METHOD WRITEUP FOR

WATER QUALITY BRANCH

ANALYTICAL METHODS MANUAL

by

Yvonne D. Stokker

Analytical Methods Division

National Water Research Institute

Canada Centre for Inland Waters

Burlington, Ontario, Canada

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METHOD FOR THE ANALYSIS OF CHLOROPHENOLS IN SEDIMENTS

(Gas Chromatographic)

1. SCOPE AND APPLICATION

1.1 This method is applicable to the qualitative and quantitative gas chromatographic determination of the following phenols in sediments:

Phenol	NAQUADAT NO.
2-chlorophenol	
3-chlorophenol	
4-chlorophenol	
2-chloro-5-methylphenol	
2,6-dichlorophenol	
4-chloro-3-methylphenol	
2,4-dichlorophenol	
3,5-dichlorophenol	
2,3-dichlorophenol	
3,4-dichlorophenol	
2,4,6-trichlorophenol	
2,3,6-trichlorophenol	
2,3,5-trichlorophenol	
2,4,5-trichlorophenol	
2,3,4-trichlorophenol	
3,4,5-trichlorophenol	
2,3,5,6-tetrachlorophenol	
2,3,4,6-tetrachlorophenol	
2,3,4,5-tetrachlorophenol	
Pentachlorophenol	

1.2 The practical limits of measurement based on a 50 g sediment sample and using electron capture and/or mass selective detection range from 0.2 to 100 µg/kg.

2. PRINCIPLE AND THEORY

2.1 The sediment sample is acidified to pH 1 or less and extracted in a Soxhlet apparatus with an acetone-hexane solvent mixture.

2.2 The organic extract is base partitioned with a 2% potassium bicarbonate solution to separate the phenols and other acidic compounds from the neutral organic compounds.

2.3 The phenols, now in an aqueous medium, undergo acetylation with acetic anhydride while simultaneously being extracted into petroleum ether.

2.4 The organic extract is dried through anhydrous sodium sulphate and concentrated to less than 3 mL.

2.5 The concentrated sample is applied to a mini Silica Gel column and one fraction, containing all the phenol acetates, is collected for determination by GC-ECD and GC-MSD analyses.

2.6 The method presented here can readily be modified for the simultaneous analysis of other classes of compounds such as PCBs, chlorobenzenes, organochlorinated insecticides, and other neutral pesticides, if required.

3. INTERFERENCES

3.1 Organic compounds co-extracted from the sediment are potential interferences. The cleanup procedures described in this method will usually eliminate this source of interference.

3.2 All reagents must be thoroughly checked and any interferences from this source eliminated.

3.3 Because the acetylation reaction is specific to phenols, the potential interference from other acidic compounds is greatly reduced.

4. SAMPLING PROCEDURE AND STORAGE

4.1 Sediment samples should be collected and frozen immediately in an all-glass system or metal container.

4.2 Teflon-lined caps are recommended for the sample jars to prevent contamination of the sediment from contact with the cap. If Teflon lining is unavailable, the use of solvent-washed aluminum foil beneath the cap is acceptable.

4.3 Samples should be kept frozen, in the dark, and extracted as soon as possible.

5. SAMPLE PREPARATION

5.1 No special preparation is required.

6. APPARATUS

6.1 Capillary GC-ECD analyses of phenol acetates.

6.1.1 A gas chromatograph with good sensitivity equipped with a split/splitless capillary column injection port and an electron capture detector (^{63}Ni) such as Hewlett-Packard Model 5880A or equivalent.

6.1.2 Automatic Liquid Sampler such as Hewlett-Packard Model 7671A or equivalent. If this is not available, use a 10-μL Hamilton micro-syringe and inject 2 μL.

