METHOD 8040A

PHENOLS BY GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 Method 8040 is used to determine the concentration of various phenolic compounds. The following compounds can be determined by this method:

		Appropriate Technique				
Compound Name	CAS No.[a]	3510	3520	3540	3550	3580
2-sec-Butyl-4,6-dinitrophenol (DNBP, Dinoseb)	88-85-7	X	ND	ND	ND	X
4-Chloro-3-methylphenol	59-50-7	X	X	X	X	X
2-Chlorophenol	95-57-8	X	X	X	X	X
Cresols (methyl phenols)	1319-77-3	X	ND	ND	ND	X
2-Cyclohexyl-4,6-dinitropheno	1131-89-5	X	ND	ND	ND	LR
2,4-Dichlorophenol	120-83-2	X	X	X	X	X
2,6-Dichlorophenol	87-65-0	X	ND	ND	ND	X
2,4-Dimethylphenol	105-67-9	X	X	X	X	X
2,4-Dinitrophenol	51-28-5	X	X	X	X	X
2-Methyl-4,6-dinitrophenol	534-52-1	X	X	X	X	X
2-Nitrophenol	88-75-5	X	X	X	X	X
4-Nitrophenol	100-02-7	X	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X	X
Phenol	108-95-2	DC(28)	X	X	X	X
Tetrachlorophenols	25167-83-3	X	ND	ND	ND	X
Trichlorophenols	25167-82-2	X	X	X	X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X	X

a Chemical Abstract Services Registry Number.
DC = Unfavorable distribution coefficient (number in parenthesis is percent recovery).
LR = Low response.
ND = Not determined.
X = Greater than 70 percent recovery by this technique.

1.2 Table 1 lists the method detection limit for the target analytes in water. Table 2 lists the estimated quantitation limit (EQL) for all matrices.

2.0 SUMMARY OF METHOD

2.1 Method 8040 provides gas chromatographic conditions for the detection of phenolic compounds. Prior to analysis, samples must be extracted using

appropriate techniques (see Chapter Two for guidance). Both neat and diluted organic liquids (Method 3580, Waste Dilution) may be analyzed by direct injection. A 2 to 5 µL sample is injected into a gas chromatograph using the solvent flush technique, and compounds in the GC effluent are detected by a flame ionization detector (FID).

2.2 Method 8040 also provides for the preparation of pentafluorobenzyl-bromide (PFB) derivatives, with additional cleanup procedures for electron capture gas chromatography. This is to lower the detection limits of some phenols and to aid the analyst in the elimination of interferences.

3.0 INTERFERENCES

3.1 Refer to Methods 3500, 3600, and 8000.

3.2 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by analyzing reagent blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.3 Interferences coextracted from samples will vary considerably from source to source, depending upon the waste being sampled. Although general cleanup techniques are recommended as part of this method, unique samples may require additional cleanup.

3.4 The decomposition of some analytes under basic extraction conditions has been demonstrated. Specifically, phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510.

3.5 The flame ionization detector (FID) is very susceptible to false positives caused by the presence of hydrocarbons commonly found in samples from waste sites. The problem may be minimized by applying acid-base cleanup (Method 3650) and/or alumina column chromatography (Method 3611) prior to GC/FID analysis or using the derivatization technique and analyzing by GC/electron capture detector. Initial site investigation should always be performed utilizing GC/MS analysis to characterize the site and determine the feasibility of utilizing Method 8040 with a GC/FID.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 Gas Chromatograph - Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak areas and/or peak heights is recommended.

4.1.2 Columns

4.1.2.1 Column for underivatized phenols - 1.8 m x 2.0 mm ID glass column packed with 1% SP-1240DA on Supelcoport 80/100 mesh, or equivalent.

4.1.2.2 Column for derivatized phenols - 1.8 m x 2 mm ID glass column packed with 5% OV-17 on Chromosorb W-AW-DMCS 80/100 mesh, or equivalent.

4.1.3 Detectors - Flame ionization (FID) and electron capture (ECD).

4.2 Reaction vial - 20 mL, with Teflon lined screw-cap or crimp top.

4.3 Volumetric flask, Class A - Appropriate sizes with ground-glass stoppers.

4.4 Kuderna-Danish (K-D) apparatus

4.4.1 Concentrator tube - 10 mL, graduated (Kontes K-570050-1025 or equivalent). Ground-glass stopper is used to prevent evaporation of extracts.

