent. The extracts are further processed by concentrating or diluting, depending on the PCB concentration, and carried through a series of clean-up techniques. The sample is then analyzed by injecting the extract onto a gas chromatographic system and detected by an electron capture detector.

3.4 This SOP provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs by gas chromatography. Sediment extraction procedures are covered in separate standard operating procedures.

4.0 Comments

4.1 One of the major sources of interference in the analysis of PCBs is that organochlorine pesticides are co-extracted from the samples. Several of the commonly found pesticides and associated degradation products (DDT, DDE, DDD) overlap the PCB profile envelope and co-elute with several PCB GC peaks and therefore cannot be accurately measured. The analyst must be careful in chromatographic pattern review and flag peaks that are suspected

of being contaminated so that they are not included in the total PCB values generated.

4.2 Laboratory contamination can occur by introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification.

5.0 Safety

5.1 Safety glasses and disposable gloves must be worn when handling samples and extracts.

5.2 All manipulations of sample extracts should be conducted inside a chemical fume hood. The analyst should minimize manipulation of sample extracts outside of a fume hood.

5.3 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions of handling applicable solvents and chemicals used to process samples. The analyst should refer to the project laboratory's internal chemical hygiene plan for further safety information.

5.4 Samples remaining after analysis should be disposed of through the project laboratory's internal disposal plan. Refer to the project laboratory's internal standard operating procedures for disposal of laboratory waste.

6.0 Requirements

6.1 Extensive knowledge of this standard operating procedure and SW-846 Method 8082 is required.

6.2 The analysis portion of this method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

7.0 Equipment

7.1 Instrumentation

7.1.1 Gas chromatograph: Varian Model 3400 or equivalent, equipped with Model 1077 split/splitless injector or equivalent, temperature programmable oven, electron capture detector, and Model 8100 autosampler or equivalent.

7.1.1.1 Column - A 30 meter, 0.25 mm ID, 0.25-micron phase DB-1 capillary column is used for analysis.

7.1.2 Chromatographic Data System: A data system for measuring peak height and peak area. A Millennium_32 computer network based workstation (Waters Associates) or equivalent, will be employed to capture detector response and digitally store the chromatographic information. This system will allow for chromatographic review of data from the gas chromatograph, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

7.2 Glassware and Accessories

7.2.1 25-mL volumetric flasks, Class A, (Baxter Cat. No. F4635-25 or equivalent)

7.2.2 5-mL volumetric flasks, Class A, (Baxter Cat. No. F4635-5 or equivalent)

7.2.3 10-mL volumetric flasks, Class A, (Baxter Cat. No. F4635-10 or equivalent)

7.2.4 50-mL volumetric flasks, Class A, (Baxter Cat. No. F4635-50 or equivalent)

7.2.5 100-mL volumetric flasks, Class A, (Baxter Cat. No. F4635-100 or equivalent)

7.2.6 4-dram vials for sample extract storage (Kimble Opticlear, part no. 60910, code no. 60910-4 or equivalent)

7.2.7 8-dram vials for sample extract storage (Kimble Opticlear, part no. 60910, code no. 60910-8 or equivalent)

7.2.8 Pasteur pipettes (Kimble, part no. 72050 or equivalent)

7.2.9 250-mL beakers, glass (Baxter Cat. No. B2650-250 or equivalent)

7.2.10 100-mL beakers, glass (Baxter Cat. No. B2650-100 or equivalent)

7.2.11 Disposable 10-mL pipettes (Baxter P4650-110 or equivalent)

7.2.12 Disposable 5-mL pipettes (Baxter P4650-15 or equivalent)

7.2.13 Disposable 1.0-mL pipette (Baxter P4650-11X or equivalent)

7.3 Chemicals

7.3.1 Pesticide-Grade Hexane, Burdick and Jackson, (Cat. No. 216-4) or equivalent

7.3.2 Pesticide-Grade Acetone, Burdick and Jackson, (Cat.No.010-4) or equivalent

7.3.3 Pesticide-Grade Toluene, Baker, (Cat. No. 9336-03) or equivalent

7.3.4 Pesticide-Grade Methylene Chloride, Burdick and Jackson, (Cat. No. 300-4) or equivalent

7.4 Analytical Standard Solutions

7.4.1 Aroclor Stock Standard Solutions

7.4.1.1 Polychlorinated Biphenyls - Neat commercial material for standard preparation. These materials are multi-component mixtures of PCB congeners and are the actual materials that were used in products such as electric power transformers and capacitors. Commercially-prepared stock standards can be used if they are certified by the manufacturer or by an independent source and traceable to National Standards of Measurement.

7.4.1.2 Stock standards are prepared from individual Aroclor formulations by weighing an exact amount of the neat material to the nearest 0.1 mg, and dissolving and diluting to volume in a 100 mL volumetric flask with hexane. See Attachment A, Table 1 for an example of exact weights of each compound. For decachlorobiphenyl (DCB), dissolve

neat formulation in 10 mL of toluene and then transfer to a 100 mL volumetric flask bringing to volume with hexane. Alternatively, commercially-prepared stock standards may be used providing they are traceable to National Standards of Measurement.

7.4.1.3 The stock standards are transferred into Boston bottles and stored in a refrigerator at 0-6°C, protected from light.

7.4.1.4 Stock PCB standards must be replaced after one year, or sooner if comparison with certified check standards indicate a problem. See 8.5.3 for limits.

7.4.1.5 The labeling and tracking of standards should be in accordance with the project laboratory's internal standard operating procedures for preparation of standards. Labeling of standards should also be in accordance with NELAC standards, section 5.10.5.

7.4.2 Calibration Standards

7.4.2.1 Calibration standards are prepared at five concentration levels using a prepared working standard. See Attachment A, Table 2 for an example of the preparation and exact concentrations of the working standards. The following five standards make

up the initial calibration curve standard set for each of Aroclor-1221, Aroclor-1242, and Aroclor-1254: 20 ng/mL, 100 ng/mL, 250 ng/mL, 500 ng/mL, and 1000 ng/mL. One calibration standard at 50 ng/mL which is below the reporting limit (80 ng/mL) will be prepared for each of Aroclor-1016, Aroclor-1232, Aroclor-1248, and Aroclor-1260 (unless observed to be present in a project sample which would require recalibration for the detected Aroclor at the five standard levels used for Aroclor-1221, Aroclor-1242, and Aroclor-1254).

7.4.2.2 The two surrogates tetrachloro-*meta*-xylene (TCMX) and DCB are included in the Aroclor-1254 calibration standards. The stock standard for TCMX is prepared by diluting 1 mL of TCMX solution (ULTRA, cat. #IST-440 or equivalent, at 2000 μg/mL) into a 100-mL volumetric flask resulting in a solution of TCMX at 20 ppm.

7.4.2.3 To prepare the working surrogate standard, pipet 5.0 mL of the 100ppm DCB stock standard and 2.5 mL of the 20 ppm TCMX stock standard into a 100 mL volumetric flask and bring to volume with hexane. The final concentration of this solution will be 5.0 ppm of DCB and 0.5 ppm of TCMX.

7.4.2.4 Refer to Attachment A, Table 4 for an example of the instructions on preparation of the calibration standards

containing Aroclor-1254 and the surrogates. Refer to Attachment A, Table 3 for an example of the instructions on preparing the remaining calibration standards.

7.4.2.5 Transfer all calibration standards to 8-dram vials (or equivalent) and store in a refrigerator at 0-6°C, protected from light. Calibration standards must be replaced after six months, or sooner, if comparison with check standards indicates a problem. See 8.5.3 for acceptance limits.

7.4.3 Continuing Calibration Check Standards

7.4.3.1 Continuing calibration check standards are prepared independently from calibration standards, by using an alternate source purchased from standard vendors. Continuing calibration check standards will be prepared for Aroclor-1221, Aroclor-1242, and Aroclor-1254 (and other Aroclors, if detected). All continuing calibration check standards will contain the surrogate compounds TCMX and DCB. Refer to Attachment B, Tables 1-3 for instructions on preparation of these standards.

8.0 Procedure

8.1 Gas Chromatographic Operating Conditions

8.1.1 Establish the gas chromatograph (GC) operating parameters as follows:

Autosampler parameters: Multi-vial mode, ECD Attenuation and range are 1.

Refer to Attachment C for all other GC operating procedures.

Note: GC helium gas flow is optimized after instrument maintenance by adjusting nitrogen flow to elute a PCB calibration standard to a known retention time.

8.2 Data Acquisition

8.2.1 Chromatographic information will be collected and processed utilizing a computer based data acquisition workstation (Waters Associates, Millennium_32 computer network based workstation or equivalent). The GC workstation acquires the millivolt detector signal, performs an analog to digital conversion and stores the digital chromatogram on the computer network's disk. The chromatography software performs all data reduction including, long term data storage on magnetic media, chromatographic peak integration, all calculations, report generation, chromatogram plots, and calibration functions.