6.1.3 Fused Silica Capillary Column (FSCC): 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.

6.1.4 Chromatographic conditions:

Injection Port: splitless mode, splitless valve on for 30 s

Injection Port Temperature: 200°C

Detector: Ni-63 ECD

Detector Temperature: 300°C

Detector Make-Up Gas: Argon/methane (95+5) at 27 mL/min

Carrier Gas: Helium

Column Head Pressure: 12.5 psi

Column Temperature Initial: 70°C; hold for 0.5 min

Programming Rate 1: 10°C/min (70° to 120°C); hold at 120°C for 5.0 min

Programming Rate 2: 2°C/min (120° to 160°C); hold at 160°C for 1.0 min

Column Post-Run Final Temperature: 200°C for 10 min

6.2 Capillary GC-MSD analyes of phenol acetates.

6.2.1 A gas chromatograph equipped with a split/splitless capillary column injection port, an oven with multi-level temperature programming capability, and a mass selective detector with direct capillary interface such as the Hewlett-Packard 5880A and 5970B combination or equivalent.

6.2.2 A laboratory data handling system complete with floppy disc drive, hard disc drive, printer, monitor and existing GC-MSD software, such as Hewlett-Packard series 200 or 300 computer, is required.

6.2.3 Automatic Liquid Sampler as in 6.1.2. Inject 4 µL.

6.2.4 Fused Silica Capillary Column (FSCC): 30 m x 0.25 mm i.d. column, coated with a 5% diphenyl/94% dimethyl/1% vinyl polysiloxane bonded phase (0.25 µm thickness), such as Supelco SPB-5 (Part No. 2-4034) or equivalent.

6.2.5 Chromatographic conditions:

Injection Port: splitless mode, splitless valve on for 30 s

Injection Port Temperature: 250°C

GC-MSD Interface Temperature: 280°C

Detector: Electron Impact Quadrupole Mass Analyzer; 70 eV

Carrier Gas: Helium

Column Head Pressure: 4 psi

Column Initial Temperature: 70°C; hold for 0.5 min

Programming Rate 1: 30°C/min (70° to 120°C);

Programming Rate 2: 2.5°C/min (120° to 180°C); hold at 180°C for 10.0 min

Column Post-Run Final Temperature: 200°C for 10 min

6.2.6 Acquire GC-MSD data in the Selected Ion Monitoring (SIM) mode by monitoring the following characteristic ions:

Parent Phenol	Ion Group	Characteristic Ions (m/z)	
		Quantitation Ion	Confirmation Ions
Phenol	1	94	43, 136
2-chlorophenol	2	128	43, 170
3-chlorophenol	2	128	43, 170
4-chlorophenol	2	128	43, 170
2-chloro-5-methylphenol	3	142	43, 184
2,6-dichlorophenol	3	162	43, 204
4-chloro-3-methylphenol	3	142	43, 184
2,4-dichlorophenol	3	162	43, 204
3,5-dichlorophenol	3	162	43, 204
2,3-dichlorophenol	3	162	43, 204
3,4-dichlorophenol	3	162	43, 204
2,4,6-trichlorophenol	4	198	43, 240
2,3,6-trichlorophenol	4	198	43, 240
2,3,5-trichlorophenol	4	198	43, 240
2,4,5-trichlorophenol	4	198	43, 240
2,3,4-trichlorophenol	4	198	43, 240
3,4,5-trichlorophenol	4	198	43, 240
2,3,5,6-tetrachlorophenol	5	232	43, 274
2,3,4,6-tetrachlorophenol	5	232	43, 274
2,3,4,5-tetrachlorophenol	5	232	43, 274
Pentachlorophenol	6	266	43, 308

