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STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: Analytical Determination of Aroclors (PCBs) Using EPA Method TO-10

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RECORD OF MAJOR REVISIONS:

NEW	1	2	3	4	5	6
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STANDARD OPERATING PROCEDURE

ANALYTICAL DETERMINATION OF PCBS USING EPA METHOD TO-10A

1.0 SCOPE AND APPLICATION

1.1 TO-10A is used to determine the concentrations of polychlorinated biphenyls (PCBs) in ambient air.

1.2 TO-10A samples are collected on low-volume polyurethane foam samplers (PUFs).

1.3 Soxhlet extraction techniques are utilized to extract the analyte from the sampler and quantitation is achieved using standard Gas Chromatography with electron capture detectors (ECD) detection.

1.4 Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation (weathered) or degradation by treatment technologies. These conditions result in "weathered" Aroclors that may have significant differences in peak patterns than those of Aroclor standards.

1.5 The results from a single column analysis require confirmation on a second GC column of dissimilar stationary phase (as in dual column analysis), based on a clearly identifiable Aroclor pattern, or using another technique such as GC/MS.

1.5.1 Second column confirmation will be utilized when interferences are present that obscure the accurate Aroclor identification/quantification.

1.6 The analyst must select columns, detectors, and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix-specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extracts, diluted oil samples, etc.)

1.7 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Air is sampled through a PUF sample cartridge for 24 hours then returned to the laboratory for analysis. Airborne particles may also be collected on a pre-filter, extracted in conjunction with the PUF material, and analyzed accordance with this procedure.

2.2 Extraction of the PUF and particulate filters, if applicable, is accomplished using 5 percent diethyl ether in hexane and traditional Soxhlet extractors.

2.2.1 The Soxhlet extraction cycle should fill and siphon at a rate of 4 to 6 cycles per hour over the course of a 16-hour period.

2.2.2 Extracts are then concentrated to 1 ml final volume using a Kuderna-Danish (K-D) apparatus.

2.3 The extract is analyzed using a gas chromatograph with a wide-bore fused silica capillary columns and dual electron capture detector (GC/ECD).

2.4 The chromatographic data may be used to determine the seven Aroclors listed in section 5.1, or a single value for total PCBs present.

3.0 SAFETY PRECAUTIONS

3.1 It is imperative that all laboratory personnel treat every sample as implicitly hazardous.

3.1.1 The toxicity or carcinogencity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

3.1.2 PCBs have been classified as a known or suspected, human or mammalian carcinogen. Many of the other common pesticides have been classified as carcinogens. Care must be exercised when working with these substances. This method does not purport to address all safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. The user should be thoroughly familiar with the chemical and physical properties of targeted substances.

4.0 SAMPLE COLLECTION AND HANDLING

4.1 PUF sampling cartridges and quartz pre-filters are currently purchased pre-cleaned from SKC, Inc., 863 Valley View Road, Eighty Four, PA 15330, (Phone: 800-752-8472). PUFs may also be purchased for additional providers if satisfactory cleaning and supply can be demonstrated.

4.2 Samples should be taken in accordance with the sampling procedure found in the EPA method TO-10A; however, ALS does not participate in sampling activities.

4.2.1 Samples should be returned to the laboratory enclosed in aluminum foil with the particulate filter included in the PUF sampling cartridge.

4.2.2 If the time span between sample collection and laboratory analysis exceeds 24 hours, place the samples in a cooler with sufficient ice, "blue ice," or dry ice to maintain the sample temperatures at 4°C.

4.2.2.1 If regular ice is used samples should be sufficiently protected for moisture from possible melting during shipment.

4.2.3 At least 1 filter/PUF blank should be returned to the laboratory with each group of field samples.

4.3 Upon receipt laboratory personnel will ensure that PUF samples are placed in environmentally controlled storage prior to sample extraction.

