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MULTI-CLASS, MULTI-RESIDUE METHOD FOR THE ANALYSIS OF CHLOROBENZENES, POLYCHLORINATED BIPHENYLS, POLYNUCLEAR AROMATIC HYDROCARBONS, AND PHTHALATE ESTERS IN NATURAL WATERS

(GAS CHROMATOGRPAHIC)

by

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EXECUTIVE SUMMARY

A multi-class, multi-residue method was developed for the simultaneous analysis of chlorobenzenes, PCBs, polynuclear aromatic hydrocarbons (PAHs), and phthalate esters in water at ng/L levels. This procedure has the potential to replace the methods currently in use for each individual class of compounds whenever multi-residue results are required. By application of this new procedure, sampling and analytical costs can be greatly reduced.

RÉSUMÉ ADMINISTRATIF

On a mis au point une méthode multiclasses et multirésidus permettant de doser simultanément les chlorobenzènes, les BPC, les hydrocarbures aromatiques polynucléaires et les esters phtaliques dans l'eau à des concentrations de l'ordre du ng/L. Cette méthode permettra peut-être de remplacer celles actuellement en usage pour chaque classe de composés, chaque fois que des résultats multiclasses sont requis. Elle permettra aussi de réduire de beaucoup les coûts d'échantillonnage et d'analyse.

ABSTRACT

A multi-class, multi-residue method was developed for the simultaneous analysis of ten chlorobenzenes, total PCBs, 16 PAHs (US EPA Method 610), and six phthalate esters (US EPA Method 606) in natural water at ng/L levels. The organics were extracted from water by methylene chloride and the solvent was then evaporated using a three-stage Snyder column. After replacement of solvent with hexane, the extract was cleaned up and fractionated on an activated silica gel column. Fraction A contained the chlorobenzenes and PCBs, fraction B contained the PAHs and fraction C contained the phthalate esters. Chlorobenzenes, PCBs and phthalate esters were analyzed by GC-ECD while the PAHs were analyzed by GC-MSD. Based on a 1 L sample and the extract reduced to a final volume of 1 mL, the method detection limits were between 0.4 and 5 ng/L for chlorobenzenes, 3 ng/L for Aroclors, between 10 and 50 ng/L for PAHs and 200 ng/L for phthalate esters.

RÉSUMÉ

On a mis au point une méthode multiclasses et multirésidus permettant de doser simultanément dix chlorobenzènes, les BPC totaux, 16 HAP (méthode 610 de l'EPA des É.-U.) et six esters phtaliques (méthode 606 de l'EPA des É.-U.) dans l'eau naturelle à des concentrations de l'ordre du ng/L. On a extrait les produits organiques de l'eau avec du chlorure de méthylène, puis on a chassé le solvant par évaporation en utilisant une colonne Snyder à trois corps. Après le remplacement du solvant par de l'hexane, l'extrait a été nettoyé et fractionné sur une colonne de gel de silice activé. La fraction A contenait les chlorobenzènes et les BPC, la fraction B les HAP et la fraction C les esters phtaliques. Les chlorobenzènes, les BPC et les esters phtaliques ont été dosés par CG-DCE et les HAP par CG-DSM. Dans le cas d'un échantillon de 1 L et d'un extrait réduit à un volume final de 1 mL, les limites de détection de la méthode étaient de 0,4-5 ng/L pour les chlorobenzènes, de 3 ng/L pour les Aroclors, de 10-50 ng/L pour les HAP et de 200 ng/L pour les esters phtaliques.

1. SCOPE AND APPLICATION

1.1 This method is applicable to the qualitative and quantitative determination of ten chlorobenzenes, polychlorinated biphenyls (PCBs), 16 polynuclear aromatic hydrocarbons (PAH) and six phthalate esters simultaneously in natural waters.

1.2 The method detection limits based on a 1-L water sample and an extract of 1.0 mL final volume are as follows:

Parameter	Method Detection Limits (ng/L)	NAQUADAT No.
Chlorobenzenes		
1,4-dichlorobenzene	5.0	
1,3-dichlorobenzene	5.0	
1,2-dichlorobenzene	5.0	
1,3,5-trichlorobenzene	1.0	
1,2,4-trichlorobenzene	1.0	
1,2,3-trichlorobenzene	1.0	
1,2,4,5-tetrachlorobenzene	1.0	
1,2,3,4-tetrachlorobenzene	1.0	
Pentachlorobenzene	1.0	
Hexachlorobenzene	0.4	
Polychlorinated biphenyls		
Aroclor 1242	3.0	
Aroclor 1254	3.0	
Aroclor 1260	3.0	