6.3 Soxhlet Apparatus

6.3.1 Electrothermal extraction apparatus with thermostatted heating mantles to accommodate 500-mL round-bottom flasks.

6.3.2 Allihn condensers (250 mm x 50 mm i.d.).

6.3.3 Soxhlet extraction tubes (200 mL capacity).

6.3.4 Glass extraction thimbles (130 mm x 45 mm i.d.) with coarse (40 - 60 µm) porosity fritted disc.

6.4 Oven, capable of maintaining 200 °C.

6.5 Magnetic stirrers with 38 mm x 13 mm o.d. Teflon-coated spin-bars.

6.6 Hamilton micro-syringes (500 µL, 100 µL, 10 µL).

6.7 Heating mantles with power regulators, to accommodate 500-mL round-bottom flasks.

6.8 Three-stage Snyder columns with a tapered ground-glass joint to fit the neck of the 500-mL round-bottom flasks.

6.9 Boiling chips, Soxhlet-cleaned for 24 hours with acetone/hexane (59+41).

6.10 Disposable Pasteur Pipettes (23 cm x 5 mm i.d.).

6.11 Silanized glass wool.

6.12 Volumetric flasks (250 mL) or Wheaton bottles (250 mL).

6.13 Volumetric flasks, "low-actinic" (100 mL).

6.14 Separatory funnels with Teflon stop-cocks (250 mL).

6.15 Coarse (70-100 µm) sintered-glass filter funnels (100 mm x 40 mm i.d.) with a tapered ground-glass joint and suction side-arm, available from Pegasus Industrial Specialties Ltd.

6.16 Round-bottom flasks (500 mL).

6.17 Volumetric pipettes (3 mL, 1 mL).

6.18 Graduated centrifuge tubes (15 mL) with ground-glass stoppers.

NOTE: All glassware must be thoroughly washed with a hot solution of laboratory detergent followed by rinses with hot tap water, 2-3 rinses of distilled water and a final acetone rinse to remove the water. The glassware should be dried at 130°C for at least two hours. Thorough rinsing with organic solvent immediately prior to using the glassware is recommended.

7. REAGENTS

7.1 All solvents must be Distilled-In-Glass, Pesticide Residue grade and must be checked before use for low blank values.

7.1.1 Acetone

7.1.2 Hexane

7.1.3 Petroleum Ether (30° - 60°C)

7.1.4 Toluene

7.1.5 Iso-octane

7.2 All chemicals must be of highest purity and should be washed with solvent and preheated where necessary.

7.2.1 Purified (organic-free) water. Pass distilled water through Millipore Super-Q unit (Millipore Corp.). Alternatively, extract 1 L distilled water three times by stirring with 50 mL dichloromethane for 30 minutes. Discard organic layers.

7.2.2 Dilute Sulphuric Acid, ACS grade or better. Prepare a (1+1) v/v solution with purified (organic-free) water.

7.2.3 2% Potassium Bicarbonate Solution. Dissolve 20 g anhydrous $KHCO_3$ in purified (organic-free) water and dilute to 1000 mL.

7.2.4 Acetic Anhydride. Distill AnalaR grade $(CH_3CO)_2O$ reagent, available from BDH chemicals, three times and collect the 138°-140°C fraction for acetylation reactions.

CAUTION: Acetic anhydride is corrosive and moisture-sensitive.

7.2.5 Sodium sulphate (anhydrous, Reagent grade), available from BDH Chemicals. Heat 18 hours at 650°C and store in a clean glass bottle in a dessicator.

7.2.6 5% Deactivated Silica Gel. Activate Silica Gel adsorbent (grade 950 for gas chromatography, 60/200 mesh, Fisher Scientific Co.) by heating for 18 hours at 130°C. Deactivate by adding 5 g purified (organic-free) water to 95 g activated Silica Gel. Mix well by tumbling for 18 hours in a tightly sealed glass container. Prepare fresh weekly.

7.2.7 Celite 545 (not Acid-washed), available from Fisher Scientific Company.

7.3 Analytical Standards. Phenols should be analytical grade (98+% purity). Obtain from manufacturers or U.S. Environmental Protection Agency and use without further purification.

7.3.1 Prepare 1000 ppm stock solutions of each individual phenol by dissolving 100 mg of pure analytical standard in toluene and diluting to 100.0 mL in "low-actinic" volumetric flasks. Store at 4°C in the dark.