4.4 Extraction must be performed within 7 days of sampling and analysis within 40 days of extraction.

5.0 DETECTION LIMITS

5.1 The Estimated Quantitation Limits (EQL) for the analytes in PUF samples are listed below:

Compound	PUF EQL (ug/Sample)
PCB-1016	0.10
PCB-1221	0.10
PCB-1232	0.10
PCB-1242	0.10
PCB-1248	0.10
PCB-1254	0.10
PCB-1260	0.10

6.0 INTERFERENCES

6.1 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents or sample processing hardware; contaminated GC carrier gas, parts, column surfaces or detector surfaces; and the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are previously referenced, samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

6.2 Interferences introduced during sample collection and preparation can pose a major problem in organic analytical determinations. Cross-contamination of improperly cleaned glassware may occur. Proper organic glassware cleaning techniques, as specified in the laboratory procedure, ENV-001, current revision, can minimize the potential for error associated with contaminated glassware.

6.3 Carryover may occur after the analysis of a high-level sample. Analysis of a PUF blank is needed to demonstrate that no carryover contamination occurs within the preparation and analytical system.

7.0 INSTRUMENTATION AND MATERIALS

7.1 Gas Chromatograph equipped with dual electron capture detectors, split/splitless injector, autosampler, and PE Nelson data system (or equivalent).

7.1.1 Primary column: DB-5 (or equivalent), 30m x .32mm I.D. fused silica capillary column bonded with 5% (phenyl)methylpolysiloxane, 0.25µm film thickness. Confirmation column: DB-1701P (or equivalent), 30m x 0.32 mm I.D. fused silica capillary column chemically bonded with 14% cyanopropylphenylmethylpolysiloxane, 0.25µm film thickness.

7.1.2 Gas chromatographic conditions (recommended) for Hewlett-Packard 6890 Plus GC:

Injector temperature:	250°C
Detector(s) temperature:	300°C
Detector(s) range:	250
Injection volume:	1 µL
Column head pressure:	17 PSI
Temperature program:	180°C for 4 minutes, 10°C per minute to 275°C and held for 9 minutes.

Instrument conditions may be modified to optimize separation.

7.2 Soxhlet extraction apparatus consisting of a heating source, 300 ml ball flask, Soxhlet Extraction Tube, Allihn condenser connected to a cool water source at the low connection and a water discharge at the top connection.

7.3 Teflon Boiling Chips

7.4 Screw cap vials

7.5 Autosampler vials with crimp caps.

7.6 Volumetric cylinder, 1000ml.

7.7 Microliter syringes.

7.8 K-D concentration apparatus consisting of an evaporator flask with a 3-ball Snyder column.

7.9 Large glass stirring rod.

7.10 Extract Receivers, graduated, for final concentration of extract on N-EVAP concentration system.

7.11 Gravity Funnel

7.12 Glass Wool

7.13 Volumetric flasks, various sizes.

8.0 REAGENTS

8.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 The following solvents are necessary for extraction and standards solution preparation. All solvent lots should be pesticide quality or equivalent and should be determined to be phthalate-free.

8.2.1 Acetone

8.2.2 Hexane

8.2.3 Diethyl Ether

8.2.4 Sodium sulfate, anhydrous.

9.0 STANDARD SOLUTIONS AND CALIBRATION PROCEDURE

9.1 Stock standard solutions:

9.1.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors.

9.1.2 This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in the sample.

9.1.3 The certified stock standard solutions of each of the seven Aroclors is purchased from Ultra Scientific, or similar provider, at a concentration of 1,000 µg/ml. The stock standard solution will be stored in screw-cap bottles at room temperature and protected from light.

9.1.3.1 Stock standards should be checked frequently for signs of degradation or evaporation, especially prior to preparation of calibration standards.