8.3 Initial GC Calibration

8.3.1 GC calibration will be performed by the external calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

8.3.2 Establish the gas chromatographic operating parameters outlined in Section 8.1. Five calibration standard levels are to be prepared for each Aroclor-1221, Aroclor-1242, and Arclor-1254 and the surrogate compounds TCMX and DCB and one calibration standard level (at 50 ng/mL) is to be prepared initially for each Aroclor-1016, Aroclor-1232, Aroclor-1248 and Aroclor-1260 as discussed in section 7.4.2. If Aroclor-1016, Aroclor-1232, Aroclor-1248 or Aroclor-1260 is detected in any project sample based on the single-point calibration, the affected samples must be reanalyzed after a five-point calibration for the detected Aroclor.

8.3.3 Inject each calibration standard using the GC autosampler and the parameters outlined in section 8.1, which are those used for actual samples.

8.3.4 For each Aroclor, 5 peaks are selected to prepare calibration curves (or calibration factor for single-point calibrations). The peaks selected from the multi-component Aroclor formulations were based on maximizing the separation for each Aroclor (*i.e.*, minimizing peak overlap in retention time). Consideration was also given to selecting peaks that normally did not have problems with co-elution with interfering peaks or possible co-elution with

organochlorine pesticides. The determined area of the five peaks selected for calibration is processed by the data workstation as a group, combining the area for calculations of the calibration factors. The following table lists the Aroclors that are included in the initial calibration and group number that represents the group of peaks selected for that Aroclor for calibration purpose.

Aroclor	Group Number
A1221	1
A1232	2
A1016	3

Aroclor	Group Number
A1242	4
A1248	5
A1254	6
A1260	7

8.3.5 Attachment D is an example of chromatograms of standards of each Aroclor for a DB-1 column with peaks selected for calibration labeled.

8.3.6 For the initial calibration curve to be considered valid, the percent relative standard deviation must be less than 20% over the working range. In addition, the correlation coefficient for the linear calibration curve must be greater than or equal to 0.99. The linear-fit calibration curve (not forced

through zero) is used for quantification in every case and is not replaced with the average calibration factor.

8.4 Retention Time Windows

8.4.1 The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Make three injections of each Aroclor at a mid-level concentration throughout a minimum 72-hour time period.

8.4.2 For the 5 peaks selected for calibration of each Aroclor, calculate the standard deviation resulting from the variation in the three retention times for that peak.

8.4.3 The retention time window is defined as plus or minus three times the standard deviation of the three retention time determinations.

8.4.4 If the standard deviation of the selected peak is zero, the standard deviation of the peak eluting after it is used. If it is the last eluting peak that has zero for the standard deviation, then substitute the standard deviation of the peak eluting before the last peak.

8.4.5 Retention time (R.T.) windows established in section 8.4.3 to 8.4.4 may not be practical when samples containing matrix interferences are injected onto the GC column. The default R.T. window of ± 0.08 minutes is employed

when the established windows are below ±0.08 minutes. Besides using retention time windows to assign peaks for quantification, the analyst should rely on their experience in pattern recognition of multi-response chromatographic response exhibited by PCB Aroclors.

8.4.6 Attachment E provides examples of calculated retention time windows generated by the above outlined procedures.

8.5 Gas Chromatographic Analysis

8.5.1 Prior to conducting any analyses on samples, calibrate the system as specified in Section 8.3

8.5.2 To start an analytical sequence, the 500 ppb calibration standard is injected and analyzed for the Aroclor-1221, Aroclor-1242, and Aroclor-1254 after the initial calibration and if more than 24-hours has elapsed since the last valid continuing calibration check standard. If less than 24-hours has elapsed since the last valid continuing calibration check standard, select one 500 ppb continuing calibration check standard (Aroclor-1221, Aroclor-1242, or Aroclor-1254, each containing the surrogate compounds TCMX and DCB). Selection of continuing calibration check standards other than Aroclor-1221, Aroclor-1242, or Aroclor-1254 should be based on anticipated Aroclor contamination that the samples may exhibit. Selection of the continuing calibration check standard after the start of a sequence should also be alternated among the three Aroclors.

8.5.3 The calculated value for each Aroclor and surrogate in the continuing calibration check standard must be ±15% of the true value for it to be valid and analysis to proceed. If this criterion is exceeded, the analyst should inspect the system to determine the cause and perform maintenance as necessary. The system can then be recalibrated and sample analysis can proceed. **Note:** If a failed continuing calibration check returns to acceptable calibration later in the sequence, samples following the acceptable continuing calibration check will be reported; and samples between the failed continuing calibration check and subsequent compliant continuing calibration check will be reanalyzed. All samples which are not bracketed by valid continuing calibration check standards must be reanalyzed when the system is in-control. The analytical sequence must end with the analysis of the CCCs for each Aroclors-1221, -1242, and -1254 (and/or other Aroclors if to be quantitated).

8.5.4 The daily retention time windows must be established. Use the retention time for the selected 5 peaks of the continuing calibration check standard as the midpoint of the window for that day. If all seven Aroclors were analyzed as the initial calibration or continuing calibration check standard, then establish retention time windows using the retention time plus or minus the windows established in Section 8.4. If not all Aroclors were analyzed as the initial calibration or continuing calibration check standard, use the retention time from these Aroclor standard(s) as the midpoint plus or minus the windows established in Section 8.4 to establish the daily retention time windows. For

the remaining Aroclors, go back to the previous time the remaining Aroclors were analyzed as the initial or continuing calibration check standards in the past 24 hours and use those retention times plus or minus the windows established in Section 8.4 to develop daily retention time windows. If greater than 24 hours have elapsed since a particular Aroclor was analyzed as part of the initial or continuing calibration check, the daily retention time window for that Aroclor will be updated by reference to the surrogate or Aroclor continuing calibration check shift(s).

8.5.6 Each Aroclor and surrogate in all succeeding continuing calibration check standards analyzed during an analysis sequence must also have a percent difference of 15% or less when compared to the initial calibration generated from the 5-point calibration curve.

8.5.7 All succeeding standards in an analysis sequence should exhibit retention times that fall within the daily retention time window established by the first continuing calibration check standard(s) of that analytical sequence. If the retention times are outside the established windows instrument maintenance must be performed and recalibration may be required.

8.5.8 The following is the order that initial calibration standards, continuing calibration check standards, method blanks, QC samples, and samples are placed in an analytical sequence. A continuing calibration check standard is run after every ten injections in the analytical sequence. All analytical

sequences must end with a continuing calibration check standard regardless of the number of samples analyzed.

ANALYTICAL SEQUENCE

INJECTION	MATERIAL INJECTED
1	Hexane Blank
2-20	Initial Calibration Standards
21-23	Continuing Calibration Check Standards (Aroclor-1221, Aroclor-1242, and Aroclor-1254 and other Aroclors if reanalysis occurs if other Aroclors were observed in the samples)
24-33	Sample analyses, including method blanks, matrix spikes, matrix spike duplicates, and QC reference check standard (LCS). A maximum of 10 samples between continuing calibration check standards.
34	Continuing calibration check standard

ANALYTICAL SEQUENCE (CONTINUED)

INJECTION	MATERIAL INJECTED
45 and higher	Repeat inject. 24-34 sequence (Alternating continuing calibration check standards between Aroclor-1221, Aroclor-1242, and Aroclor-1254 and other Aroclors [reanalysis occurs if other Aroclors were observed in the samples])
Closing injections:	Continuing calibration check standards (Aroclor-1221, Aroclor-1242, and Aroclor-1254 and other Aroclors [reanalysis occurs if other Aroclors were observed in the samples])

8.6 Quality Control (Refer to Attachment F for a summary of the quality control requirements.)

8.6.1 This section outlines the necessary quality control samples that need to be instituted at the time of sample extraction. The data from these quality control samples is maintained to document the quality of the data generated.

8.6.2 Each analyst must demonstrate competence in accuracy and precision on quality control samples before beginning analysis on samples. This demonstration must be on-going and be repeated if there is any modification to the method.

8.6.3 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For sediment/solid samples, a laboratory sodium sulfate blank is processed.

8.6.4 The method blank must exhibit PCB levels less than the matrix-defined reporting limit. If the method blank exhibits PCB contamination above the reporting limit, the samples associated with the contaminated blank should be re-extracted and analysis repeated. If there is no original sample available for re-extraction, then the results should be flagged with a "B" indicating blank contamination. The value measured in the blank is reported for those samples associated with the particular blank out of criteria.

8.6.5 At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, a matrix spike for Aroclor-1242 is to be analyzed at a rate of 1 matrix spike per every 20 samples at a concentration of 20,000 ng/mL in the extract (**Note:** this spike concentration will require a sample dilution to be performed). Also a matrix spike duplicate sample is to be analyzed at a rate of 1 per every 20 samples.