4.4.2 Evaporation flask - 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps or equivalent.

4.4.3 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

4.4.4 Snyder column - Two ball micro (Kontes K-569001-0219 or equivalent).

4.4.5 Springs - 1/2 inch (Kontes K-662750 or equivalent).

4.5 Boiling chips - Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control ($\pm$ 5°C). The bath should be used in a hood.

4.7 Microsyringe - 10 µL.

4.8 Syringe - 5 mL.

4.9 Balance - analytical, 0.0001 g.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the 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.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Hexane, $CH_3(CH_2)_4CH_3$ - Pesticide quality or equivalent.

5.4 2-Propanol, $(CH_3)_2CHOH$ - Pesticide quality or equivalent.

5.5 Toluene, $C_6H_5CH_3$ - Pesticide quality or equivalent.

5.6 Derivatization reagent - Add 1 mL pentafluorobenzyl bromide and 1 g 18-crown-6-ether to a 50 mL volumetric flask and dilute to volume with 2-propanol. Prepare fresh weekly. This operation should be carried out in a hood. Store at 4°C and protect from light.

5.6.1 Pentafluorobenzyl bromide (alpha-Bromopentafluorotoluene), $C_6F_5CH_2Br$. 97% minimum purity.

NOTE: This chemical is a lachrymator.

5.6.2 18-crown-6-ether (1,4,7,10,13,16-Hexaoxacyclooctadecane) - 98% minimum purity.

NOTE: This chemical is highly toxic.

5.7 Potassium carbonate (Powdered), K_2CO_3.

5.8 Stock standard solutions

5.8.1 Prepare stock standard solution at a concentration of 1000 mg/L by dissolving 0.0100 g of assayed reference material in 2-propanol and diluting to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

5.8.2 Transfer the stock standard solutions into bottles with Teflon lined screw-caps or crimp tops. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.8.3 Stock standard solutions must be replaced after one year, or sooner if comparison with check standards indicates a problem.

5.9 Calibration standards - Prepare calibration standards at a minimum of five concentrations through dilution of the stock standards with 2-propanol. One of the concentrations should be at a concentration near, but above, the method detection limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

5.10 Internal standards (if internal standard calibration is used) - To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

5.10.1 Prepare calibration standards at a minimum of five concentrations for each analyte as described in Section 5.9.

5.10.2 To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with 2-propanol.

5.10.3 Analyze each calibration standard according to Section 7.0.

5.11 Surrogate standards - The analyst should monitor the performance of the extraction, cleanup (if necessary), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and organic-free reagent water blank with phenolic surrogates (e.g. 2-fluorophenol and 2,4,6-tribromophenol) recommended to encompass the range of the temperature program used in this method. Method 3500 details instructions on the preparation of acid surrogates. Deuterated analogs of analytes should not be used as surrogates for gas chromatographic analysis due to coelution problems.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1. Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction

7.1.1 Refer to Chapter Two for guidance on choosing the appropriate extraction procedure. In general, water samples are extracted at a pH of less than or equal to 2 with methylene chloride, using either Method 3510 or 3520. Solid samples are extracted using either Method 3540 or 3550, and non-aqueous samples using Method 3580. Extracts obtained from application of either Method 3540 or 3550 should undergo Acid-Base Partition Cleanup, using Method 3650.

7.1.2 Prior to gas chromatographic analysis, the extraction solvent must be exchanged to 2-propanol. The exchange is performed as follows:

7.1.2.1 Following concentration of the extract to 1 mL using the macro-Snyder column, allow the apparatus to cool and drain for at least 10 minutes.

7.1.2.2 Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of 2-

propanol. Adjust the extract volume to 1.0 mL. Stopper the concentrator tube and store refrigerated at 4°C if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a vial with a Teflon lined screw-cap or crimp top. If the extract requires no further derivatization or cleanup, proceed with gas chromatographic analysis.

7.2 Gas chromatographic conditions (Recommended)

7.2.1 Column for underivatized phenols -

Carrier gas (N_2) flow rate:	30 mL/min
Initial temperature:	80°C
Temperature program:	80°C to 150°C at 8°C/min
Final Temperature:	150°C, hold until all compounds have eluted.

7.2.2 Column for derivatized phenols -

Carrier gas (5% methane/95% argon) flow rate:	30 mL/min
Initial temperature:	200°C
Temperature program:	isothermal, hold until all compounds have eluted.