Polynuclear aromatic hydrocarbons	
Napthalene	10.0
Acenaphthylene	10.0
Acenaphthene	10.0
Fluorene	10.0
Phenanthrene	10.0
Anthracene	10.0
Fluoranthene	10.0
Pyrene	10.0
Benzo[a]anthracene	10.0
Chrysene	10.0
Benzo[b]fluoranthene	20.0
Benzo[k]fluoranthene	20.0
Benzo[a]pyrene	20.0
Indeno[123-cd]pyrene	50.0
Dibenz[ah]anthracene	50.0
Benzo[ghi]perylene	50.0
Phthalate esters	
Dimethyl phthalate	200*
Diethyl phthalate	200
Dibutyl phthalate	200
Butyl benzyl phthalate	200
Di(2-ethylhexyl) phthalate	200
Di-n-octyl phthalate	200

*Method detection limits for phthalate esters are highly blank dependent.

2. PRINCIPLE AND THEORY

2.1 Organics in water are extracted at neutral pH with dichloromethane.

2.2 The organic extract is dried and solvent is evaporated down and replaced by hexane using a three-stage Snyder column.

2.3 Using an activated silica gel column and solvents of increasing polarity, the sample extract is cleaned and fractionated to give three fractions. Each fraction is again evaporated down to either 1.0 or 10.0 mL. Fraction A contains the chlorobenzenes and PCBs, fraction B contains the PAH and fraction C contains the phthalate esters.

2.4 Fractions A and C are analyzed by GC-ECD and fraction B is analyzed by GC-MSD with capillary and packed columns.

2.5 The method presented here can be used to analyze any one of, or combination of, the four classes of compounds, if required.

3. INTERFERENCES

3.1 Extraneous matter, especially in highly coloured water samples, is a potential interference. The cleanup procedures described in this method will usually eliminate this source of interference.

3.2 Other chlorinated compounds, pesticide residues, metabolites or degradation products may interfere in the GC-ECD analysis. In such cases, confirmation of compound identity by a second high resolution capillary column of different polarity or by GC-MS is necessary.

3.3 Other hydrocarbons and aromatic hydrocarbons may interfere in the GC-MSD analysis of PAH. Monitoring of a second charasteristic ion will usually be able to eliminate such interference.

3.4 The use of plastic tubing and/or containers in the sampling, extraction and cleanup procedures must be completely avoided. Contact of sample or sample extract with any plastic substance may cause severe interference for phthalate ester analysis. See also Section 11.6.

4. SAMPLING PROCEDURE AND STORAGE

4.1 Water samples should be collected and stored in an all-glass system since this method is concerned with organic constituents only.

4.2 Teflon-lined bottle caps are recommended to prevent contact and contamination of the sample from the plastic cap. An acceptable alternative is the use of solvent-washed aluminum foil beneath the cap.

4.3 Samples should be stored in the dark at 4°C and extracted as soon as possible.

5. APPARATUS

5.1 Packed column GC-ECD analysis for PCBs.

5.1.1 A gas chromatograph equipped with a heated injection port and a Ni-63 electron-capture detector such as Hewlett-Packard Model 5710A or equivalent can be used.

5.1.2 Automatic liquid sampler such as Hewlett-Packard Model 7671A or 7672A or equivalent. If this is not available, use a 10 µL Hamilton micro-syringe and the solvent-flush technique for injections.

5.1.3 GLC glass columns (1.8 m x 2 mm i.d.) packed with 3% OV-1 on Gas Chrom Q, 100/120 mesh, available from Chromatographic Specialties Ltd.

5.1.4 Chromatographic conditions for GC-ECD analysis of PCBs in Fraction A:

Injection port temperature:	250°C
Detector temperature:	300°C
Column oven temperature:	185°C (isothermal)
Carrier gas:	Argon/methane (95+5) at 30 mL/min
Injection volume:	5 to 10 µL

5.2 Capillary column GC-ECD analysis of chlorobenzenes.

5.2.1 A gas chromatograph equipped with a split/splitless injection port, Ni-63 electron-capture detector, and an oven with multi-level temperature programming capability such as Hewlett-Packard Model 5880A or 5890A or equivalent is suitable.

5.2.2 Sample introduction device same as 5.1.2.

5.2.3 Fused silica capillary column: 12 m x 0.2 mm i.d. column coated with cross-linked dimethyl silicone gum (0.33 µm thickness) and surface deactivated by siloxane, such as Hewlett-Packard OV-1 (Part No. 19091-60312), or equivalent.