7.3.2 Prepare a mixed Phenol Stock Solution by combining appropriate aliquots of the individual phenol stock solutions and diluting to 100.0 mL with acetone. Prepare Spiking Solutions by diluting further with acetone as required. Store all solutions at 4°C in the dark.

7.3.3 To prepare a GLC Standard for calibration, fortify a 150 mL sample of 2% potassium bicarbonate with 100 µL of an appropriate Spike Solution. Extract/acetylate alongside the sample extracts as per Steps 8.2 and 8.3 of the Procedure.

8. PROCEDURE

8.1 Sediment Extraction

8.1.1 Weigh a 50.0 g sample of homogeneous sediment into a 250-mL beaker or other suitable container. Simultaneously, weigh representative sample aliquots into tared containers and heat at 105°C to constant weight for moisture content determination.

8.1.2 Wet the sample in the beaker with purified (organic-free) water to an estimated 30% moisture content. Carefully, acidify the sediment by adding dilute sulphuric acid (1+1) in drops until the pH is 1 or less (use pH paper). Quantitatively transfer the sample to an extraction thimble containing 50 mm Celite 545.

8.1.3 Place the thimble in a Soxhlet extraction tube fitted with a 500-mL round-bottom flask containing 350 mL acetone/hexane (59+41) and a few boiling chips.

8.1.4 Adjust the temperature of the Soxhlet heating mantles to obtain a reflux rate of 6-8 cycles/hour.

8.1.5 After extracting for 20 hours, dismantle the Soxhlet apparatus and discard the sediment. When the solvent in the 500-mL round-bottom flask has cooled, add 50 mL of potassium bicarbonate solution (2%).

8.1.6 Attach a three-stage Snyder column to the flask and clamp securely in a heating mantle. Wet the column with 5 mL of hexane and reduce the volume of the extract to approximately 100 mL.

CAUTION: All Snyder column evaporation steps must be performed in a Fume Hood.

8.1.7 Transfer the concentrated sample to a 250-mL separatory funnel. Rinse the Snyder column and the flask with 30 and 20 mL aliquots of hexane and add the rinsings to the sample extract in the separatory funnel.

8.1.8 Add 40 mL of potassium bicarbonate (2%) to the same separatory funnel and shake vigorously for 2 min. Vent often to release the gas. Ensure the pH of the aqueous layer is 8 or greater. (Use pH paper.)

8.1.9 Allow the layers to separate and drain the aqueous (lower) layer to a 250-mL volumetric flask or other suitable container.

8.1.10 Repeat the aqueous partitioning in Steps 8.1.8 and 8.1.9 with two 30-mL portions of potassium bicarbonate (2%), transferring the aqueous layer each time to the same 250-mL flask. After the final back-extraction, save the organic layer for analysis of other classes of neutral organic compounds, if necessary.

8.2 Acetylation of Phenols

8.2.1 To the 150-mL sample of combined potassium bicarbonate extracts in the 250-mL volumetric flask, add 3 mL acetic anhydride and 25 mL petroleum ether (30°-60°C). Tightly cover the flask with an aluminum-lined or Teflon-lined cap. Stir the sample on a magnetic stirrer using a Teflon-coated stirring bar so that the vortex formed at the surface almost reaches the bottom of the flask.

8.2.2 After stirring for 30 min, transfer the contents of the flask to a clean 250-mL separatory funnel and allow the layers to separate.

8.2.3 Drain the aqueous (lower) layer into the original 250-mL flask.

8.2.4 Pass the organic layer through a (vacuum) sintered-glass filter funnel containing 50 mm of anhydrous sodium sulphate. Collect the dried extract in a clean 500-mL round-bottom flask.

8.2.5 Repeat the extractive acetylation procedure twice, adding fresh aliquots of 3 mL acetic anhydride and 25 mL petroleum ether to the aqueous layer in the 250-mL flask each time. Discard the aqueous sample after the third extraction.