7.3 Calibration - Refer to Method 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.3.1 The procedure for internal or external calibration may be used for the underivatized phenols. Refer to Method 8000 for a description of each of these procedures. If derivatization of the phenols is required, the method of external calibration should be used by injecting five or more concentrations of calibration standards that have also undergone derivatization and cleanup prior to instrument calibration.

7.4 Gas chromatographic analysis

7.4.1 Refer to Method 8000. If the internal standard calibration technique is used, add 10 µL of internal standard to the sample prior to injection.

7.4.2 Phenols are to be determined on a gas chromatograph equipped with a flame ionization detector according to the conditions listed for the 1% SP-1240DA column (Section 7.2.1). Table 1 summarizes estimated retention times and sensitivities that should be achieved by this method for clean water samples. Estimated quantitation limits for other matrices are list in Table 2.

7.4.3 Method 8000 provides instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-concentration standard after each group of 10 samples in the analysis sequence.

7.4.4 An example of a GC/FID chromatogram for certain phenols is shown in Figure 1. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

7.4.5 Record the sample volume injected and the resulting peak sizes (in area units or peak heights).

7.4.6 Using either the internal or external calibration procedure (Method 8000), determine the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes. See Method 8000 for calculation equations.

7.4.7 If peak detection using the SP-1240DA column with the flame ionization detector is prevented by interferences, PFB derivatives of the phenols should be analyzed on a gas chromatograph equipped with an electron capture detector according to the conditions listed for the 5% OV-17 column (Section 7.2.2). The derivatization and cleanup procedure is outlined in Sections 7.5 through 7.6. Table 3 summarizes estimated retention times for derivatives of some phenols using the conditions of this method.

7.4.8 Figure 2 shows a GC/ECD chromatogram of PFB derivatives of certain phenols.

7.4.9 Record the sample volume injected and the resulting peak sizes (in area units or peak heights).

7.4.10 Determine the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes. The method of external calibration should be used (see Method 8000 for guidance). The concentration of the individual compounds in the sample is calculated as follows:

$$\text{Concentration } (\mu g/L) = \frac{[(A)(V_t)(B)(D)]}{[(V_i)(X)(C)(E)]}$$

where:

A = Mass of underivatized phenol represented by area of peak in sample chromatogram, determined from calibration curve (see Method 8000), ng.

V_t = Total amount of column eluate or combined fractions from which V_i was taken, µL.

B = Total volume of hexane added in Section 7.5.5, mL.

D = Total volume of 2-propanol extract prior to derivatization, mL.

V_i = Volume injected, µL.

X = Volume of water extracted, mL, or weight of nonaqueous sample extracted, g, from Section 7.1. Either the dry or wet weight of the nonaqueous sample may be used, depending upon the specific application of the data.

C = Volume of hexane sample solution added to cleanup column (Method 3630), mL.

E = Volume of 2-propanol extract carried through derivatization in Section 7.5.1, mL.

7.5 Derivatization - If interferences prevent measurement of peak area during analysis of the extract by flame ionization gas chromatography, the phenols must be derivatized and analyzed by electron capture gas chromatography.

7.5.1 Pipet a 1.0 mL aliquot of the 2-propanol stock standard solution or of the sample extract into a glass reaction vial. Add 1.0 mL derivatization reagent (Section 5.3). This amount of reagent is sufficient to derivatize a solution whose total phenolic content does not exceed 300 mg/L.

7.5.2 Add approximately 0.003 g of potassium carbonate to the solution and shake gently.

7.5.3 Cap the mixture and heat it for 4 hours at 80°C in a hot water bath.

7.5.4 Remove the solution from the hot water bath and allow it to cool.

7.5.5 Add 10 mL hexane to the reaction vial and shake vigorously for 1 minute. Add 3.0 mL organic-free reagent water to the reaction vial and shake for 2 minutes.

7.5.6 Decant the organic layer into a concentrator tube and cap with a glass stopper. Proceed with cleanup procedure.

7.6 Cleanup

7.6.1 Cleanup of the derivatized extracts takes place using Method 3630 (Silica Gel Cleanup), in which specific instructions for cleanup of the derivatized phenols appear.

7.6.2 Following column cleanup, analyze the samples using GC/ECD, as described starting in Section 7.4.7.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method used. If extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

8.2 Procedures to check the GC system operation are found in Method 8000, Section 8.6.

8.2.1 The quality control check sample concentrate (Method 8000, Section 8.6) should contain each analyte of interest at a concentration of 100 mg/L in 2-propanol.