9.1.3.1 Stock standard solutions must be replaced after one year or sooner if routine QC indicates a problem.

9.2 Working Aroclor standard solutions:

9.2.1 An intermediate stock standard (ISS): Prepare an intermediate stock standard (ISS) in hexane as follows:

Compound	Amount Spiked	Final Volume	Concentration in Hexane
Aroclor 1016	100 µL of 1,000 µg/ml stock	10 ml	10 µg/ml
Aroclor 1260	100 µL of 1,000 µg/ml stock	10 ml	10 µg/ml
TCMX (Surrogate)	20 µL of 200 µg/ml stock	10 ml	.4 µg/ml
DCBP (Surrogate)	20 µL of 200 µg/ml stock	10 ml	.4 µg/ml

9.2.2 Store the ISS in a screw cap vial with Teflon-lined seals at room temperature. The ISS solution is stable for a six-month period.

9.2.3 Calibration standards, for both Aroclor 1016 and Aroclor 1260, are prepared in hexane at a minimum of five concentration levels as follows:

Standard	Amount of ISS Spiked	Final Volume	Aroclor 1016/1260	TCMX/DCBP Surrogates
0.05	5 µL	1 ml	0.05 µg/ml	0.002 µg/ml
0.50	50 µL	1 ml	0.50 µg/ml	0.02 µg/ml
1.0	100 µL	1 ml	1.0 µg/ml	0.04 µg/ml
2.0	200 µL	1 ml	2.0 µg/ml	0.08 µg/ml
4.0	400 µL	1 ml	4.0 µg/ml	0.16 µg/ml

9.2.3.1 Working calibration solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

9.2.4 Single working standards at a concentration of .50 µg/ml of each of the other five Aroclors are required to aid the analyst in pattern recognition. The Aroclor 1016/1260 standards, described in section 9.2.3, are used to demonstrate the linearity of the detector, but the remaining five Aroclors are used to determine the calibration factor for the quantitation of each Aroclor.

9.3 Surrogate spiking solutions: Sample preparation and extraction is monitored using surrogate compounds. Surrogate standards are added to all samples, blanks, and laboratory control samples.

9.3.1 The surrogate stock standard is purchased from Ultra Scientific, or similar provider, at concentrations of 200 µg/ml for decachlorobiphenyl and tetrachloro-meta-xylene and is diluted to 1 µg/ml of each by placing 0.5 ml of the stock solution into 100 ml hexane.

9.3.2 Working surrogate spiking solutions must be replaced after six months, or sooner, if comparison to check standards indicates a problem.

9.4 Quality control spiking solution: the Laboratory Control Sample (LCS) spiking solution should be prepared at 10 µg/ml of the suspected Aroclor. If the Aroclor is unknown, Aroclor 1260 will be used.

9.4.1 The quality control stock standard is purchased from Ultra Scientific, or similar provider, at concentrations of 1000 µg/ml and is diluted to 10 µg/ml by placing 1 ml of the stock solution into 100 ml hexane.

9.4.2 Additional or Aroclors other than 1260 may be used for spiking depending on the project or client needs.

9.5 Calibration Procedure for Aroclors: Inject into the gas chromatograph each level of the 1016/1260 calibration standards as described in section 9.2.3. Record peak area or peak height for a minimum of three, and preferably five, peaks for each Aroclor for each injection. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak and at least one peak should be unique to that Aroclor. Using the external standard calibration procedure construct a calibration curve by entering into the PE Nelson TurboChrome data quantitation program the concentration and instrument response of each analyte for the five concentration levels. Typically the calibration curve is constructed using a linear fit of the data. A (r^2) of 0.99 or better is required.

9.5.1 In general a linear fit equation follows the $y = mx + b$ formula. The analyst must be careful that no sample values exceed the value of the high standard.

9.6 Inject the other five mid-level Aroclor standards used for pattern recognition. These standards are also used to determine a single-point calibration for each Aroclor, assuming that the Aroclor 1016/1260 mixture has been used to describe the detector's linear response.

9.6.1 The pattern recognition standards should be analyzed prior to the analysis of any samples, and may be analyzed before or after the analysis of the five 1016/1260 standards.

9.6.2 When specified Aroclors are listed for a project and those Aroclors have been substituted for the 1016/1260 mixture, the pattern recognition samples may be disregarded.

