METHOD 8091

NITROAROMATICS AND CYCLIC KETONES BY GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 Method 8091 is a gas chromatographic (GC) method used to determine the concentration of nitroaromatics and cyclic ketones. It describes wide-bore, open-tubular, capillary column gas chromatography procedures using either electron capture (ECD) or nitrogen-phosphorous (NPD) detectors. The following RCRA analytes can be determined by this method:

Compound	CAS No.[a]
1,4-Dinitrobenzene	100-25-4
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
1,4-Naphthoquinone	130-15-4
Nitrobenzene	98-95-3
Pentachloronitrobenzene	82-68-8

[a] Chemical Abstract Service Registry Number.

1.2 The following non-RCRA analytes can also be determined by this method:

Compound	CAS No.[a]
Benefin	1861-40-1
Butralin	33629-47-9
1-Chloro-2,4-dinitrobenzene	97-00-7
1-Chloro-3,4-dinitrobenzene	610-40-2
1-Chloro-2-nitrobenzene	88-73-3
1-Chloro-4-nitrobenzene	100-00-5
2-Chloro-6-nitrotoluene	83-42-1
4-Chloro-2-nitrotoluene	89-59-8
4-Chloro-3-nitrotoluene	89-60-1
2,3-Dichloronitrobenzene	3209-22-1
2,4-Dichloronitrobenzene	611-06-3
3,5-Dichloronitrobenzene	618-62-2
3,4-Dichloronitrobenzene	99-54-7
2,5-Dichloronitrobenzene	89-61-2

Compound	CAS No.[a]
Dinitramine	29091-05-2
1,2-Dinitrobenzene	528-29-0
1,3-Dinitrobenzene	99-65-0
Isopropalin	33820-53-0
1,2-Naphthoquinone	524-42-5
2-Nitrotoluene	88-72-2
3-Nitrotoluene	99-08-1
4-Nitrotoluene	99-99-0
Penoxalin (Pendimethalin)	40487-42-1
Profluralin	26399-36-0
2,3,4,5-Tetrachloronitrobenzene	879-39-0
2,3,5,6-Tetrachloronitrobenzene	117-18-0
1,2,3-Trichloro-4-nitrobenzene	17700-09-3
1,2,4-Trichloro-5-nitrobenzene	89-69-0
2,4,6-Trichloronitrobenzene	18708-70-8
Trifluralin	1582-09-8

1.3 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs and skilled 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 Method 8091 provides gas chromatographic conditions for the detection of ppb concentrations of nitroaromatics and cyclic ketones in water and soil or ppm concentrations in waste samples. Prior to use of this method, appropriate sample extraction techniques must be used for environmental samples (refer to Chapter Two and Method 3500). Both neat and diluted organic liquids (Method 3580) may be analyzed by direct injection. Analysis is accomplished by gas chromatography utilizing an instrument equipped with wide bore capillary columns and one or more electron capture detectors or nitrogen-phosphorus detectors (NPD).

3.0 INTERFERENCES

3.1 Refer to Method 3500, 3600, and 8000.

3.2 The electron capture detector responds to all electronegative compounds. Therefore, interferences are possible from other halogenated compounds, as well as phthalates and other oxygenated compounds such as organonitrogen, organosulfur, and organophosphorus compounds. Second column confirmation or GC/MS confirmation is necessary to ensure proper analyte identification unless previous characterization of the sample source will ensure proper identification.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the syringe used for injection must be thoroughly rinsed between samples with solvent. Whenever a highly concentrated sample extract is encountered, it should be followed by the analysis of a solvent blank to check for

cross-contamination. Additional solvent blanks interspersed with the sample extracts should be considered whenever the analysis of a solvent blank indicates cross-contamination problems.

3.4 In certain cases some compounds coelute on either one or both columns. In these cases the compounds must be reported as coeluting. The mixture can be reanalyzed by GC/MS techniques if concentration permits (see Method 8270).

3.4.1 DB-5 column:

2,4,6-trichloronitrobenzene/1,3-dinitrobenzene
1-chloro-2,4-dinitrobenzene/1-chloro-3,4-dinitrobenzene/
1,2,3-trichloro-4-nitrobenzene

3.4.2 DB-1701 column:

2,4-dichloronitrobenzene/4-chloro-3-nitrotoluene
2,4,6-trichloronitrobenzene/1,4-naphthoquinone
1-chloro-2,4-dinitrobenzene/2,3,4,5-tetrachloronitrobenzene

3.4.3 In addition, on the DB-5 column, 2,5-dichloronitrobenzene is not well resolved from 4-chloro-3-nitrotoluene. Also, Trifluralin is not well resolved from Benefin. On the DB-1701 column, compound pairs that are not well resolved include 4-nitrotoluene/1-chloro-3-nitrobenzene and Trifluralin/Benefin.