8.6.6 If requested, analyze one unspiked and two spiked samples. Calculate the percent recovery based on Aroclor concentration of both samples as follows:

A = concentration of spiked sample
B = concentration of unspiked sample (background)
T = known true value of the spike

Percent Recovery (p) = 100 (A-B) %/T

Compare the percent recovery calculated with the project limits of 60-140%. If the concentrations of the matrix spikes are *greater* than four times the calculated sample amount then the quality control limits should be applied. If the concentrations of the matrix spikes are *less* than four times the sample than there are no established limits applicable. If the percent recovery falls outside the acceptance range for the given Aroclor used as the spiking analyte, then the matrix spike recovery failed the acceptance criteria. Check for documentable errors (*e.g.,* calculations and spike preparations) and then check the unspiked sample results and surrogate recoveries for indications of matrix effects. If no errors are found and the associated QC reference check standard (Laboratory Control Sample [LCS]) is within 60-140%, then sample matrix effects are the most likely cause. Note this in the Case Narrative.

A relative percent difference (RPD) must also be calculated on the matrix spike set recoveries. This is calculated as follows:

A = % recovery of matrix spike sample
B = % recovery of matrix spike duplicate sample

RPD = [(A-B)/{(A+B)/2}] x 100
where (A-B) *is taken as an absolute value*

If the concentrations of the matrix spike set are *greater* than four times the calculated sample amount, then an RPD of 40% or less is acceptable. If the concentrations of the matrix spike set are *less* than four times the calculated sample amount than there are no established limits applicable to the RPD. If the criterion is not met, check for documentable errors (*e.g.,* calculations and spike preparations) and then check the unspiked sample results and surrogate recoveries for indications of matrix effects. If no errors are found and the associated LCS is within 60-140%, then sample matrix effects are the most likely cause. Note this in the Case Narrative.

8.6.7 A QC reference check standard (LCS) is also prepared and analyzed for Aroclor-1242 at a concentration of 500 ng/mL in the extract. For sediment/solid samples, sodium sulfate is used for the QC reference check standard (LCS). Calculate the percent recovery for the Aroclor spike and compare to the project limits of 60-140%. If the percent recovery for the QC reference check standard (LCS) is out of criteria, the analysis is out of the control for that analyte and the problem should be immediately corrected. The entire batch of samples will need to be re-extracted and re-run (Exception: If the LCS recovery is high and there were no associated positive results for any Aroclor, then address the issue in the Case Narrative and no further action is needed).

8.6.8 Surrogate compounds are added to each sample, matrix spike, matrix spike duplicate, method blank, and QC reference check standard (LCS) at time of extraction. The surrogate compounds TCMX and DCB are to be added prior to extraction for final extract concentrations of 10 ng/mL and 100 ng/mL, respectively (refer to extraction SOPs).

8.6.9 Only one surrogate analyte needs to meet established control limits for the analysis to be valid. For samples analyzed at a five-fold dilution of the extract or less, the data is compared to the project limits of 60-140%. If percent surrogate recovery is not within limits for either surrogate, the following steps are required.

8.6.9.1 Review calculations that were used to generate surrogate percent recovery values to make certain there are no errors.

8.6.9.2 Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

8.6.9.3 Re-analyze the extracts that did not meet control limits at the previously analyzed dilution to assess if the sample matrix interfered with surrogate measurement.

8.6.9.4 If the above steps do not give satisfactory results, re-extract and re-analyze the sample. Report this data if surrogate recovery is within limits. If surrogate percent recovery is out of limits for the re-extracted samples, low or high surrogate recovery is due to matrix affects and the data can be reported as estimated and the data user is notified in the form of a case narrative.

8.7 Qualitative/Quantitative Issues

8.7.1 Quantitation of Aroclors is complex. In each case, the Aroclor is made up of numerous compounds and, consequently, the chromatograms are multi-peak; also, in each case, the chromatogram of the residue may not match that of the standard. These residues are quantitated by comparison to one or more of the Aroclor mixtures, depending on the chromatographic pattern of the

residue. A choice must be made as to which Aroclor or mixture of Aroclors will produce a chromatogram most similar to that of the residue.

8.7.2 If Aroclors-1016, -1232, -1248, and/or -1260 are detected in a project sample, the instrument must be calibrated using 5 concentration levels (20 ng/mL, 100 ng/mL, 250 ng/mL, 500 ng/mL, and 1000 ng/mL) for the detected Aroclor(s) and the sample reanalyzed for quantitation by a 5-point linear fit calibration curve. The same acceptance criteria that applied to initial calibration and continuing calibration check standard analysis for Aroclors -1221, -1242, and -1254 will apply to Aroclors-1016, -1232, -1248, and/or -1260 when samples are reanalyzed for quantitation of any of these Aroclors.

8.7.3 All quantitations are to be based on 5-point initial calibrations (using external standard calibration techniques). The concentration of each Aroclor and surrogate in the sample will be determined by using the linear-fit calibration curve (see section 8.7.5) determined from the initial calibration standards. Refer to section 8.3 for initial calibration procedures. The final calculated sample concentration will take into account the sample-specific dilution factor, initial sample weight, final extract volume, and percent solids. All solids will be reported on a dry-weight basis.

8.7.4 If the instrument level of any Aroclor in a sample exceeds the instrument level of that Aroclor in the highest level standard, the sample must be diluted to approximately mid-level of the calibration range and reanalyzed for quantitation.

8.7.5 Calibration Curve by First Order Linear Regression External Standard Calibration.

Five selected Aroclor quantitation peaks are calibrated by first order linear regression with intercept. The surrogates TCMX and DCB are calibrated and quantified in the same manner using the individual peak areas for these analytes:

Equation of Line: $Y = aX + b$

where:

Y = summed total peak area of quantitation peaks used (uV-sec)
a = coefficient constant (slope)
X = calibration concentration (ng/mL)
b = first order coefficient (intercept)

8.7.6 Sample Concentration result calculation (solid samples)

$$C = \frac{(Y_i - b) * V_e * df}{a * M * \%TS * 1000}$$

where:

C = sample concentration (μg/g)
Y_i = summed total area of quantitation peaks in sample. (uV-sec)
b = intercept from (#1 above) (uV-sec)
V_e = concentrated extract volume (mL)
df = analytical dilution factor of extract
a = slope (from #1 above)
M = mass of sample in (g)
%TS = Percent Total Solid (in decimal format)
1000 = units conversion ng to μg

9.0 References

9.1 U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update, December 1996.

9.2 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants", July 1988.

9.3 New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual", Wadsworth Center for laboratories and Research, 1988, updated 1998.

9.4 "Guide to Environmental Analytical Methods", fourth edition, Genium Publishing Corporation, 1998.

10.0 Attachments (**Note:** Attachments are not paginated.)

10.1 Attachment A: Example PCB Standards Preparation Tables

10.2 Attachment B: PCB Continuing Calibration Tables

10.3 Attachment C: Gas Chromatograph Operating Procedures

10.4 Attachment D: Chromatograms of PCB standards.

10.5 Attachment E: Retention Time Windows

10.6 Attachment F: Quality Control Requirements Summary Table for SOP GEH8082

ATTACHMENT A

Table 1
Example PCB Stock Standard Preparation Table

PCB Formulation	Supplier	Catalog #	Std. weight (mg)	Conc.(PPM)
A1016	Neat Material Source	NA	93.2	932.0
A1221	Neat Material Source	NA	106.8	1068.0
A1232	Neat Material Source	NA	103.3	1033.0
A1242	Neat Material Source	NA	99.0	990.0
A1248	Neat Material Source	NA	101.9	1019.0
A1254	Neat Material Source	NA	99.6	996.0
A1260	Neat Material Source	NA	99.2	992.0
DCB	Chem Service	F2170	10	100.0

Unless otherwise noted hexane is the solution used to make all dilutions.

Table 2
Example PCB Working Standard Preparation Table

PCB Stock Standards	Init. Volume(mL)	Final Volume(mL)	Conc.(PPM)
A1016	1.0	100	9.32
A1221	1.0	100	10.68
A1232	1.0	100	10.33
A1242	1.0	100	9.90
A1248	1.0	100	10.19
A1254	1.0	100	9.96
A1260	1.0	100	9.92

ATTACHMENT A cont'd

Table 3
Example PCB Calibration Standard Preparation Table

Init. Volume (mL)	Final Volume (mL)	Final Concentration (PPM)					
		A1016	A1221	A1232	A1242	A1248	A1260
5.0	50	0.932	1.068	1.033	0.990	1.019	0.992
2.5	50	0.466	0.534	0.5165	0.495	0.5095	0.496
1.25	50	0.233	0.267	0.25825	0.2475	0.2548	0.248
1.00	50	0.1864	0.2136	0.2066	0.198	0.2038	0.1984
0.500	50	0.0932	0.1068	0.1033	0.0990	0.1019	0.0992
5.0*	50	0.01864	0.02136	0.02066	0.0198	0.02038	0.01984

*This initial volume is of the nominal 0.250 ppm standard. All others are from the nominal 10 ppm standard.