8.2.2 Table 4 indicates the calibration and QC acceptance criteria for this method. Table 5 gives method accuracy and precision as functions of concentration for the analytes. The contents of both tables should be used to evaluate a laboratory's ability to perform and generate acceptable data by this method.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in Method 8000, Section 8.10).

8.3.1 If recovery is not within limits, the following is required.

- Check to be sure that there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

9.0 METHOD PERFORMANCE

9.1 The method was tested by 20 laboratories using organic-free reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 12 to 450 µg/L. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships for a flame ionization detector are presented in Table 5.

9.2 The accuracy and precision obtained will be affected by the sample matrix, sample-preparation technique, and calibration procedures used.

10.0 REFERENCES

1. Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters. Category 3 - Chlorinated Hydrocarbons and Category 8 - Phenols. Report for EPA Contract 68-03-2625 (in preparation).

2. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

3. "Determination of Phenols in Industrial and Municipal Wastewaters," Report for EPA Contract 68-03-2625 (in preparation).

4. "EPA Method Validation Study Test Method 604 (Phenols)," Report for EPA Contract 68-03-2625 (in preparation).

5. Kawahara, F.K. "Microdetermination of Derivatives of Phenols and Mercaptans by Means of Electron Capture Gas Chromatography," Analytical Chemistry, 40, 1009, 1968.

6. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," Journal of the Association of Official Analytical Chemists, 48, 1037, 1965.

TABLE 1.
FLAME IONIZATION GAS CHROMATOGRAPHY OF PHENOLS[a]

Analyte	Retention time (minutes)	Method Detection limit (µg/L)
2-sec-Butyl-4,6-dinitrophenol (DNBP)		
4-Chloro-3-methylphenol	7.50	0.36
2-Chlorophenol	1.70	0.31
Cresols (methyl phenols)		
2-Cyclohexyl-4,6-dinitrophenol		
2,4-Dichlorophenol	4.30	0.39
2,6-Dichlorophenol		
2,4-Dimethylphenol	4.03	0.32
2,4-Dinitrophenol	10.00	13.0
2-Methyl-4,6-dinitrophenol	10.24	16.0
2-Nitrophenol	2.00	0.45
4-Nitrophenol	24.25	2.8
Pentachlorophenol	12.42	7.4
Phenol	3.01	0.14
Tetrachlorophenols		
Trichlorophenols		
2,4,6-Trichlorophenol	6.05	0.64

[a] - 1% SP-1240DA on Supelcoport 80/100 mesh column.

TABLE 2.
DETERMINATION OF ESTIMATED QUANTITATION LIMITS (EQLs) FOR VARIOUS MATRICES[a]

Matrix	Factor[b]
Ground water	10
Low-concentration soil by sonication with GPC cleanup	670
High-concentration soil and sludges by sonication	10,000
Non-water miscible waste	100,000

a Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable.

b EQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 3.
ELECTRON CAPTURE GAS CHROMATOGRAPHY OF PFB DERIVATIVES[a]

Parent compound	Retention time (min)	Method detection limit (µg/L)
4-Chloro-2-methylphenol	4.8	1.8
2-Chlorophenol	3.3	0.58
2,4-Dichlorophenol	5.8	0.68
2,4-Dimethylphenol	2.9	0.63
2,4-Dinitrophenol	46.9	
2-Methyl-4,6-dinitrophenol	36.6	
2-Nitrophenol	9.1	0.77
4-Nitrophenol	14.0	0.70
Pentachlorophenol	28.8	0.59
Phenol	1.8	2.2
2,4,6-Trichlorophenol	7.0	0.58

[a] - 5% OV-17 on Chromosorb W-AW-DMCS 80/100 mesh column.

TABLE 4.
QC ACCEPTANCE CRITERIA[a]

Analyte	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Recovery Range (%)
4-Chloro-3-methylphenol	100	16.6	56.7-113.4	99-122
2-Chlorophenol	100	27.0	54.1-110.2	38-126
2,4-Dichlorophenol	100	25.1	59.7-103.3	44-119
2,4-Dimethylphenol	100	33.3	50.4-100.0	24-118
4,6-Dinitro-2-methylphenol	100	25.0	42.4-123.6	30-136
2,4-Dinitrophenol	100	36.0	31.7-125.1	12-145
2-Nitrophenol	100	22.5	56.6-103.8	43-117
4-Nitrophenol	100	19.0	22.7-100.0	13-110
Pentachlorophenol	100	32.4	56.7-113.5	36-134
Phenol	100	14.1	32.4-100.0	23-108
2,4,6-Trichlorophenol	100	16.6	60.8-110.4	53-119

s = Standard deviation of four recovery measurements, in µg/L.