3.5 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph: An analytical system complete with a gas chromatograph suitable for on-column and split/splitless injection, and all accessories, including syringes, analytical columns, gases, electron capture detectors or nitrogen-phosphorus detectors. A GC equipped with a single GC column and detector or other configurations of column and detector is also acceptable. A data system for measuring peak areas and/or peak heights, and dual display of chromatograms is recommended.

4.1.1 Suggested GC Columns: Alternative columns may be used to provide the separation needed to resolve all target analytes listed in Sec. 1.1 of this method. Refer to Chapter One for additional information regarding column performance and QA requirements.

4.1.1.1 Column 1 - 30 m x 0.53 mm ID fused-silica open- tubular column, crosslinked and chemically bonded with 95 percent dimethyl and 5 percent diphenyl-polysiloxane (DB-5, RT_x-5, SPB-5, or equivalent), 0.83 µm or 1.5 µm film thickness.

4.1.1.2 Column 2 - 30 m x 0.53 mm ID fused-silica open-tubular column crosslinked and chemically bonded with 14 percent cyanopropylphenyl and 86 percent dimethyl-polysiloxane (DB-1701, RT_x-1701, or equivalent), 1.0 µm film thickness.

4.1.2 Splitter: If the splitter approach to dual column injection is chosen, following are three suggested splitters. An equivalent splitter is acceptable. See Sec. 7.5.1 for a caution on the use of splitters.

4.1.2.1 Splitter 1 - J&W Scientific press-fit Y-shaped glass 3-way union splitter (J&W Scientific, Catalog No. 705-0733).

4.1.2.2 Splitter 2 - Supelco 8-in glass injection tee, deactivated (Supelco, Catalog No. 2-3665M).

4.1.2.3 Splitter 3 - Restek Y-shaped fused-silica connector (Restek, Catalog No. 20405).

4.1.3 Column rinsing kit (optional): Bonded-phase column rinse kit (J&W Scientific, Catalog No. 430-3000 or equivalent).

4.2 Microsyringes - 100 µL, 50 µL, 10 µL (Hamilton 701 N or equivalent), and 50 µL (Blunted, Hamilton 705SNR or equivalent).

4.3 Balances - Analytical, 0.0001 g, Top-loading, 0.01 g.

4.4 Volumetric flasks, Class A - 10 mL to 1000 mL.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, 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 chemicals are of sufficiently high purity to permit their use without affecting the accuracy of the determinations.

5.2 Solvents

5.2.1 Hexane, C_6H_{14} - Pesticide quality or equivalent.

5.2.2 Acetone, CH_3COCH_3 - Pesticide quality or equivalent.

5.2.3 Isooctane, $(CH_3)_3CCH_2CH(CH_3)_2$ - Pesticide quality or equivalent.

5.3 Stock standard solutions (1000 mg/L): Can be prepared from pure standard materials or can be purchased as certified solutions.

5.3.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure compound. Dissolve the compound in isooctane or hexane and dilute to volume in a 10 mL volumetric flask. (Isooctane is preferred because it is less volatile than hexane.) If compound purity is 96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution. Commercially prepared stock standard solutions can be used at any concentration if they are certified by the manufacturer or by an independent source.

5.3.2 For those compounds which are not adequately soluble in hexane or isooctane, dissolve the compound initially with a small volume of toluene, ethyl acetate or acetone and dilute to volume with isooctane or hexane.

5.4 Composite stock standard: Can be prepared from individual stock solutions. For composite stock standards containing less than 25 components, transfer exactly 1 mL of each individual stock solution at 1000 mg/L, add solvent, mix the solutions, and bring to volume in a 25 mL volumetric flask. For example, for a composite containing 20 individual standards, the resulting concentration of each component in the mixture, after the volume is adjusted to 25 mL, will be 40 mg/L. This composite solution can be further diluted to obtain the desired concentrations. For composite stock standards containing more than 25 components, use volumetric flasks of the appropriate volume (e.g., 50 mL, 100 mL).

5.5 Calibration standards: These should be prepared at a minimum of five concentrations by dilution of the composite stock standard with isooctane or hexane. The standard concentrations should correspond to the expected range of concentrations present in the field samples and should bracket the linear range of the detector.