8.2.6 Rinse the 250-mL separatory funnel twice with 10 mL petroleum ether and drain the rinsings through the sodium sulphate column into the 500-mL round-bottom flask.

8.2.7 Wash the sodium sulphate column with another 25 mL petroleum ether and apply a vacuum until the sodium sulphate is dry. Remove the column and add 2 mL iso-octane to the extract as a keeper.

8.2.8 Add a few boiling chips to the extract and attach a three-stage Snyder column to the 500-mL round-bottom flask. Wet the Snyder column with 5 mL hexane and clamp securely in a heating mantle.

8.2.9 Gently concentrate the sample to 2-3 mL. This extract contains the phenol acetates and is ready for Silica Gel cleanup and analysis by GLC.

8.3 Silica Gel Column Cleanup

8.3.1 Prepare micro-columns by plugging clean disposable Pasteur pipettes (23 cm x 5 mm i.d.) with a clean piece of silanized glass wool.

8.3.2 Fill the columns with 5 cm of Silica Gel (5% deactivated) and tap them gently with a pencil to uniformly settle the solid. Add 0.5 cm anhydrous sodium sulphate to the top of the column.

8.3.3 Prewet the columns with 5 mL hexane and permit the hexane to drain just to the top of the sodium sulphate layer. Discard hexane eluant.

8.3.4 With a disposable pipette, apply the concentrated sample extract (from Step 8.2.9) to the column. Rinse the 500-mL round-bottom flask 1 mL at a time with hexane. Apply the rinses to the column, never permitting the solution to drain below the sodium sulphate layer.

8.3.5 Collect a total of 5 mL hexane in a centrifuge tube. Discard this Fraction A, containing side-products and contaminants.

8.3.6 Elute with 10 mL toluene into a clean 15-mL centrifuge tube. Adjust the volume to precisely 10.00 mL. This Fraction B contains the chlorophenol acetates of interest.

8.3.7 For ultra-trace level work, where further concentration of the extract before GLC analysis is required, use an acetone/hexane (5+95) solvent mixture to elute Fraction B from the Silica Gel column. Add 1 or 2 mL iso-octane to the 10 mL eluant and concentrate under a gentle stream of nitrogen to 1.00 or 2.00 mL as required.

8.3.8 Analyze the sample extract by means of gas chromatography using electron capture and/or mass selective detection.

8.3.9 For calibration standards acetylate a known amount of phenols as per Step 7.3.3.

9. CALCULATIONS

9.1 The concentration of each phenol 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:

$$X_{sam} = \left(\frac{H_{sam}}{H_{std}}\right) x \left(\frac{V_{inj\ std}}{V_{inj\ sam}}\right) x \left(X_{std}\right) x \left(\frac{V_{ext}}{W_{sam}}\right)$$

where X_{sam} = phenol concentration in original sediment sample (ng/g);

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} = phenol concentration in standard solution (pg/μL);

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

W_{sam} = weight of orginal sediment sample extracted (g).

9.2 The determination of moisture content in the sediment samples is as follows:

$$\%\ \text{moisture} = \left(\frac{A - B}{A}\right) x\ 100$$

where A = weight in grams of a homogeneous sediment subsample before drying, and

B = weight in grams of the same sample after being dried to constant weight at 105°C.