$\bar{x}$ = Average recovery for four recovery measurements, in µg/L.

a Criteria from 40 CFR Part 136 for Method 604. These criteria are based directly upon the method performance data in Table 5. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 5.

TABLE 5.
METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Analyte	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
4-Chloro-3-methylphenol	0.87C-1.97	$0.11\bar{x}-0.21$	$0.16\bar{x}+1.41$
2-Chlorophenol	0.83C-0.84	$0.18\bar{x}+0.20$	$0.21\bar{x}+0.75$
2,4-Dichlorophenol	0.81C+0.48	$0.17\bar{x}-0.02$	$0.18\bar{x}+0.62$
2,4-Dimethylphenol	0.62C-1.64	$0.30\bar{x}-0.89$	$0.25\bar{x}+0.48$
4,6-Dinitro-2-methylphenol	0.84C-1.01	$0.15\bar{x}+1.25$	$0.19\bar{x}+5.85$
2,4-Dinitrophenol	0.80C-1.58	$0.27\bar{x}-1.15$	$0.29\bar{x}+4.51$
2-Nitrophenol	0.81C-0.76	$0.15\bar{x}+0.44$	$0.14\bar{x}+3.84$
4-Nitrophenol	0.46C+0.18	$0.17\bar{x}+2.43$	$0.19\bar{x}+4.79$
Pentachlorophenol	0.83C+2.07	$0.22\bar{x}-0.58$	$0.23\bar{x}+0.57$
Phenol	0.43C+0.11	$0.20\bar{x}-0.88$	$0.17\bar{x}+0.77$
2,4,6-Trichlorophenol	0.86C-0.40	0.10x+0.53	0.13x+2.40

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of x, in µg/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in µg/L.

C = True value for the concentration, in µg/L.

$\bar{x}$ = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

[a]From 40 CFR Part 136 for Method 604.