5.2.4 Chromatographic conditions for chlorobenzene analysis (Fraction A):

Injection port:	splitless mode, splitless valve on for 30 s
Injection port temperature:	250°C
Detector temperature:	300°C
Detector make-up gas:	Argon/methane (95+5) at 25 mL/min
Carrier gas:	Helium at 10 psi
Septum purge:	3 mL/min
Split vent:	50 mL/min
Column oven initial temperature:	40°C, hold for 0.5 min
Programming rate 1:	30°C/min (40° to 70°C), hold for 5.0 min at 70°C
Programming rate 2:	8°C/min (70° to 140°C), hold for 10 min at 140°C.

5.3 Capillary column GC-ECD analysis of phthalate esters.

5.3.1 The gas chromatograph, automatic liquid sampler and capillary column used are the same as 5.2.1, 5.2.2 and 5.2.3.

5.3.2 Chromatographic conditions for phthalate ester analysis (Fraction C):

Injection port:	splitless mode, splitless valve on for 30 s
Injection port temperature:	250°C
Detector temperature:	300°C
Detector make-up gas:	Argon/methane (95+5) at 25 mL/min
Carrier gas:	Helium at 10 psi
Septum purge:	3 mL/min
Split vent:	50 mL/min
Column oven initial temperature:	70°C, hold for 0.5 min
Programming rate 1:	25°C/min (70° to 180°C)
Programming rate 2:	2°C/min (180° to 230°C), hold for 15 min at 230°C.

5.4 Capillary column GC-MSD analysis of PAH.

5.4.1 A gas chromatograph equipped with a split/splitless injection port, an oven with multi-level temerature programming capability, and a mass selective detector with direct capillary interface such as the Hewlett-Packard 5890A and 5970B combination is suitable.

5.4.2 A Hewlett-Packard series 200 or 300 computer complete with floppy disc drive, hard disc drive, printer, monitor and existing GC-MSD software is required.

5.4.3 Automatic liquid sampler same as 5.1.2.

5.4.4 Fused silica capillary column: 30 m x 0.25 mm i.d. DB-5 (0.25 µm film thickness) available from J&W Scientific, Inc., or equivalent.

5.4.5 Chromatographic conditions for PAH analysis (Fraction B):

Injection port:	splitless mode, splitless valve on for 1.5 min
Injection port temperature:	275°C
Interface temperature:	280°C
Carrier gas:	Helium at 4 psi
Septum purge:	3 mL/min
Split vent:	50 mL/min
Column oven initial temperature:	70°C, hold for 1.5 min
Programming rate 1:	30°C/min (70° to 160°C)
Programming rate 2:	2.5°C/min (160° to 280°C), hold for 20 min at 280°C.

5.4.6 Acquire GC-MSD data in the SIM mode by monitoring the following characteristic ions:

	Ion Group	Characteristic Ions (m/z)	
		Quantitation Ion	Confirmation Ion
Naphthalene	1	128	129
Acenaphthylene	2	152	151
Acenaphthene	2	154	153
Fluorene	3	166	165
Phenanthrene	4	178	179
Anthracene	4	178	179
Fluoranthene	5	202	101
Pyrene	5	202	101
Benzo[a]anthracene	6	228	229
Chrysene	6	228	229
Benzo[b]fluoranthene	7	252	253
Benzo[k]fluoranthene	7	252	253
Benzo[a]pyrene	7	252	253
Indeno[123-cd]pyrene	8	276	138
Dibenz[ah]anthracene	8	278	139
Benzo[ghi]perylene	8	276	138

6. **REAGENTS**

6.1 All solvents must be distilled-in-glass, pesticide residue grade and must be checked for low blank values.

6.1.1 Dichloromethane.

6.1.2 Hexane.

6.1.3 Pentane.

6.1.4 Acetone.

6.1.5 Isooctane.

6.1.6 Toluene.

6.2 All chemicals must be of highest purity.

6.2.1 Organic-free water. Pass distilled water through Millipore Super-Q unit (Millipore Corp.).

6.2.2 Sodium sulfate (anhydrous, reagent grade) available from BDH chemicals. Heat at 650°C for 18 hours and store in a clean glass bottle in a dessicator.

6.2.3 Activated silica gel. Heat Grade 950 silica gel (100 to 200 mesh) at 130°C for 18 hours. Cool to room temperature and store in a tightly-capped glass bottle in a dessicator until use. Reactivate adsorbent weekly.