10.0 SAMPLE EXTRACTION

10.1 Prepare sufficient quantity of 5% diethyl ether in hexane to allow approximately 250-300 ml per PUF sampler, blank and LCS to be extracted.

10.2 Each polyurethane foam plug is removed from the filter cartridge, spiked with the appropriate spiking solutions and placed in a clean Soxhlet extraction tube.

10.1.1 If a particulate filter is included in the sampling apparatus, add the filter to the Soxhlet tube with the appropriate PUF sample.

10.1.2 For blank analysis, place a clean PUF in a Soxhlet extractor tube.

10.1.3 For LCS analysis, place a clean quality control solution spiked PUF in a Soxhlet extractor tube.

10.1.4 Surrogate spiking solution is spiked directly onto each PUF sample, blank, and LCS.

10.2 The Soxhlet extraction tube is connected to a clean, labeled ball flask containing approximately 6-10 Teflon boiling chips.

10.3 Carefully pour approximately 300 ml of 5% diethyl ether in hexane into the Soxhlet extractor to ensure that the siphon tube is clear and that the extractor will cycle as necessary.

10.4 The Allihn condenser is then placed into the ground glass joint of the Soxhlet extractor and the ball flask is placed into a heating mantle. The complete Soxhlet extractor apparatus is seen in Appendix 16.1.

10.4.1 Check each of the connections on the Soxhlet extraction apparatus to ensure that each ground glass connection is tight and ensure that there will be no excessive solvent loss due to loose connections.

10.5 The water flow to the Allihn condenser is adjusted to maintain a reduced temperature in the condenser to prevent solvent loss during the extraction cycle.

10.6 The heating mantle is adjusted to enable the solvent to cycle through the Soxhlet extractor from 4 to 6 times per hour.

10.7 The Soxhlet extractor should remain on the heating mantle for approximately 16 hours allowing for 64 to 96 total cycles of solvent through the PUF cartridge.

10.8 Following extraction the heating mantle is turned off and the extractors are allowed to cool.

10.9 Remove the Soxhlet extractor and the ball flask from the Allihn condenser and the heating mantle. Do not compromise the seal between the Soxhlet extraction tube and the ball flask.

10.10 Insert a large glass-stirring rod into the Soxhlet extraction tube to depress the PUF material to enable the final siphon of solvent from the Soxhlet. Additional clean hexane may be added to the Soxhlet to aid in the siphon process and to rinse the Soxhlet extraction tube and PUF plug.

10.11 Remove the Soxhlet extraction tube from the ball flask containing the entire sample extract.

10.12 Assemble a gravity funnel with the stem plugged loosely with glass wool.

10.13 Place approximately 20 grams of anhydrous sodium sulfate in the bell of the funnel.

10.14 Place the stem of the funnel into a Kuderna-Danish Evaporator (K-D) and slowly pour the ball flask contents into the gravity funnel, through the sodium sulfate, and into the K-D to remove any possible water from the extract. Rinse the ball flask with a small amount of clean hexane and pour the rinsate through the gravity funnel allowing collection in the K-D.

10.15 Place approximately 3-5 Teflon boiling chips to the K-D and attach a 3-ball Snyder column to the ground glass opening of the K-D. See Appendix 16.2.

10.16 Place the K-D assembly onto the water bath so that the bottom of the K-D evaporator is submerged in the water to allow for concentration of the sample to a working volume.

10.17.1 The temperature of the water bath should be between 89-99°C.

10.17 When the extract reaches a volume of approximately 10 ml, remove the K-D assembly from the water bath and allow the assembly to cool sufficiently to pour the contents from the K-D to the N-EVAP receiver.

10.18 Rinse the K-D with a small amount of clean hexane and include the rinsate in the receiver.

10.19 Place the receiver and its contents on the N-EVAP for concentration to a final volume of 1 ml. Place a disposable Pasteur pipette in the N-EVAP system and adjust the nitrogen flow to assist in concentration, but not so vigorously as to cause sample loss from agitation.