10. PRECISION AND ACCURACY

10.1 Data indicating the single-operator precision and accuracy are summarized in the following table:

Parent Phenol	Mean Recovery (%)	Relative Standard Deviation (%)
2-chloro-	74	10.1
3-chloro-	86	3.1
4-chloro-	65	11.6
2-chloro-5-methyl-	51	12.3
2,6-dichloro-	76	13.6
4-chloro-3-methyl-	50	14.5
2,4-dichloro-	88	6.8
3,5-dichloro-	83	4.0
2,3-dichloro-	87	4.4
3,4-dichloro-	85	5.5
2,4,6-trichloro-	88	5.2
2,3,6-trichloro-	83	5.6
2,3,5-trichloro-	91	3.0
2,4,5-trichloro-	87	2.5
2,3,4-trichloro-	93	3.6
3,4,5-trichloro-	91	7.3
2,3,5,6-tetrachloro-	90	4.6
2,3,4,6-tetrachloro-	94	8.8
2,3,4,5-tetrachloro-	95	4.7
Pentachlorophenol	96	4.3

NOTE: (a) Validation data were obtained from fortified 50-g subsamples of a Lake Superior/Battle River blended sediment having approximately 40% moisture content.

(b) No. of replicates = 6.

(c) Fortification level for each phenol = 1.0 µg/kg.

(d) For the above data, the Silica Gel Fraction B eluant used was acetone/hexane (5+95) and was concentrated five-fold under N_2 before GC-MSD analysis.

11. CONFIRMATION OF IDENTITY

11.1 The identity of each GC peak in the mixture may be assigned by comparison with the retention time of each chlorophenol acetate obtained by reacting individual phenols under identical experimental conditions.

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

11.3 Additional confirmation of identity may also be obtained by the formation and analysi of different derivatives.

11.4 Whenever sample concentration permits, confirmation of identity may readily be obtained by combined GC-MS (EI and/or CI) operating at selected ion monitoring (SIM) or full scan mode. The characteristic ions of the chlorophenol acetates, acquired in the SIM mode of the GC-MSD, are listed in Section 6.2.6.

12. REMARKS

12.1 It is recommended that a phenol standard be prepared alongside each set of sediment samples to account for any minor variations in the extractive acetylation or column cleanup steps from one set to another.

12.2 Extreme care must be exercised by the analyst in the steps in which extracts are concentrated. Samples must never be allowed to go dry as the lesser chlorinated phenols are quite volatile.

12.3 All concentration steps using a Snyder column for evaporation of the solvent must be carried out in a Fume Hood.

12.4 For complete recovery of the phenols during the base-partitioning steps, it is extremely important that the aqueous phase have a pH of 8 or greater. (Steps 8.1.8 to 8.1.10.). It may be necessary to increase the strength or quantity of the potassium bicarbonate solution in order to neutralize the extracted acid in the sample extracts.

12.5 Special attention should be taken in the handling of acetic anhydride because it is corrosive and moisture-sensitive.

12.6 Sediment extracts may be cleaned up on a micro-Silica Gel column using either toluene or a 5% acetone in hexane solvent mixture. For elution of Fraction B, containing all the chlorophenol acetates, toluene was the preferred eluant because it yielded a cleaner extract. However, for extracts with very low levels of phenol acetates, the acetone/hexane blend was used because of the greater ease in further concentration (under N_2).

12.7 Because of the poor sensitivity of the ECD toward the monochlorophenol acetates, these compounds were analyzed by GC-MSD.

12.8 On both the 12 m OV-1 and 30m SPB-5 FSCCs, the 2,5-dichlorophenol acetate and 2,4-dichlorophenol acetate were an unresolved pair. Therefore, 2,5-dichlorophenol was omitted in the recovery studies because it does not often appear in environmental samples, nor is it a U.S. Environmental

Protection Agency priority pollutant. However, these two dichlorophenols can be simultaneously analyzed by the formation of their respective PFB ether derivatives. (Ref. 13.4).

12.9 Phenol showed very erratic results in its GC analyses and was therefore omitted from further recovery studies.

12.10 The recoveries of 2-chloro-5-methylphenol and 4-chloro-3-methylphenol were fairly consistent at 45±5% over a 100-fold concentration range.

12.11 To reduce tailing of the phenol acetates on the OV-1 FSCC, it is necessary, after about 100 injections, to replace the splitless insert and to cut off approximately 10-20 cm of the injection port end of the column.