6.3 All analytical standards must be of highest (98+) and known purity. Obtain from manufacturers or U.S. Environmental Protection Agency and use without further purification.

6.3.1 Prepare 1000 ppm stock solutions of each individual compound of interest by dissolving 100 mg of pure analytical standard in an appropriate solvent and diluting to 100.0 mL in "low-actinic"

volumetric flasks. Stock solutions of chlorobenzenes and Aroclors are prepared in isooctane. Stock solutions for PAH and phthalate esters are prepared in toluene. Store all stock solutions as 4°C in the dark.

Note: Some PAH may precipitate out upon cold storage at the 1000 ppm level. If this happens, a stock solution of lower concentration should be prepared.

6.3.2 Prepare mixed stock solutions for each class of compounds by combining appropriate aliquots of each individual stock solution and dilute to 100.0 mL with either isooctane (for chlorobenzenes and PCBs) or toluene (for PAH and phthalate esters).

6.3.3 Prepare working standards for each class of compounds by further dilution of the mixed stock solutions with appropriate solvents. Use these working standards for instrument calibration.

7. PROCEDURE

7.1 Extraction

7.1.1 Stir a 1-L water sample in a 1.14 L whiskey bottle or other suitable glass container on a magnetic stirrer, using a Teflon-coated stirring bar so that the vortex formed at the surface almost reaches the bottom of the bottle.

7.1.2 Add 50 mL of dichloromethane and tightly cover the bottle with a Teflon-lined cap. After stirring for 30 min, transfer the contents of the bottle to a 1-L separatory funnel.

7.1.3 Drain the organic (bottom) layer into a clean 500-mL round bottom flask.

7.1.4 Return the aqueous layer to its original container. Rinse the 1-L separatory funnel with 30 and 20 mL aliquots of dichloromethane and transfer to the sample bottle. Tightly cover the bottle and stir for 30 min. Transfer the contents of the bottle to the 1-L separatory funnel.

7.1.5 Transfer the organic layer to the 500-mL round bottom flask containing the first 50 mL of dichloromethane extract.

7.1.6 Repeat steps 7.1.4 and 7.1.5 with another 50 mL of dichloromethane. Discard the aqueous layer after the last extraction.

7.1.7 Drain the combined organic layer through a vacuum sintered glass funnel containing 50 mm of anhydrous sodium sulfate. Collect the dried extract in a clean 500-mL round bottom flask.

7.1.8 Rinse the separatory funnel and the empty 500 mL round bottom flask twice with 25 mL dichloromethane, pass the rinsings through the sodium sulfate column and collect the filtrate in the 500-mL round bottom flask in 7.1.7. Remove the last trace of solvent in the sodium sulfate column with suction.

7.1.9 Add 3 mL of isooctane and a few boiling chips to the organic extract. Attach a three-stage Snyder column prewet with 5 mL

of hexane to the flask and evaporate the solvent to ca. 5 to 10 mL with a heating mantle.

7.1.10 Allow the column and the flask to cool to room temperature and carefully add 50 mL of hexane to the concentrated extract. Repeat evaporation to 3 mL. Do not let the extract go dry.

7.2 Column Cleanup with Activated Silica Gel

7.2.1 Fill a 500 mm x 10 mm i.d. chromatographic column equipped with a sintered disc and teflon stopcock with 5.00 g of activated silica gel using a hexane slurry. Be careful to avoid any air bubble trapped inside the column bed. Top column with 1 cm of anhydrous sodium sulfate.

7.2.2 Rinse the column with another 50 mL of hexane and discard the rinsing.

7.2.3 Quantitatively transfer the concentrated sample extract from step 7.1.10 plus rinsings onto the column with a disposable Pasteur pipet.

7.2.4 When the extract just enters the sodium sulfate layer, elute the column with a 50 mL of pentane and collect the eluant in a 250-mL round bottom flask. This is Fraction A and it contains the chlorobenzenes and PCBs.

7.2.5 Continue the elution of the silica gel column with 60 mL of 40+60 dichloromethane/hexane and collect the eluant into a second 250-mL round bottom flask. This is Fraction B and it contains the PAH.

7.2.6 Elute the same column again with 50 mL of 10+90 acetone/hexane and collect the eluant into a third 250 mL round bottom flask. This is Fraction C and it contains the phthalate esters.