10.19.1 The N-EVAP suggested temperature for final concentration is 39-49°C.

11.0 ANALYTICAL PROCEDURE

11.1 Optimize the instrumental conditions for resolution of the target compounds and sensitivity. A final temperature of 240-270°C may be required to elute decachlorobiphenyl. The use of an injector pressure program will improve the chromatography of late eluting peaks.

11.1.1 When the operating conditions are established, the same parameters must be used for both calibrations and sample analysis.

11.2 Prepare calibration standards using the procedures in Sections 9.1 through 9.2. Calibrate the instrument by using the procedure found in Sections 9.5 through 9.6.

11.3 Initial calibration verification is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. The instrument response must be treated as the dependent variable (y) and the concentration as the independent variable (x).

11.3.1 Linear regression will produce the slope and intercept terms for a linear equation in the form: y=mx+b where, y is the instrument response (peak area or height), m is the slope of the line (also called the coefficient of x), x is the concentration of the calibration standard and b is the intercept.

11.3.2 The analyst should not force the line through the origin, but have the intercept calculated from the five data points.

11.3.3 The regression calculation will generate a correlation coefficient (r^2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

11.4 For continuing calibration verification (prior to the analysis of samples at the beginning of each 12 hr shift, after at least every 20 samples, and at the completion of the analytical sequence), a calibration check standard, or the specific project defined mix used for initial calibration, must be injected. The Aroclors, analyzed during the initial calibration, and used for pattern recognition are not re-injected as a part of the calibration verification process.

11.4.1 The response (or calculated concentration) for each analyte calculated from the continuing calibration verification standard (CF_v) must not exceed 15% when compared to the initial calibration curve. The difference is calculated by:

$$\% \text{ Difference} = (C_i - C_c)/C_i * 100$$

where:

C_i = Calibration Check Compound standard concentration
C_c = Measured concentration using selected quantitation method.

11.4.2 If the 15% criteria is exceeded for any analyte response (or concentration), inspect the instrument to determine the cause and perform any maintenance necessary before re-verifying the calibration and proceeding with sample analysis.

11.4.3 If routine maintenance does not return the instrument performance to meet the requirements based on the last initial calibration, the analyst must generate a new initial calibration.

11.5 Retention time windows: The retention time range for Aroclors is defined during initial calibration. For multi-component standards, such as Aroclors, the analyst should use the retention time window but should primarily rely on pattern recognition. The experience of the analyst should weigh heavily in the interpretation of the chromatograms.

11.6 Following acceptable calibration, sample injection may begin and continue for as long as the calibration verification standards interspersed with the samples meet instrument QC requirements. The sequence ends when the set of samples has been injected or when quantitative calibration verification criteria are exceeded.

11.6.1 Each positive sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12 hr shift), or calibration standards interspersed within the samples.

11.6.1.2 If the calibration verification criteria is exceeded prior to the completion of the analysis, all positive samples that were injected following the standard that last met the QC criteria must be re-analyzed within acceptable verification standards.

11.6.1.3 If the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit and the analyte was not detected in any of the previous samples during the analytical sequence, then the sample extracts do not require re-analysis, as the verification standard has demonstrated that the analyte would have been detected were it present.

11.6.2 Analyze an extraction blank to verify that there is no contamination caused by reagents or the extraction process. A set of blanks is required for every group of samples extracted as a batch. A batch may include up to 20 samples of a similar matrix extracted at one time.

11.6.3 A laboratory control sample (LCS) is required for every batch of samples extracted. Report spike recoveries as part of the analytical report. See Section 14.2 for recovery limits.

11.6.4 Calculate surrogate spike recoveries. Report surrogate spike recoveries as part of the analytical report. See Section 14.2 for recovery limits.

12.0 CALCULATIONS

12.1 When the linear calibration curve is employed, the µg/ml analyte concentration in the sample extract (x) is determined by the TurboChrome software program using the formula:

$$x=(y-b)/m$$

where y is the instrument response, b is the intercept of the initial calibration curve and m is the slope of the initial calibration curve.