12.12 The determination of other classes of compounds such as PCBs, chlorobenzenes, organochlorinated insecticides and other neutral pesticides can be included by analyzing the organic layer from the base-partitioning step in Procedure Step 8.1.10.

13. **REFERENCES**

13.1 Lee, Hing-Biu, Li-Da Weng and Alfred S.Y. Chau. 1984. Chemical Derivatization Analysis of Pesticide Residues. VIII. Analysis of 15 Chlorophenols in Natural Water by *in situ* Acetylation. J. Assoc. Off. Anal. Chem. 67[4]: 789-794.

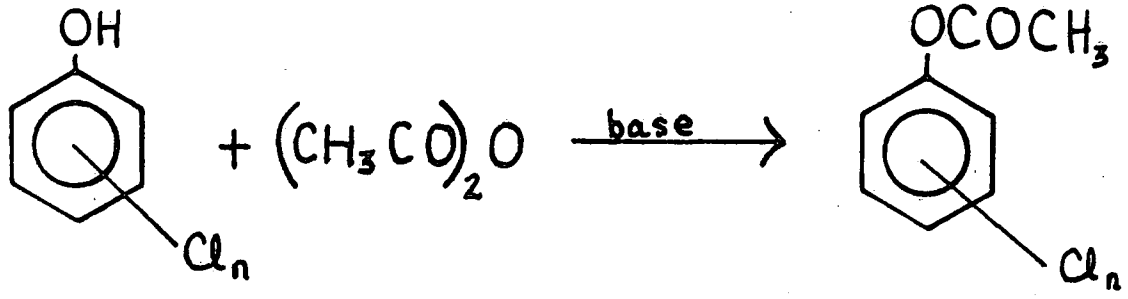
13.2 Wegman, R.C.C. and H.H. van den Broek. 1983. Chlorophenols in River Sediment in the Netherlands. Water Res. 17: 227-230.

13.3 Xie, Tian-Min. 1983. Determination of Trace Amounts of Chlorophenols and Chloroguaiacols in Sediment. Chemosphere 12: 1183-1191.

13.4 Lee, Hing-Biu, Li-Da Weng and Alfred S.Y. Chau. 1984. Chemical Derivitization Analysis of Pesticide Residues. IX. Analysis of Phenol and 21 Chlorinated Phenols in Natural Waters by Formation of Pentafluorobenzyl Ether Derivatives. J. Assoc. Off. Anal. Chem. 67[6]: 1086-1091.

APPENDIX

1. Figure A-1. Acetylation of Chlorophenols.



2. Figure A-2. GC-MSD chromatogram of acetate derivatives of 21 phenols as resolved on a 30 m x 0.25 mm i.d. SPB-5 fused silica capillary column. GLC conditions are outlined in Section 6.2.

	Parent Phenol	Retention Time (min)
1.	phenol	4.50
2.	2-chlorophenol	6.41
3.	3-chlorophenol	6.86
4.	4-chlorophenol	6.98
5.	2-chloro-5-methylphenol	8.56
6.	2,6-dichlorophenol	9.10
7.	4-chloro-3-methylphenol	9.33
8.	2,4-dichlorophenol	9.62
9.	3,5-dichlorophenol	9.95
10.	2,3-dichlorophenol	10.50
11.	3,4-dichlorophenol	11.24
12.	2,4,6-trichlorophenol	12.44
13.	2,3,6-trichlorophenol	13.93
14.	2,3,5-trichlorophenol	14.20
15.	2,4,5-trichlorophenol	14.40
16.	2,3,4-trichlorophenol	15.97
17.	3,4,5-trichlorophenol	16.48
18.	2,3,5,6-tetrachlorophenol	18.91
19.	2,3,4,6-tetrachlorophenol	19.08
20.	2,3,4,5-tetrachlorophenol	21.39
21.	Pentachlorophenol	26.17