Table 4
Example PCB Aroclor-1254 Calibration Standard Preparation Table

Init. Volume (mL) A1254	Init. Volume (mL) Surrogate	Final Volume (mL)	Final Concentration(PPM)		
			A1254	TCMX	DCB
5.0	0	50	0.996	0	0
10.0	4.0	100	0.996	0.020	0.200
25.0*	0	50	0.498	0.010	0.100
1.25	0.800	50	0.249	0.008	0.080
0.500	0.500	50	0.0996	0.005	0.050
0.100**	0.200	50	0.01992	0.002	0.020

*This initial volume is of the A1254 0.996ppm solution with surrogates.
**This initial volume is of the A1254 0.996ppm solution without surrogates.
All others are from the A1254 9.96ppm working standard.

ATTACHMENT B

Table 1
PCB Continuing Calibration Stock Standards

PCB	Supplier*	Catalog #*	Conc. (μg/mL)
A1016	Chem Service	F107AS	1000
A1221	Chem Service	F108AS	1000
A1232	Chem Service	F113AS	1000
A1242	Chem Service	F109AS	1000
A1248	Chem Service	F110AS	1000
A1254	Chem Service	F111AS	1000
A1260	Chem Service	F112BS	1000

*Or Equivalent.

Table 2
PCB Continuing Calibration Working Standards

PCB	Initial Volume(mL)	Final Volume(mL)	Concentration(PPM)
A1016	1.0	100	10
A1221	1.0	100	10
A1232	1.0	100	10
A1242	1.0	100	10
A1248	1.0	100	10
A1254	1.0	100	10
A1260	1.0	100	10

ATTACHMENT B cont'd

Table 3
PCB Continuing Calibration Standards

PCB	Initial Volume(mL)	Final Volume(mL)	Concentration (PPM)
A1016	2.5	50	0.500
A1221	2.5	50	0.500
A1232	2.5	50	0.500
A1242	2.5	50	0.500
A1248	2.5	50	0.500
A1254	2.5	50	0.500
A1260	2.5	50	0.500

ATTACHMENT C

Gas Chromatograph Operating Procedures[1]

Column Type	Capillary
Column ID	DB-1
Vendor	J&W (or equivalent)
Part Number	122-1032
Column Length(m)	30
ID(mm)	0.25
Film Thick.(um)	0.25
1)Initial Col. Temp. (^{o}C)	140
1)Col. Hold Time (min.)	1.0
1)Col. Temp. Rate (^{o}C/min.)	10
1)Final Col. Temp. (^{o}C)	200
1)Col. Hold Time (min.)	NA
2)Col. Temp. Rate (^{o}C/min.)	5
2)Final Col. Temp. (^{o}C)	245
2)Col. Hold Time (min.)	14.50
GC Col. gas flow rate (mL/min.)	17-24
ECD autozero	Yes
Detector Temp.(^{o}C)	300
Init. Injector Temp. (^{o}C)	300
A/S Vial Needle Depth	85
A/S Solvent Select	3
A/S Upper Air Gap	Yes
A/S Lower Air Gap	Yes
A/S Viscosity Factor	1

ATTACHMENT C cont'd

Gas Chromatograph Operating Procedures[1]

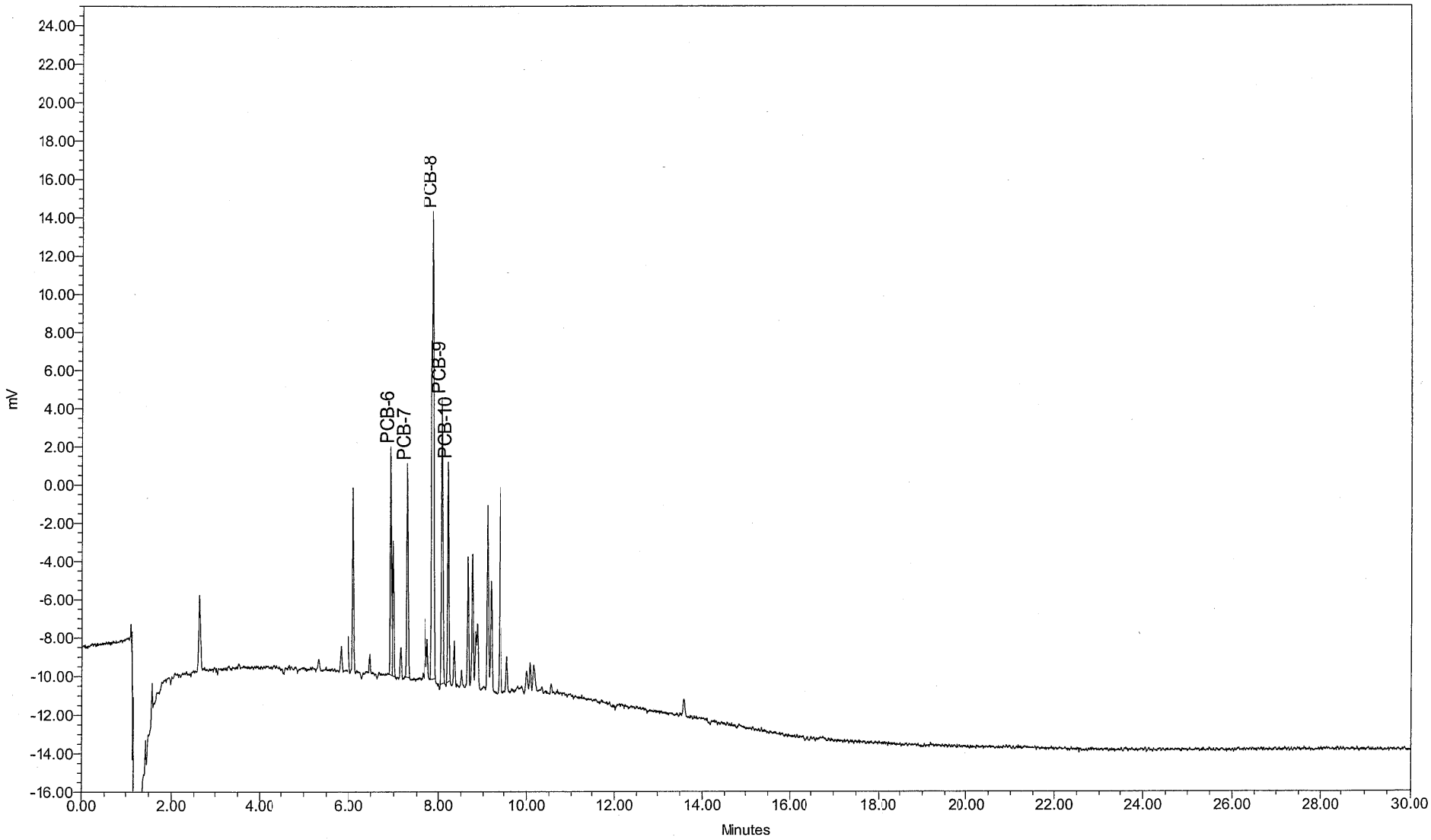
A/S Hot Needle Time (min.)	0.05
Autosampler (A/S) Model Number	8100 (or equivalent)
A/S Injection Volume (uL)	Lab-determined
A/S Injection Time (min.)	0.01
A/S Injection Rate (uL/sec.)	Fast 4.0
A/S Solvent Inj. plug size (uL)	0.2

Note:
1 – Parameters can be adjusted as necessary for the specific instrument used by the laboratory provided that chromatography for quantitation peaks is consistent with the examples in this SOP.