Figure 1
Gas Chromatogram of Phenols

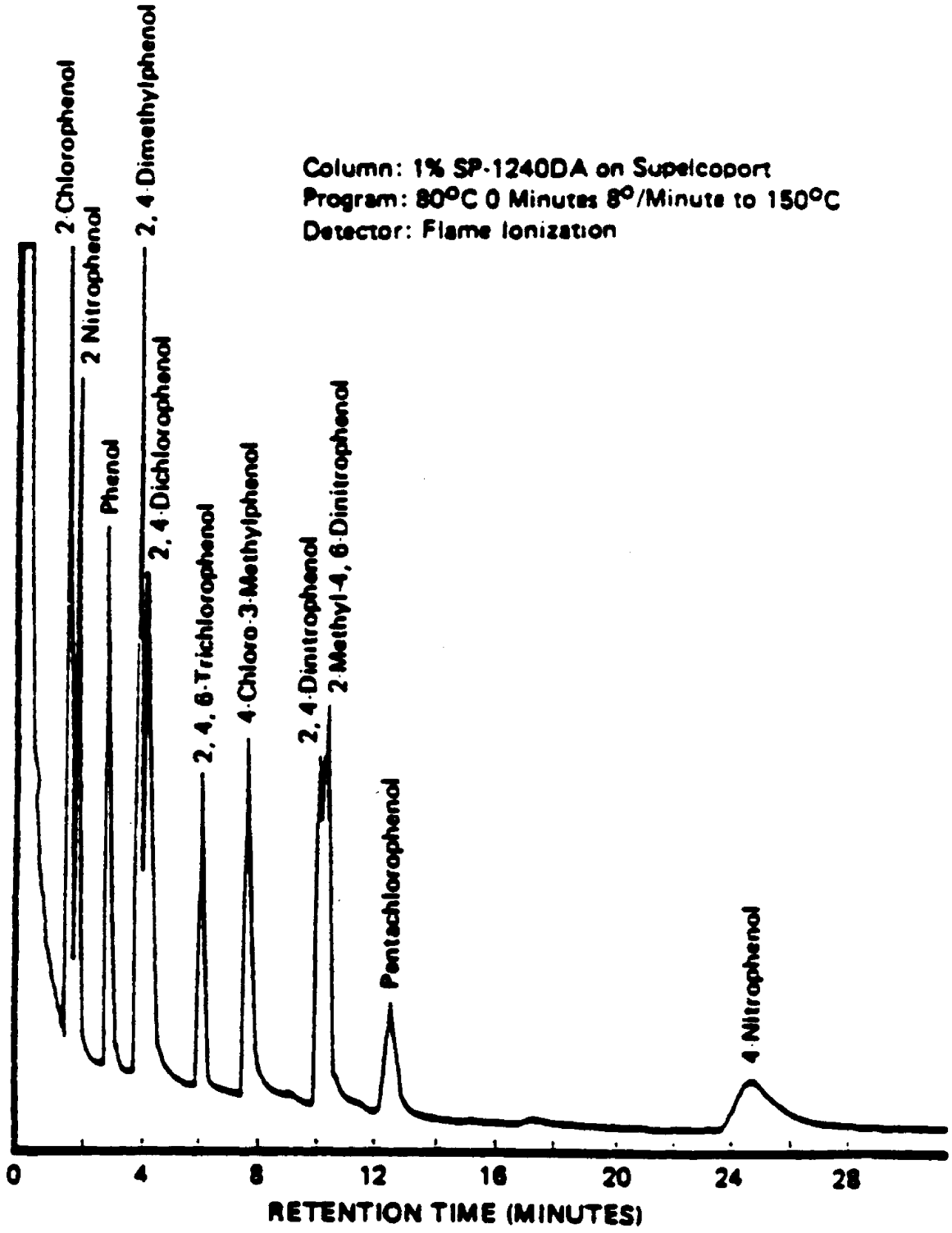
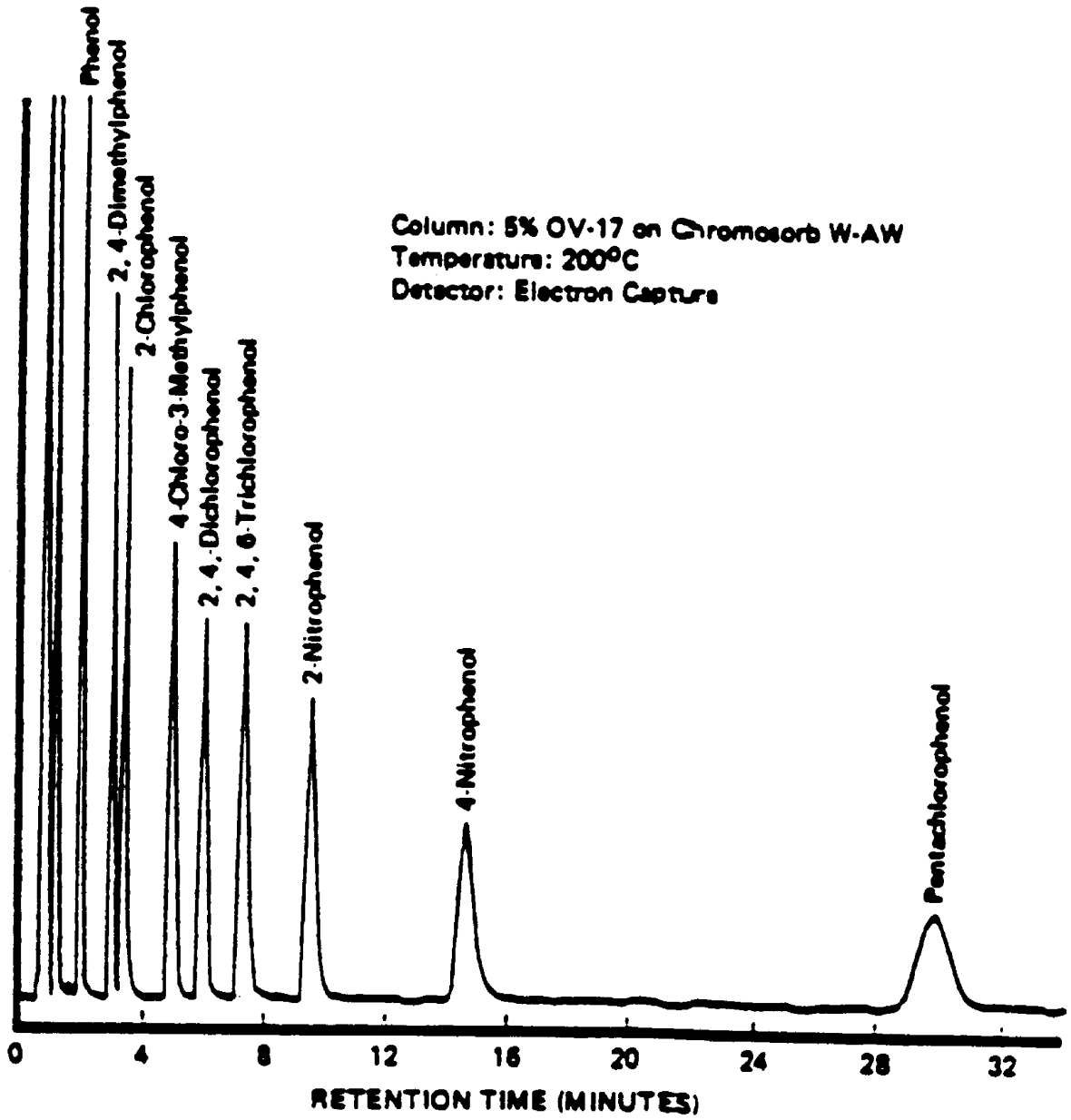
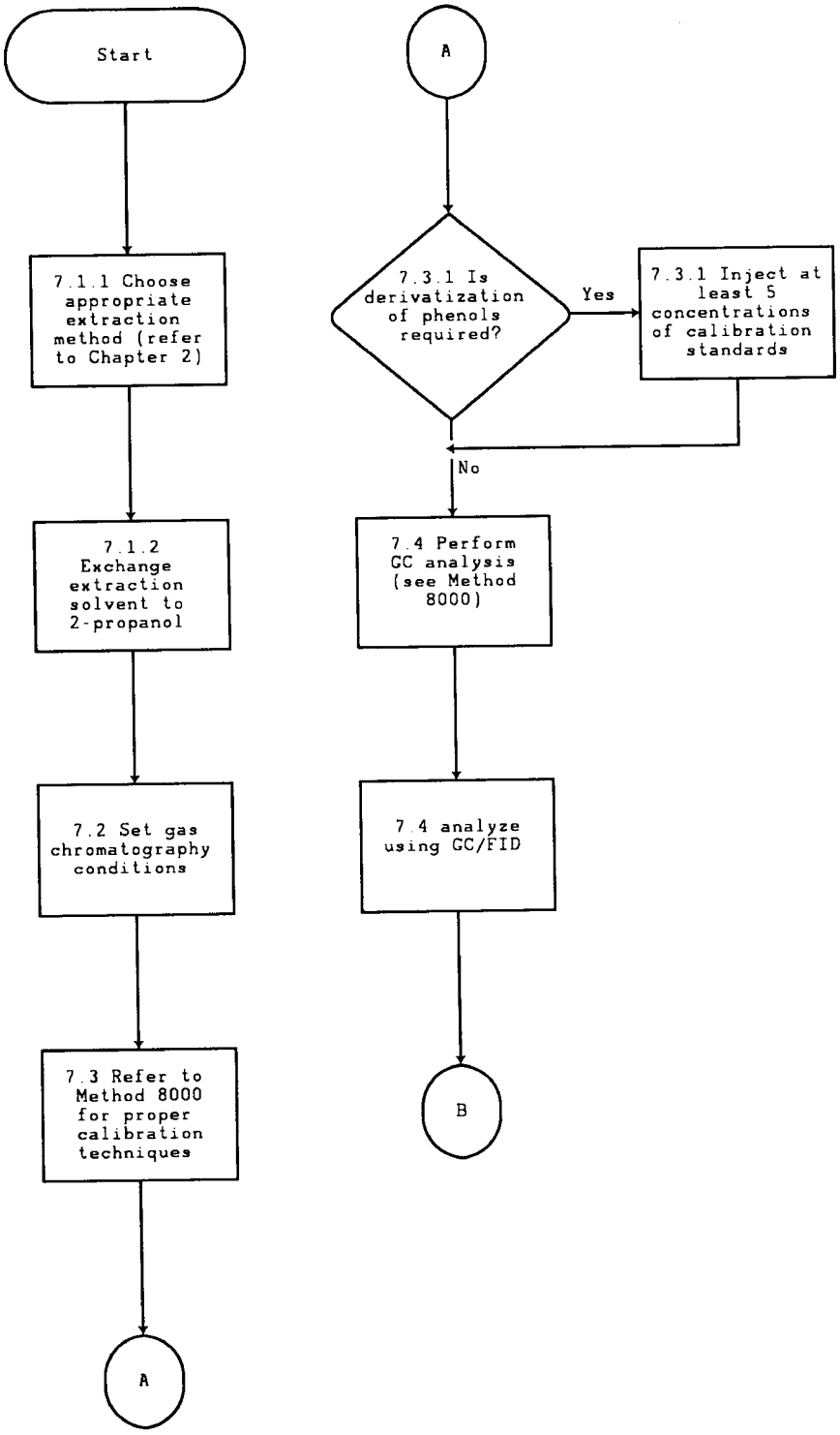
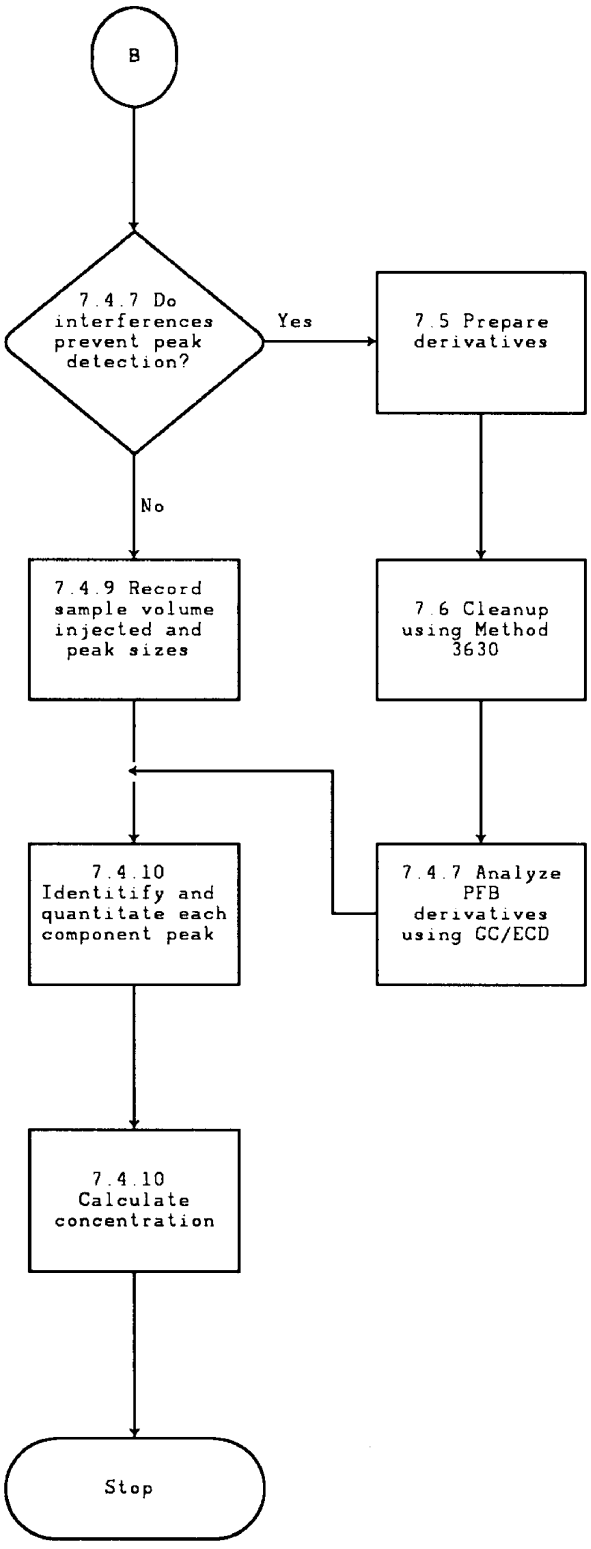


Figure 2
Gas Chromatogram of PFB Derivatives of Phenols




Start
7.1.1 Choose appropriate extraction method (refer to Chapter 2)
7.1.2 Exchange extraction solvent to 2-propanol
7.2 Set gas chromatography conditions
7.3 Refer to Method 8000 for proper calibration techniques
A
A
7.3.1 Is derivatization of phenols required?
Yes
7.3.1 Inject at least 5 concentrations of calibration standards
No
7.4 Perform GC analysis (see Method 8000)
7.4 analyze using GC/FID
B


B
7.4.7 Do interferences prevent peak detection?
Yes
7.5 Prepare derivatives
No
7.4.9 Record sample volume injected and peak sizes
7.6 Cleanup using Method 3630
7.4.10 Identitify and quantitate each component peak
7.4.7 Analyze PFB derivatives using GC/ECD
7.4.10 Calculate concentration
Stop