5.6 Recommended internal standard: Prepare a solution of 1000 mg/L of hexachlorobenzene. For spiking, dilute this solution to 50 ng/µL. (This concentration may need to be more dilute depending on the detector chosen and its sensitivity. The internal standard response should be approximately 50 to 90% of full scale.) Use a spiking volume of 10.0 µL/mL of extract. The spiking concentration of the internal standards should be kept constant for all samples and calibration standards.

5.7 Recommended surrogate standard: Monitor the performance of the method using surrogate compounds. Surrogate standards are added to all samples, method blanks, matrix spikes, and calibration standards. Prepare a solution of 1000 mg/L of 1-chloro-3-nitrobenzene and dilute it to 10 ng/µL. (This concentration may need to be adjusted depending on the detector chosen and its sensitivity. The surrogate standard response should be approximately 100% of full scale.) Use a spiking volume of 100 µL for a 1 L aqueous sample.

5.8 Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4°C or cooler in polytetrafluoroethylene(PTFE)-sealed containers in the dark. All standard solutions must be replaced after six months or sooner if routine QC (Sec. 8.0) indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Extracts must be stored in the dark at or below 4°C and analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction and Cleanup:

7.1.1 Refer to Chapter Two and Method 3500 for guidance on choosing the appropriate extraction procedure. In general, water samples are extracted at a pH between 5 to 9 with

methylene chloride, using either Method 3510 or 3520. Solid samples are extracted using any of the extraction methods for solids listed in Method 3500, as appropriate.

7.1.2 If necessary, the samples may be cleaned up using Method 3620 (Florisil) and/or Method 3640 (Gel Permeation Chromatography). See Chapter Two, Sec. 2.0 and Method 3600 for general guidance on cleanup and method selection. Method 3660 is used for sulfur removal.

7.1.3 Prior to gas chromatographic analysis, the extraction solvent needs to be exchanged to hexane. The exchange is performed using the K-D procedures listed in each of the extraction methods. Any methylene chloride remaining in the extract will cause a very broad solvent peak.

7.2 Gas Chromatographic Conditions: Retention time information for each of the analytes is presented in Tables 1 and 3. The recommended GC operating conditions are provided in Tables 2 and 4. Figures 1, 2, and 3 illustrate typical chromatography of the method analytes for both columns when operated at the conditions specified.

7.3 Calibration:

7.3.1 Prepare calibration standards using the procedures in Sec. 5.0. Refer to Method 8000, Sec. 7.0 for proper calibration procedures. The procedure for internal or external calibration may be used.

7.3.2 Refer to Method 8000, Sec. 7.0 for the establishment of retention time windows.

7.4 Gas chromatographic analysis:

7.4.1 Method 8000, Sec. 7.0 provides instructions on calibration, establishing retention time windows, the analysis sequence, appropriate dilutions, and identification criteria.

7.4.2 Automatic injections of 1 µL are recommended. Hand injections of no more than 2 µL may be used if the analyst demonstrates quantitation precision less than or equal to 10 percent relative standard deviation. The solvent flush technique may be used if the amount of solvent is kept at a minimum. If the internal standard calibration technique is used, add 10 µL of the internal standard to each mL of sample extract prior to injection.

7.4.3 Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window. Normally, confirmation is necessary. Confirmation techniques include analysis on a second column with dissimilar stationary phase, by GC/MS (full scan or SIM) or by using a different detector and getting comparable data. See Sec. 7.0 of Method 8000 on "Compound Identification" for further information.

7.4.3.1 If partially overlapping or coeluting peaks are present, install columns with a dissimilar liquid phase or use a GC/MS technique. Interferences that prevent analyte identification and/or quantitation may possibly be removed by the cleanup techniques mentioned above.

7.4.4 Record the volume injected to the nearest 0.05 µL and the resulting peak size in area units or peak height. Using either the internal or the external calibration procedure (Method 8000), determine the quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes.

7.4.4.1 If the responses exceed the linear range of the system, dilute the extract and reanalyze. Peak height measurements are recommended, rather than peak area integration, when overlapping peaks may cause errors in area integration.

7.4.4.2 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

7.4.5 Determine the concentration of each identified analyte using the calculation formulae in Sec. 7.0 of Method 8000.

7.5 Instrument Maintenance:

7.5.1 Injection of sample extracts from waste sites often leaves a high boiling residue in the injection port area, splitters when used, and the injection port end of the chromatographic column. This residue affects chromatography in many ways (i.e., peak tailing, retention time shifts, analyte degradation, etc.) and, therefore, instrument maintenance is very important. Residue buildup in a splitter may limit flow through one leg and therefore change the split ratios. If this occurs during an analytical run, the quantitative data may be incorrect. Proper cleanup techniques will minimize the problem and instrument QC will indicate when instrument maintenance is required.