ATTACHMENT D
DB-1 CHROMATOGRAMS

Chromatogram Report, PCB by SW846 Method 8082

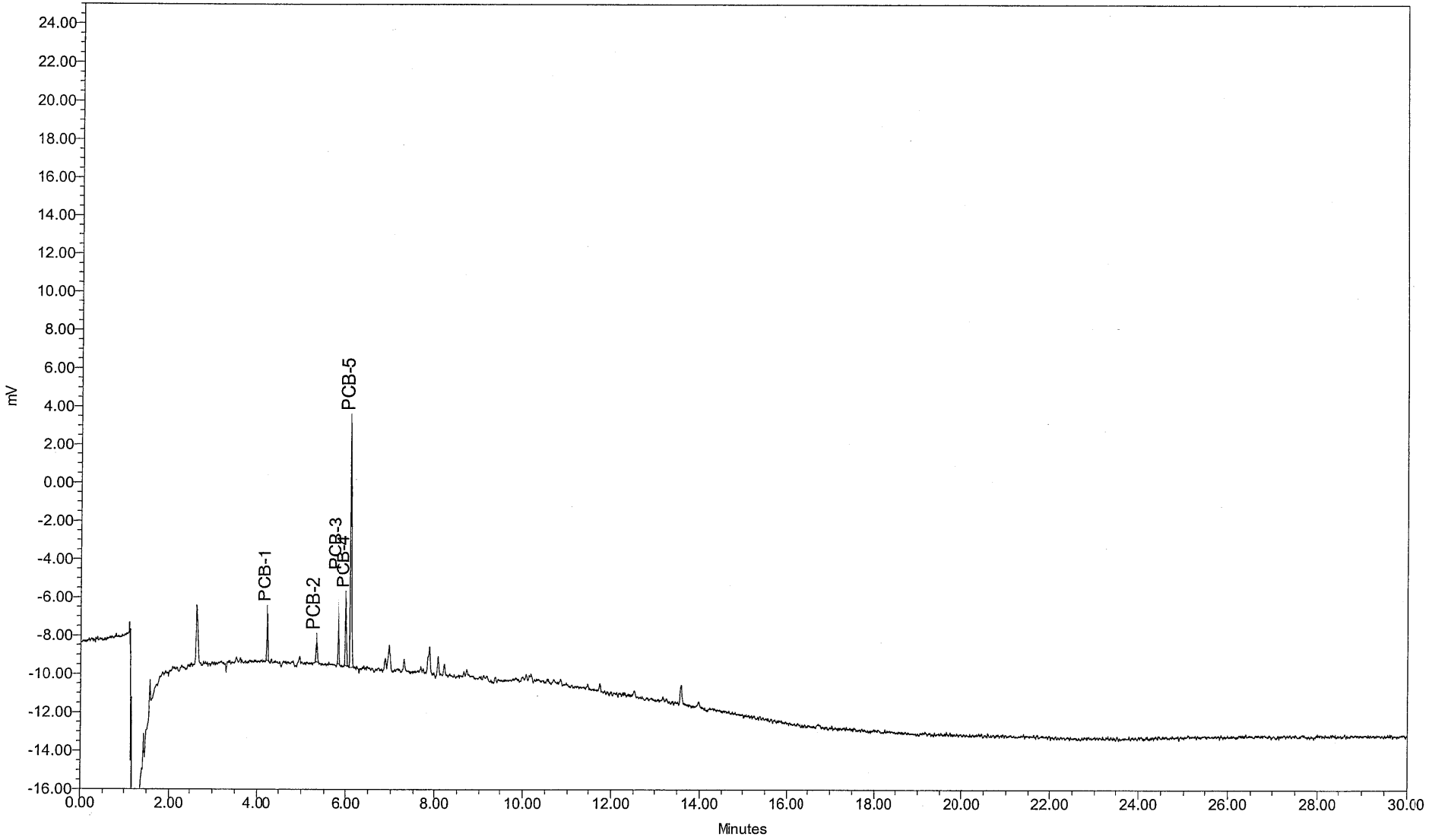
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS160725	Sample Amount:	1
Sample ID:	A1016 500 PPB	Dilution:	1
Date Acquired:	07/26/1999 09:32:16	Processing Method:	GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082

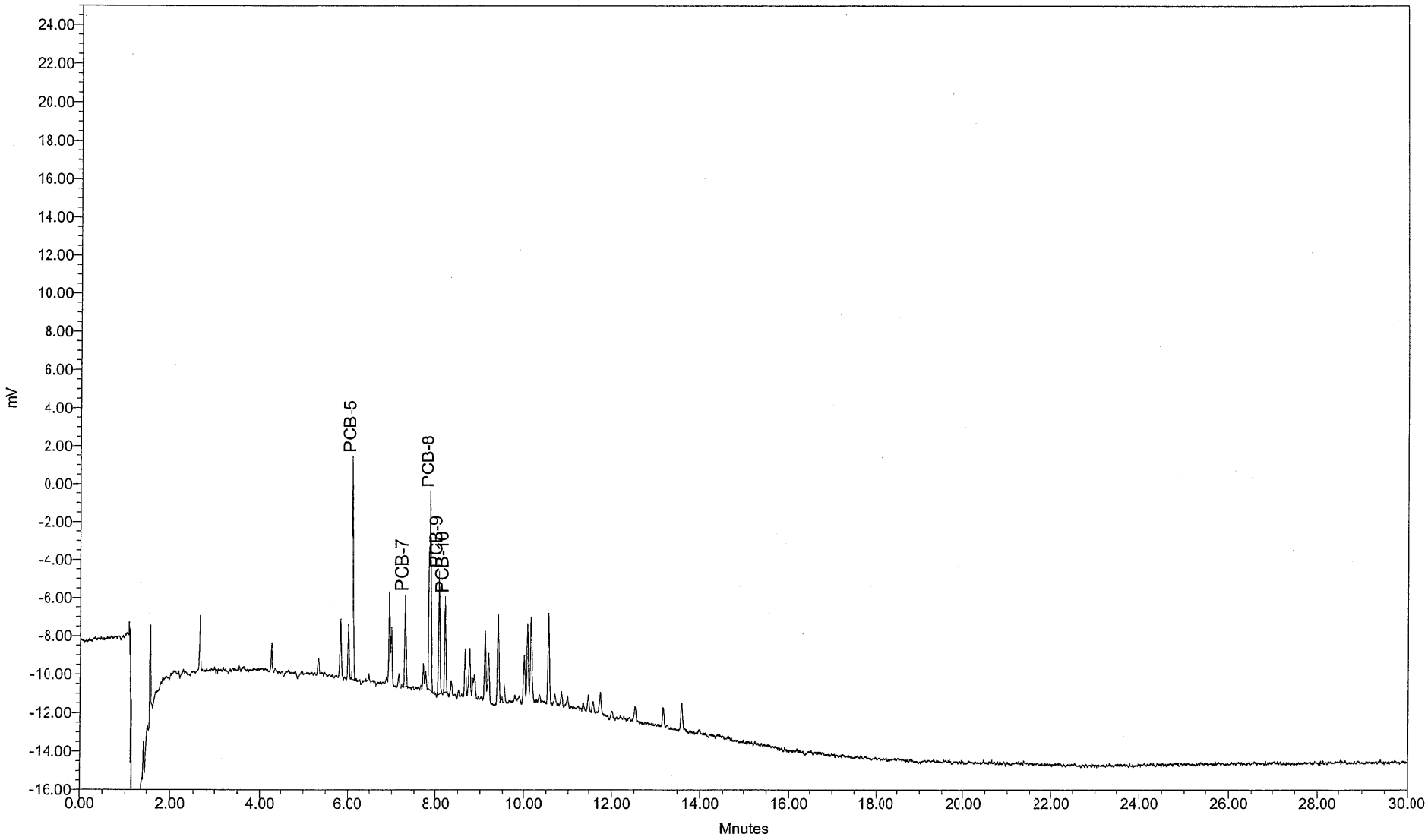
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS210725	**Sample Amount:**	1
Sample ID:	A1221 500 PPB	**Dilution:**	1
Date Acquired:	07/26/1999 10:08:26	**Processing Method:**	GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082