12.2 When using linear calibration, it is the analyst's responsibility to ensure that the reported values take into account the volume or weight of the original sample and the dilution factor, if applicable. The following formulas are used to determine the concentration of the analyte in the original sample extracted.

12.2.1 $\mu g / \text{Sample} = X * B$

where: X is the concentration of the analyte found in the sample extract,
B is the final volume of extract in milliliters.

12.3 LCS/Surrogate percent recovery determination:

$$\text{Percent Recovery} = 100 * \frac{\text{Measured Value}}{\text{Target Value}}$$

13.0 QUALITY ASSURANCE PROVISIONS

13.1 Quality control requirements are included in the procedure section of SOP (section 11.0).

13.2 Control limits can be found under the TO-10A testcode in the laboratory's LIMS. Control limits are subject to change and may be updated according to laboratory standard operating procedure. The default surrogate recovery limit of 60-120 is used for accuracy until sufficient data is accumulated to determine laboratory limits.

13.2.2 If spike recovery of the above listed parameters is not within limits, the following is required:

13.2.2.1 Check to be sure there are no errors in calculations, surrogate solutions, or compound identification. Also, check instrument performance.

13.2.2.2 Re-calculate the data and/or re-analyze the extract if any of the above checks reveal a problem.

13.2.2.3 If recovery still fails the QC criteria, the data will be flagged with an appropriate qualifier and explained.

13.3 A PUF blank shall be analyzed for each analytical batch of up to 20 samples. (An analytical batch is defined as a group of samples analyzed at the same time and having similar matrix).

13.3.1 PUF control limit:

Less than the highest of either:

(1) The method detection limit,
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

Corrective Action:

(1) Check for calculation errors, instrument performance
(2) Re-analyze blank and samples
(3) Re-extract and re-analyze samples
(4) Flag data if there is not sufficient sample for re-analysis or if the contamination persists

13.4 Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method as specified in this procedure. Initial proficiency is generally demonstrated by the preparation and analysis of a method detection limit study on clean blank PUF cartridges. The laboratory must also repeat the following operations whenever new staff members are trained or significant changes in instrumentation are made.

13.4.1 Reference samples are prepared from a spiking solution containing Aroclor 1016/1260. The reference sample concentrate(s) may be prepared from pure standard materials or purchased as certified solutions. The reference sample concentrate(s) must be made using stock standards prepared independently from those used for calibration.

13.4.2 To adequately evaluate the performance of the total analytical process, the reference samples must be handled in exactly the same manner as actual samples.

13.4.3 Analyze seven replicate extracts and perform the calculations as listed in the laboratory procedure for method detection limits, DCLC-SOP-020 or current revision.

13.4.4 The performance of the method is acceptable as long as the client's detection limit needs are met within this study.

14.0 REPORTING RESULTS

14.1 Results should be reported in units of µg/sample or in accordance with the requirements of the specific client. Results will also be reported in µg/m^3 if the client provides sample air volumes.

14.1 To convert µg/sample to µg/m^3 simply divide the ug/sample by the air volume in liters.

15.0 PREVENTIVE MAINTENANCE

15.1 Due to the sensitivity of the electron capture detector, the injection port liner should be cleaned or replaced prior to performing the initial calibration.