7.5.2 Suggested chromatograph maintenance: Corrective measures may require any one or more of the following remedial actions. Also see Sec. 7.0 in Method 8000 for additional guidance on corrective action for capillary columns and the injection port.

7.5.2.1 Splitter connections: For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter or replace with a cleaned and deactivated splitter. Break off the first few inches (up to one foot) of the injection port side of the column. Remove the columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

7.5.2.2 Column rinsing: The column should be rinsed with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone; methylene chloride is a satisfactory final rinse and in some cases may be the only solvent required. The column should then be filled with methylene chloride and allowed to remain flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen passing through the column.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. Each laboratory should

maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and includes evaluation of retention time windows, calibration verification and chromatographic analysis of samples.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, a matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.2 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.3 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 Table 1 lists the retention times of the target analytes. Figure 1 shows a chromatogram of the target analytes eluted from a pair of DB-5/DB-1701 columns and detected using electron capture detectors (ECD) under the GC conditions listed in Table 2.

9.2 Table 3 provides the retention times and recovery data of the target analytes. GC conditions used during the recovery study are listed in Table 4. Chromatograms of the standard mixes used in the recovery study are provided in Figures 2 and 3.

9.3 The laboratory should perform a Method Detection Limit (MDL) study and generate its own performance data (precision and accuracy) for matrix spike and surrogate compounds. Refer to Method 8000 for guidance.

10.0 REFERENCES

1. Lopez-Avila, V., Baldin, E., Benedicto, J, Milanes, J., Beckert, W.F., "Application of Open-Tubular Columns to SW 846 GC Methods", final report to the U.S. Environmental Protection Agency on Contract 68-03-3511, Mid-Pacific Environmental Laboratory, Mountain View, CA, 1990.

2. Tsang, S., Marsden, P.J., Chau, N., "Performance Data for Methods 8041, 8091, 8111, and 8121A", draft report to U.S. Environmental Protection Agency on Contract 68-W9-0011, Science Applications International Corp., San Diego, CA, 1992.

TABLE 1

RETENTION TIMES[a] OF THE NITROAROMATICS AND CYCLIC KETONES

Compound No.	Compound	CAS No.	DB-5 RT(min)	DB-1701 RT(min)
1	Nitrobenzene	98-95-3	4.71	4.23
2	2-Nitrotoluene	88-72-2	6.08	5.32
3	3-Nitrotoluene	99-08-1	6.93	6.22
4	4-Nitrotoluene	99-99-0	7.35	6.73
5	1-Chloro-3-nitrobenzene (Surr.)	121-73-3	7.66	6.85
6	1-Chloro-4-nitrobenzene	100-00-5	7.9	7.15
7	1-Chloro-2-nitrobenzene	88-73-3	8.09	7.78
8	2-Chloro-6-nitrotoluene	83-42-1	9.61	8.32
9	4-Chloro-2-nitrotoluene	89-59-8	9.76	8.62
10	3,5-Dichloronitrobenzene	618-62-2	10.42	8.84
11	2,5-Dichloronitrobenzene	89-61-2	11.46	10.62
12	2,4-Dichloronitrobenzene	611-06-3	11.73	10.84
13	4-Chloro-3-nitrotoluene	89-60-1	11.31	10.84
14	3,4-Dichloronitrobenzene	99-54-7	12.24	11.04
15	2,3-Dichloronitrobenzene	3209-22-1	12.58	12.01
16	2,4,6-Trichloronitrobenzene	18708-70-8	13.97	12.31
17	1,4-Naphthoquinone	130-15-4	12.98	12.31
18	1,2,4-Trichloro-5-nitrobenzene	89-69-0	15.97	14.46
19	1,4-Dinitrobenzene	100-25-4	13.41	14.72
20	2,6-Dinitrotoluene	606-20-2	14.44	15.16
21	1,3-Dinitrobenzene	99-65-0	13.97	15.68
22	1,2,3-Trichloro-4-nitrobenzene	17700-09-3	17.61	16.51
23	2,3,5,6-Tetrachloronitrobenzene	117-18-0	19.41	17.11
24	1,2-Dinitrobenzene	528-29-0	14.76	17.51
25	2,4-Dinitrotoluene	121-14-2	16.92	18.16
26	1-Chloro-2,4-dinitrobenzene	97-00-7	17.85	19.55
27	2,3,4,5-Tetrachloronitrobenzene	879-39-0	21.51	19.55
28	1-Chloro-3,4-dinitrobenzene	610-40-2	17.85	19.85
29	Trifluralin	1582-09-8	21.81	20.31
30	Benefin	1861-40-1	21.94	20.46
31	Pentachloronitrobenzene	82-68-8	25.13	22.33
32	Profluralin	26399-36-0	25.39	23.81
33	Dinitramine	29091-05-2	26.45	27.06
34	Butralin	33629-47-9	32.41	31.03
35	Isopropalin	33820-53-0	32.71	31.33