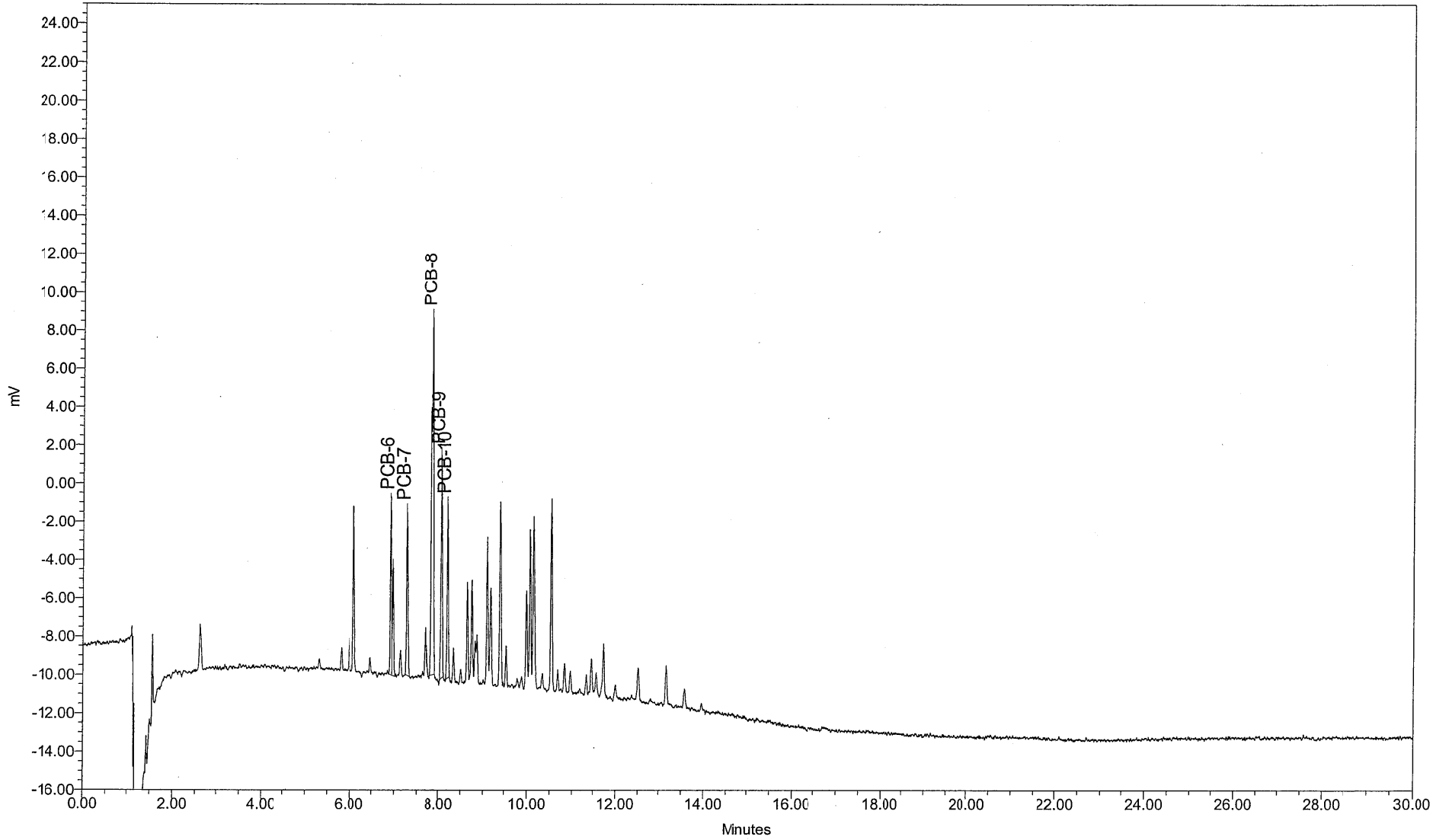
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS320725	**Sample Amount:**	1
Sample ID:	A1232 500 PPB	**Dilution:**	1
Date Acquired:	07/26/1999 11:09:59	**Processing Method:**	GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082

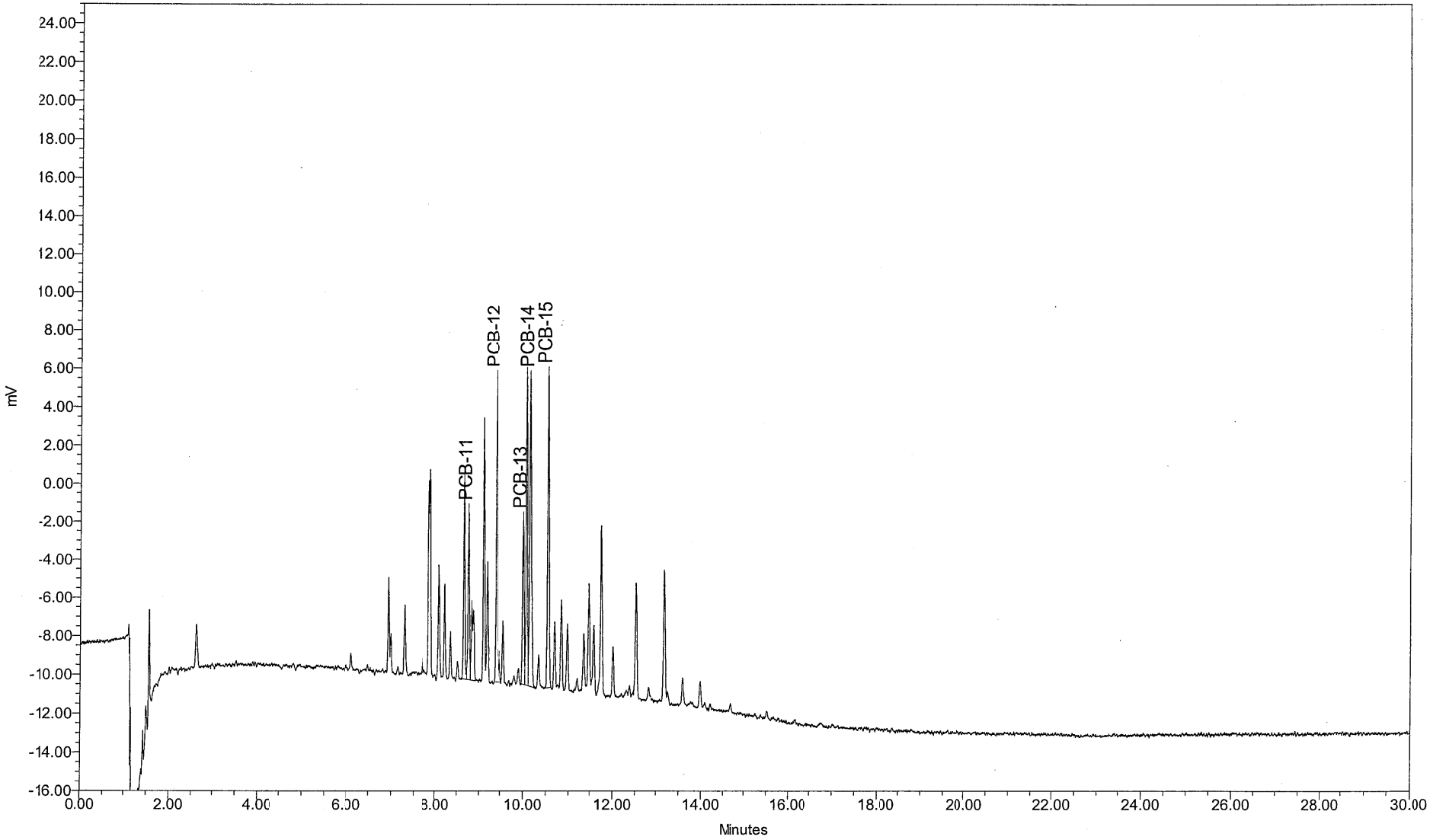
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS420725	Sample Amount:	1
Sample ID:	A1242 500 PPB	Dilution:	1
Date Acquired:	07/26/1999 11:46:07	Processing Method:	GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082

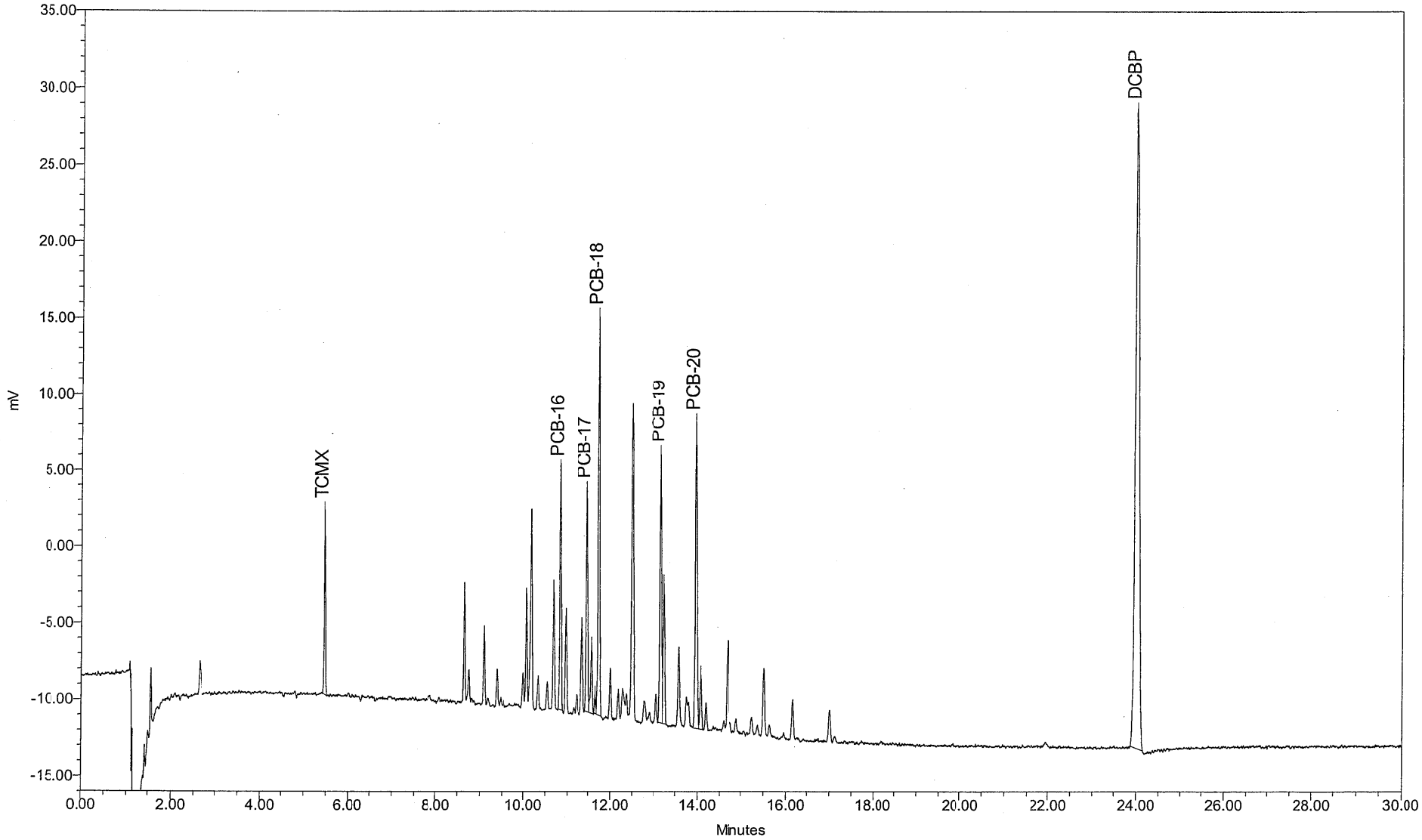
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS480725	**Sample Amount:**	1
Sample ID:	A1248 500 PPB	**Dilution:**	1
Date Acquired:	07/26/1999 12:22:14	**Processing Method:**	GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082