7.2.7 Add 1 mL of isooctane and a few boiling chips to Fraction A. Attach a three-stage Snyder column prewet with pentane and evaporate the solvent down to ca. 3 to 5 mL.

7.2.8 Allow the Snyder column and round bottom flask to cool to room temperature. Carefully detach the column from the round bottom flask and rinse the joint twice into the round bottom flask with 1 mL of hexane. Quantitatively transfer the extract to a graduated test tube. Reconnect the Snyder column to the round bottom flask and rinse the column with three 1-mL aliquots of hexane. Collect rinsings in the flask and transfer to the test tube. Make up to 10.0 mL with hexane.

7.2.9 For ultra-trace level work, further evaporation of the fraction in step 7.2.8 to 1.0 mL with a two-stage micro Snyder column is required.

7.2.10 Add 1 mL of toluene and a few boiling chips to Fraction B. Evaporate this fraction down to ca. 3 to 5 mL with a three-stage Snyder column as described before. Further reduce the volume to 1.0 mL as per steps 7.2.8 and 7.2.9.

7.2.11 Add 1 mL of toluene to Fraction C and evaporate solvent down to 5 mL with a Rotavapor and a water bath temperature of 35°C. Then add 20 mL of hexane and evaporate solvent again to ca.

3 mL. Make up to 10.0 mL with isooctane in a graduated test tube.

7.3 **GC-ECD and GC-MSD Analysis**

7.3.1 Analyze PCBs in Fraction A by packed column GC-ECD using Webb-McCall quantitation technique as per Section 5.1. Also analyze chlorobenzenes in Fraction A and phthalate esters in Fraction C by capillary column GC-ECD as per Sections 5.2 and 5.3. (Alternatively, PCBs and chlorobenzenes in Fraction A can be analyzed simultaneously if a capillary column GC-ECD method is set up for PCB analysis.)

7.3.2 Analyze PAHs in Fraction B by capillary column GC-MSD as per Section 5.4. (Alternatively, PAHs can be analyzed by HPLC using a combination of UV and fluorescence detectors.)

8. CALCULATIONS

8.1 The concentration of each compound of interest is determined by comparison of peak height or area of the samples with those of the standards. This can be done by using the following equation:

$$X_{sam} = \frac{H_{sam}}{H_{std}} \times \frac{V_{inj\ std}}{V_{inj\ sam}} \times X_{std} \times \frac{V_{ext}}{V_{sam}}$$

where X_{sam} = concentration of organic compound in original water sample (µg/L);

H_{sam} = peak height (or area) of sample;

H_{std} = peak height (or area) of standard;

$V_{inj\ std}$ = volume of standard injected (µL);

$V_{inj\ sam}$ = volume of sample injected (µL);

X_{std} = concentration of organic compound in standard solution (pg/µL);

V_{ext} = final volume of sample extract (mL); and

V_{sam} = volume of original water sample extracted (mL).

9. PRECISION AND ACCURACY

9.1 Single-laboratory precision and accuracy data for this method are summarized below:

Parameter	Fortification Level in 1-L Water (ng/L)	% Recovery Mean ± S.D.
Chlorobenzenes		
1,4-Dichlorobenzene	100	63.1±5.6
1,3-Dichlorobenzene	100	82.6±8.8
1,2-Dichlorobenzene	100	68.8±6.1

1,3,5-Trichlorobenzene	20	68.1±6.4
1,2,4-Trichlorobenzene	20	79.6±6.9
1,2,3-Trichlorobenzene	20	76.1±6.6
1,2,4,5-Tetrachlorobenzene	10	84.0±4.5
1,2,3,4-Tetrachlorobenzene	10	81.2±7.6
Pentachlorobenzene	10	85.1±7.7
Hexachlorobenzene	10	83.2±8.1
Polychlorinated biphenyls		
Aroclor 1242	25	
Aroclor 1254	25	82.5±6.5
Aroclor 1260	25	(total PCBs)
Polynuclear aromatic hydrocarbons		
Naphthalene	100	74.8±7.5
Acenaphthylene	100	80.1±7.0
Acenaphthene	100	80.5±5.2
Fluorene	100	92.8±5.7
Phenanthrene	100	89.0±7.0
Anthracene	100	87.2±4.6
Fluoranthene	100	93.4±9.1
Pyrene	100	92.0±6.7
Benzo[a]anthracene	100	87.7±8.8
Chrysene	100	88.5±8.0
Benzo[b]fluoranthene	100	88.9±8.4
Benzo[k]fluoranthene	100	94.7±7.4