15.2 Remove the first few inches of the injector side of the column.

15.3 Solvent flush the metal injector body.

15.4 Replace the column.

15.5 Clean or re-foil ECD (must be performed by manufacturer).

16.0 APPENDICES

16.1 Completed Soxhlet Extractor Apparatus

16.2 Kuderna-Danish Concentration Assembly

17.0 PROCEDURAL REVISIONS & MODIFICATIONS

17.1 Revisions from Revision 0 to Revision 1 of this procedure.

17.1.1 Addition of section 1.5.1.

17.1.2 Addition of section 4.1.

17.1.3 Addition of section 9.4.2.

17.1.4 Update of section 13.2

17.1.5 Addition of Section 17.0.

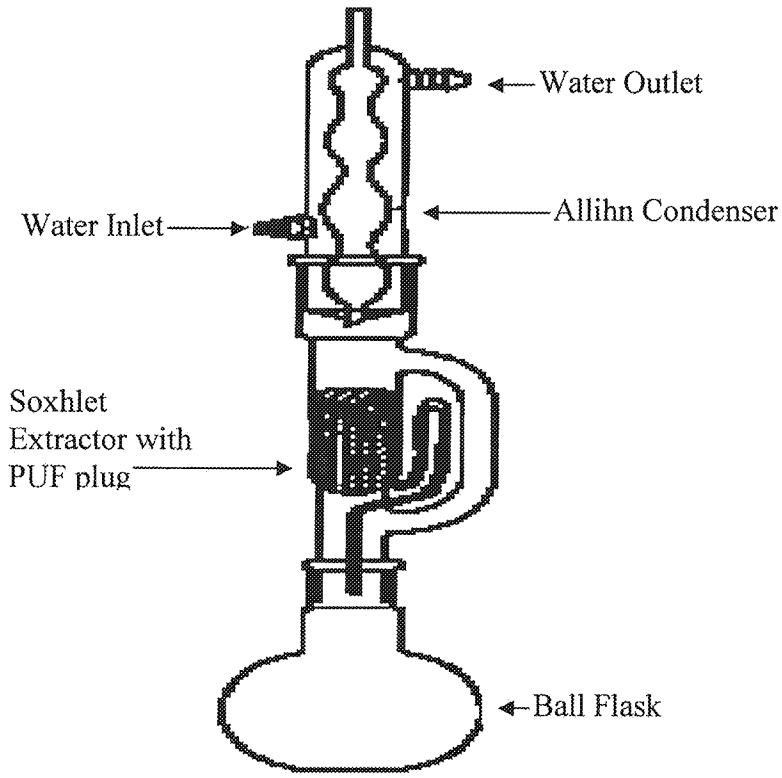
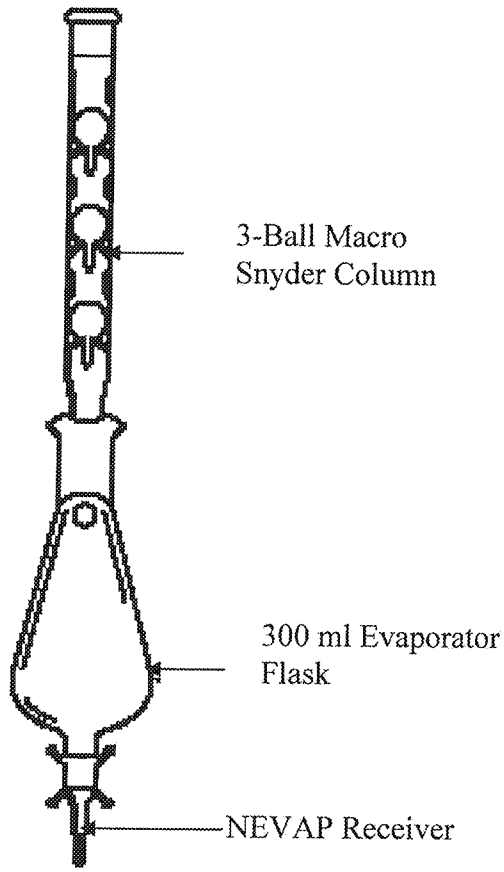
17.1.6 Update section 18.0.

18.0 REFERENCES

18.1 "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," EPA Method 8082, Version 2, USEPA SW-846, December 1997.

18.2 "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air," EPA Method TO-10A, Second Edition, January 1999, EPA/625/R-96/010b.

Appendix 16.1 Completed Soxhlet Extractor Apparatus



Appendix 16.2 Kuderna-Danish Concentration Assembly