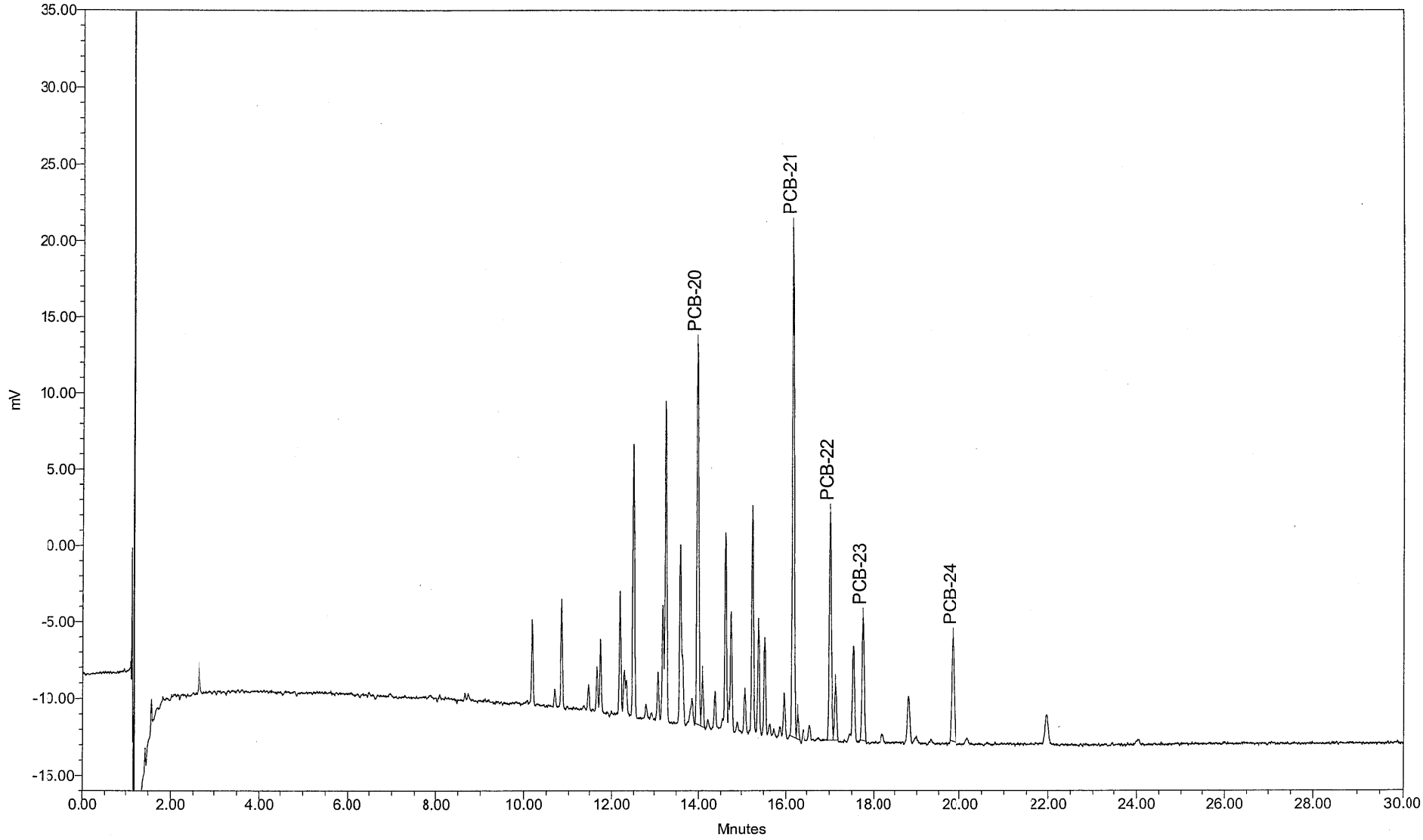
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS540725	Sample Amount:	1
Sample ID:	A1254 500 PPB	Dilution:	1
Date Acquired:	07/26/1999 12:58:21	Processing Method:	GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS500725	**Sample Amount:**	1
Sample ID:	A1260 500 PPB	**Dilution:**	1
Date Acquired:	07/26/1999 13:34:27	**Processing Method:**	GC7_8082_060899

ATTACHMENT E
RETENTION TIME WINDOWS

Northeast Analytical Inc 11/01/99

Retention Time Window Study
for Aroclors (PCB) by GC/ECD
EPA Method 8082

Instrument: GC 7
Column: DB-1

Analyte	PEAK	Standard 1 500 PPB R.T. Min	Standard 2 500 PPB R.T. Min	Standard 3 500 PPB R.T. Min	STD. DEV Min	%RSD	Window +/- Min.
		CS__0919	CS__1003	CS__1011			
Aroclor 1016	6	6.902	6.922	6.876	0.0231	0.33	0.069
	7	7.260	7.228	7.232	0.0174	0.24	0.052
	8	7.852	7.818	7.823	0.0184	0.23	0.055
	9	8.051	8.018	8.022	0.0180	0.22	0.054
	10	8.185	8.151	8.155	0.0186	0.23	0.056
Aroclor 1221	1	4.212	4.199	4.190	0.0111	0.26	0.033
	2	5.294	5.277	5.269	0.0128	0.24	0.038
	3	5.787	5.775	5.765	0.0110	0.19	0.033
	4	5.962	5.951	5.941	0.0105	0.18	0.032
	5	6.072	6.062	6.051	0.0105	0.17	0.032
Aroclor 1232	5	6.080	6.050	6.059	0.0154	0.25	0.046
	7	7.258	7.227	7.237	0.0158	0.22	0.047
	8	7.852	7.819	7.829	0.0169	0.22	0.051
	9	8.050	8.018	8.028	0.0164	0.20	0.049
	10	8.184	8.152	8.163	0.0163	0.20	0.049
Aroclor 1242	6	6.894	6.927	6.872	0.0277	0.40	0.083
	7	7.251	7.234	7.228	0.0119	0.16	0.036
	8	7.844	7.826	7.820	0.0125	0.16	0.037
	9	8.043	8.025	8.020	0.0121	0.15	0.036
	10	8.178	8.159	8.155	0.0123	0.15	0.037
Aroclor 1248	11	8.724	8.689	8.700	0.0179	0.21	0.054
	12	9.352	9.313	9.324	0.0201	0.22	0.060
	13	9.965	9.927	9.938	0.0196	0.20	0.059
	14	10.122	10.082	10.094	0.0205	0.20	0.062
	15	10.511	10.470	10.480	0.0214	0.20	0.064
Arolcor 1254	16	10.795	10.773	10.767	0.0147	0.14	0.044
	17	11.431	11.409	11.403	0.0147	0.13	0.044
	18	11.703	11.680	11.673	0.0157	0.13	0.047
	19	13.139	13.113	13.108	0.0166	0.13	0.050
	20	13.931	13.907	13.902	0.0155	0.11	0.047
Arolcor 1260	20	13.942	13.896	13.911	0.0235	0.17	0.070
	21	16.125	16.081	16.093	0.0227	0.14	0.068
	22	16.985	17.049	16.943	0.0534	0.31	0.160
	23	17.717	17.665	17.675	0.0276	0.16	0.083
	24	19.799	19.732	19.750	0.0347	0.18	0.104
TCMX (SURROGATE)	Surr.	5.445	5.429	5.425	0.0106	0.19	0.032
DCB (SURROGATE)	Surr.	23.984	23.91	23.91	0.0439	0.18	0.132