Benzo[a]pyrene	100	79.1±4.5
Indeno[123-cd]pyrene	100	87.6±9.0
Dibenz[ah]anthracene	100	92.7±13.0
Benzo[ghi]perylene	100	93.1±12.4
Phthalate esters		
Dimethyl phthalate	1000	104.6±5.3
Diethyl phthalate	1000	108.4±16.9
Dibutyl phthalate	1000	120.8±12.9
Butyl benzyl phthalate	1000	114.3±10.6
Di(2-ethylhexyl)phthalate	1000	131.0±9.1
Di-n-octylphthalate	1000	77.6±10.6

10. CONFIRMATION OF IDENTITY

10.1 The identity of each GC peak in the sample extract may be assigned by comparison with the retention time of each authentic standard analyzed individually under identical chromatographic conditions.

10.2 The identity of each sample peak may be tentatively confirmed on retention time basis by analyzing the sample and standard with another high resolution capillary column of different polarity.

10.3 Whenever sample concentration permits, additional confirmation of identity may be obtained by combined GC-MS (EI and/or CI) operating at selected ion monitoring or full scan mode. For chlorobenzenes, PCBs and phthalate esters, the following characteristic ions may be used for EI-GC-MS-SIM confirmation purposes.

	Characteristic ions (m/z)
Dichlorobenzenes	146 , 148
Trichlorobenzenes	180 , 182
Tetrachlorobenzenes	216 , 214
Pentachlorobenzenes	250 , 248
Hexachlorobenzenes	284 , 142
Monochlorobiphenyls	188 , 190
Dichlorobiphenyls	222 , 224
Trichlorobiphenyls	256 , 258
Tetrachlorobiphenyls	292 , 290
Pentachlorobiphenyls	326 , 328
Hexachlorobiphenyls	360 , 362
Heptachlorobiphenyls	394 , 396
Octachlorobiphenyls	430 , 432
Nonachlorobiphenyls	464 , 466
Decachlorobiphenyls	498 , 500
Dimethyl phthalate	163 , 194
Diethyl phthalate	149 , 177

Dibutyl phthalate	149 , 150
Butyl benzyl phthalate	149 , 91
Di(2-ethylhexyl)phthalate	149 , 167
Di-n-octyl phthalate	149 , 150

11. REMARKS

11.1 Extreme care must be exercised by the analyst in the concentration of sample extracts. Samples containing chlorobenzenes and PAH must never be evaported by Rota-vapor, otherwise, severe losses of most chlorobenzenes and the volatile PAH will be experienced.

11.2 Analysis of 1,2,3,5-tetrachlorobenzene was omitted because it could not be resolved from the more abundant 1,2,4,5-tetrachlorobenzene on a non-polar OV-1 column. If the analysis of both isomers is required, a polar Carbowax 20 M or DX-4 column can be used.

11.3 Although OC's are not included in this multi-residue method, the analysis of such insecticides can be accommodated without any change in the extraction and cleanup steps. Since all OC's are eluted in fractions A and B, additional capillary column GC-ECD analyses for these parameters are required for both fractions.

11.4 The analysis of chlorinated hydrocarbons such as hexachloroethane, hexachlorobutadiene and octachlorostyrene can again be included without changes in the present analytical scheme. These compounds are eluted with the chlorobenzenes and PCBs in Fraction A.

11.5 Simultaneous analysis of chlorobenzenes and PCBs can be done if a capillary column GC-ECD method is set up for PCB analysis. In this case, a 30 m or longer DB-1 or DB-5 column should be used instead of the 12 m OV-1 column.

11.6 Analysis of phthalate esters at 1 µg/L or lower levels is extremely vulnerable to in-house contamination. Contact of sample or sample extract with any plastics must be completely avoided. All glasswares including the disposable ones such as Pasteur pipets and automatic liquid sampler vials must be scrupulously cleaned, rinsed with pesticide grade toluene, and heated at 200°C for three hours before use. Anhydrous sodium sulfate should also be heated at 650°C for five hours or more before use.

11.7 Chromatographic separation of closely eluted isomeric PAH pairs on the capillary column used must be demonstrated since the isomers are not distinguished by GC-MS. The isomeric pairs are: phenanthrene and anthracene, benzo[a]anthracene and chrysene, benzo[b]fluoranthene and benzo[k]fluoranthene, as well as benzo[e]pyrene and benzo[a]pyrene. It should be noted that chrysene and triphenylene, which have the same molecular ion, are not resolvable on most capillary columns.