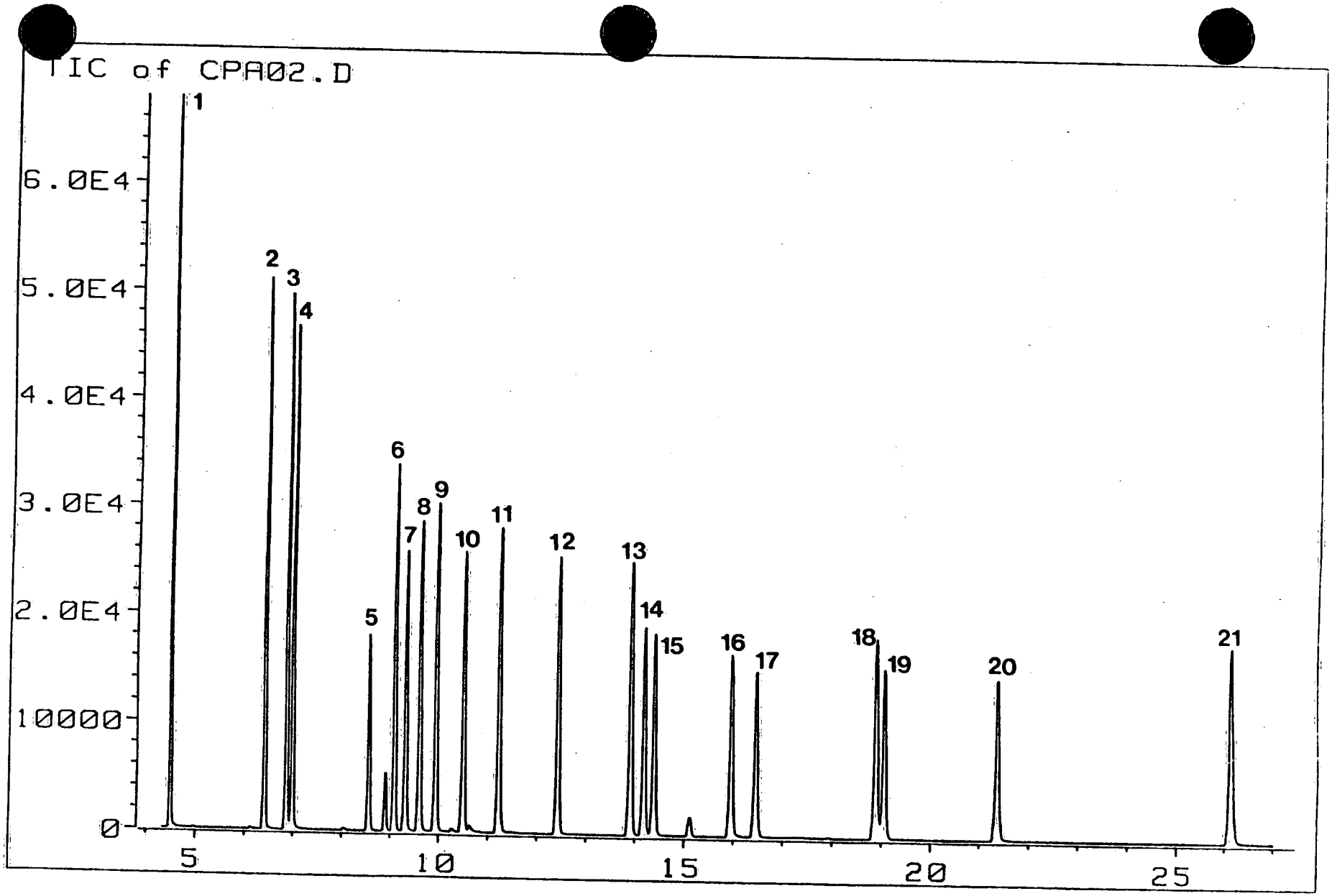


Figure A-2 GC-MSD Chromatogram of Acetate Derivatives of 21 Phenols