ATTACHMENT F
QUALITY CONTROL REQUIRMENTS
SUMMARY TABLE

Summary Table GEHR8082

Polychlorinated Biphenyls - SW-846 Method 8082
Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Sulfuric Acid Cleanup, Sulfur Cleanup, Florisil Cleanup	All samples for PCB only.	Not applicable.	Not applicable.
Initial Calibration	• Established initially and when CCC fails criteria. • At 5 concentration levels for Aroclors -1221, -1242, and -1254 and surrogate compounds (TCMX and DCBP). The 5 concentration levels are to be 20 ng/mL, 100 ng/mL, 250 ng/mL, 500 ng/mL, and 1000 ng/mL for each Aroclor. The surrogate compounds are to be combined with the Aroclor 1254 standards at concentrations of 2 ng/mL, 5 ng/mL, 8 ng/mL, 10 ng/mL and 20 ng/mL (TCMX) and 20 ng/mL, 50 ng/mL, 80 ng/mL, 100 ng/mL, and 200 ng/mL (DCB). • One standard calibration for each of the remaining Aroclor mixtures (1016, 1232, 1248, and 1260), at the reporting limit. If any one of these Aroclors is detected in a sample, the sample must be reanalyzed under a 5-point calibration for the detected Aroclor(s) for quantitation.	• %RSD≤20% among calibration factors (CFs) AND correlation coefficient ≥ 0.99 for each Aroclor mixture and surrogate (to be quantitated using linear-fit calibration curve not forced through zero). • Calibration factors are to be calculated using the total area for 5 peaks for each Aroclor. (Refer to SOP GEHR8082 Section 8.3.4 for selection of peaks.) • Each Aroclor must display distinctive pattern.	• Reanalyze the initial calibration curve and/or evaluate/correct instrument malfunction to obtain initial calibration which meets criteria. • Sample results above highest standard concentration require dilution and reanalysis. • If Aroclors-1016, -1232, -1248, and/or -1260 is detected in a project sample, the instrument must be calibrated using 5 concentration levels (20 ng/mL, 100 ng/mL, 250 ng/mL, 500 ng/mL, and 1000 ng/mL) for the detected Aroclor(s) and the sample reanalyzed. Same acceptance criteria that applied to 5-point calibration for Aroclors -1221, -1242, and -1254 will apply to these Aroclors.

Summary Table GEHR8082

Polychlorinated Biphenyls - SW-846 Method 8082
Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Check (CCC) Standard	• CCC for each Aroclors -1221, -1242, and -1254 at the beginning of the daily sequence (when >24-hour break in continuous analysis), unless initial calibration is performed. • If <24 hours break, CCCs will be alternated among Aroclors -1221, -1242, and -1254 after each analysis of 10 samples. • All CCCs must contain TCMX and DCB. • If analytical run is being performed for quantitation of Aroclors-1016, -1232, -1248, and/or -1260 (refer to initial calibration), CCCs for Aroclor(s) to be quantitated must be analyzed at the same frequency required for Aroclors -1221, -1242, and -1254. • Analytical sequence must end with analysis of CCCs for each Aroclors-1221, -1242, and -1254 (and/or other Aroclors if to be quantitated).	• ≤15% Drift based on "true" concentration for each Aroclor and surrogate when quantitated as a sample. • RT of each peak used for identification of the Aroclor must be within RT window (reset daily at the beginning of the sequence for the 24-hour day). • All samples must be bracketed by CCCs for Aroclors -1221, -1242, and -1254 (and/or other Aroclors if to be quantitated) that meet all criteria stated above.	• Correct system, if necessary, and recalibrate. Criteria must be met before sample analysis may begin. Samples that are not bracketed by compliant CCCs must be reanalyzed. • If a failed CCC returns to acceptable calibration later in the sequence, samples following the acceptable CCC will be reported; and samples between the failed CCC and subsequent compliant CCC will be reanalyzed.
Retention Time (RT) Windows	1. Established at ± 3 × std. dev. of RT of three standard analyses over 72-hour period. Must establish whenever a new column is installed. (Default RT window is ±0.08 minutes - Refer to SOP GEHR8082 Section 8.4 for additional guidance.) 2. RT windows are recentered daily based on RT of each of the peaks used for Aroclor identification in the first CCC of the day. (Refer to SOP GEHR8082 Section 8.5.4 for guidance on setting daily RT windows for Aroclors not analyzed as part of initial CCC.)	• RT of CCC peaks must be within established windows in the CCCs analyzed throughout day. • Recentering windows is permitted only once per 24 hours.	Adjust system, reestablish RT windows, and recalibrate.
Retention Time (RT) Shift	Each CCC analysis: RT of the peaks chosen for the identification of the Aroclors in the CCC are evaluated against the first CCC of the day.	Each quantitation peak for each Aroclor and each surrogate peak should be within window established.	Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate.

Summary Table GEHR8082

Polychlorinated Biphenyls - SW-846 Method 8082
Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Method Blank	• One per extraction batch of ≤20 samples of the same matrix per day. • Must be analyzed on each instrument used to analyze associated samples. • Must undergo all sample preparative procedures.	• Concentration does not exceed the reporting limit of any Aroclor. Not applicable if positive results were not reported for any associated samples. • Must meet surrogate criteria.	1. Reanalyze blank to determine if instrument contamination was the cause. If the method blank is still non-compliant, then follow 2 below. 2. Reextract and reanalyze all associated samples.
QC Reference Standard - Laboratory Control Sample (LCS)	One per extraction batch of ≤20 samples per matrix per day. The LCS must be from a second source and contain Arcolor 1242 at a concentration of 500 ng/mL at the instrument.	• % Recovery of Aroclor 1242 within project limits of 60-140%. • Must meet surrogate criteria.	Reanalyze LCS. If still out, reextract and reanalyze all associated samples. (Exception: If LCS recovery is high and no associated positives, then address in Case Narrative and no further action needed.)
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, one MS/MSD pair per extraction batch of ≤20 samples per matrix per day. The MS/MSD samples must be spiked with Arcolor 1242 at a concentration of 20,000 ng/mL at the instrument (note: this will require dilution).	• Aroclor 1242 % Recoveries within 60-140% (when MS/MSD spike concentration is greater than 4× the unspiked sample amount). • RPD within 40% (when MS/MSD spike concentration is greater than 4× the unspiked sample amount). • Must meet surrogate criteria (unless also outside of criteria in unspiked sample).	1. If recoveries for the spiked Aroclor are not within 60-140% or the RPD is >40%, check for documentable errors (*e.g.*, calculations and spike preparation). 2. Check unspiked sample results and surrogate recoveries for indications of matrix effects. 3. If no errors are found, and the associated LCS is within 60-140%, then sample matrix effects are the most likely cause. Note in Case Narrative.

Summary Table GEHR8082

Polychlorinated Biphenyls - SW-846 Method 8082
Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Surrogates	• TCMX and DCB are added to all standards, blanks, samples, and QC samples at a concentration of 10 ng/mL TCMX and 100 ng/mL DCB at instrument level. • Calibrated as a target compound in the Aroclor 1254 initial calibration standards.	% Recovery of at least one surrogate within 60-140% for samples analyzed at an extract dilution factor of 5 or less.	If both recoveries are not within limits: 1. Check to be sure that there are no errors in calculations and surrogate solutions. Also, check instrument performance. 2. If no problem is found, reextract and reanalyze the sample. 3. If the reanalysis is within limits and holding time, then report only the reanalysis. 4. If the reanalysis is within limits, but out of holding time, then report both sets of data. 5. If the reanalysis is still out of limits, then report both sets of data. 6. No reanalysis is required if the sample was chosen for the MS/MSD analysis and the MS and/or MSD are also outside limits.
Qualitative/Quantitative Issues	If Aroclors-1016, -1232, -1248, and/or -1260 are detected in a project sample analyzed under a single-point calibration for the detected Aroclor, the sample must be reanalyzed under a 5-point calibration for the detected Aroclor(s).	All positive results for Aroclors must be quantitated using a 5-point linear-fit calibration curve and must be bracketed by compliant CCCs containing the detected Aroclor.	If Aroclors-1016, -1232, -1248, and/or -1260 are detected in a project sample, the instrument must be calibrated using 5 concentration levels for the detected Aroclor(s) and the sample reanalyzed. Same acceptance criteria that applied to initial calibration and CCC analysis for Aroclors -1221, -1242, and -1254 will apply to these Aroclors.
	If instrument level of any Aroclor in a sample exceeds the instrument level of that Aroclor in the highest level standard, the sample must be diluted to approximately mid-level of the calibration range and reanalyzed.	The instrument level of all Aroclors must be within the calibration range for all samples.	Dilute the sample to bring the level of the highest concentration of Aroclors within the calibration range.