(continued)

TABLE 1 (continued)

Compound No.	Compound	CAS No.	RT (min) DB-5	RT (min) DB-1701
36	Penoxalin (Pendimethalin)	40487-42-1	33.05	31.67
37	1,2-Naphthoquinone	524-42-5	c	c
38	2-Chloro-4-nitrotoluene	121-86-8	b	b
Int. Std.	Hexachlorobenzene	118-74-1	23.18	18.72

[a] See Table 2 for operating conditions.
b Not available.
c Not detected at 1 ng per injection.
NOTE: These data are from Reference 1.

TABLE 2

DUAL COLUMN GC OPERATING CONDITIONS FOR NITROAROMATICS

GC Instrument: Varian 6000 with dual electron capture detectors

Column 1: Type: DB-5 (J&W Scientific)
Dimensions: 30 m x 0.53 mm ID
Film Thickness: 1.5 µm

Column 2: Type: DB-1701 (J&W Scientific)
Dimensions: 30 m x 0.53 mm ID
Film Thickness: 1.0 µm

Type of splitter: J&W Scientific press-fit Y-shaped inlet splitter

Carrier gas flowrate (mL/min): 6 (Helium)
Makeup gas flowrate (mL/min): 20 (Nitrogen)

Injector temperature: 250°C
Detector temperature: 320°C
Temperature program: 120°C (1.0 min hold) to 200°C (1 min hold) at 3°C/min then to 250°C (4 min hold) at 8°C/min.
Injection volume: 2 µL
Type of injection: Flash vaporization
Solvent: Hexane
Range: 10
Attenuation: 64 (DB-1701)/64 (DB-5)

TABLE 3

RETENTION TIMES AND RECOVERY OF NITROAROMATICS				
Analyte	R_t, min	Spiking Conc. (ng/g)	Recovery (%)	% RSD
MIX 1				
1,2:3,4-diepoxy butane	3.23	5,000	22	18.1
Nitrobenzene	11.51	5,000	85	6.9
2-Nitrotoluene	14.13	5,000	80	5.4
3-Nitrotoluene	15.52	5,000	83	6.8
4-Nitrotoluene	16.22	5,000	97	6.2
1-Chloro-3-nitrobenzene[a]	16.64	100	103	6.2
2,3-Dichloronitrobenzene	22.48	100	102	7.3
1,4-Naphthoquinone	23.29	200	35	23.1
1,3-Dinitrobenzene	24.25	400	80	13.1
1,2-Dinitrobenzene	24.69	200	99	17.0
3-Nitroaniline	25.44	10,000	54	17.8
2,4-Dinitrotoluene	26.95	200	75	13.9
4-Nitroaniline	28.91	5,000	53	29.6
Trifluralin	30.25	200	127	4.4
Pentachloronitrobenzene	32.26	100	129	5.8
4-Nitroquinoline-1-oxide	36.05	5,000	6.7	18.5
MIX 2				
1-Chloro-3-nitrobenzene[a]	16.64	100	98	3.0
2-Nitroaniline	22.87	5,000	88	3.6
1,4-Dinitrobenzene	23.82	200	142	2.9
2,6-Dinitrotoluene	24.49	200	192	6.2
5-Nitro-o-toluidine	28.91	5,000	60	42

[a] Recommended Surrogate
n = 5 samples

NOTE: This table is from Reference 2. See Table 4 for operating conditions used in this table.

TABLE 4

GC OPERATING CONDITIONS USED FOR RECOVERY DATA IN TABLE 3

Column: DB-5 30 m x 0.53 mm ID.
Carrier gas: Nitrogen at 6 mL/min with hydrogen at 30 mL/min.
Total nitrogen flow: 60 mL/min (carrier and makeup).
Injector: Packed, megabore liner at 200°C.
Detector: ECD at 300°C.

Temperature Program:

70°C held for 1.5 minutes
4°C/min to 170°C
8°C/min to 275°C and held for 5.4 minutes

The total run time was 45 minutes.

NOTE: This table is from Reference 2.

FIGURE 1

GC/ECD CHROMATOGRAM OF NITROAROMATICS ANALYZED ON A
DB-5/DB-1701 FUSED-SILICA, OPEN-TUBULAR COLUMN PAIR

See Table 2 for operating conditions.

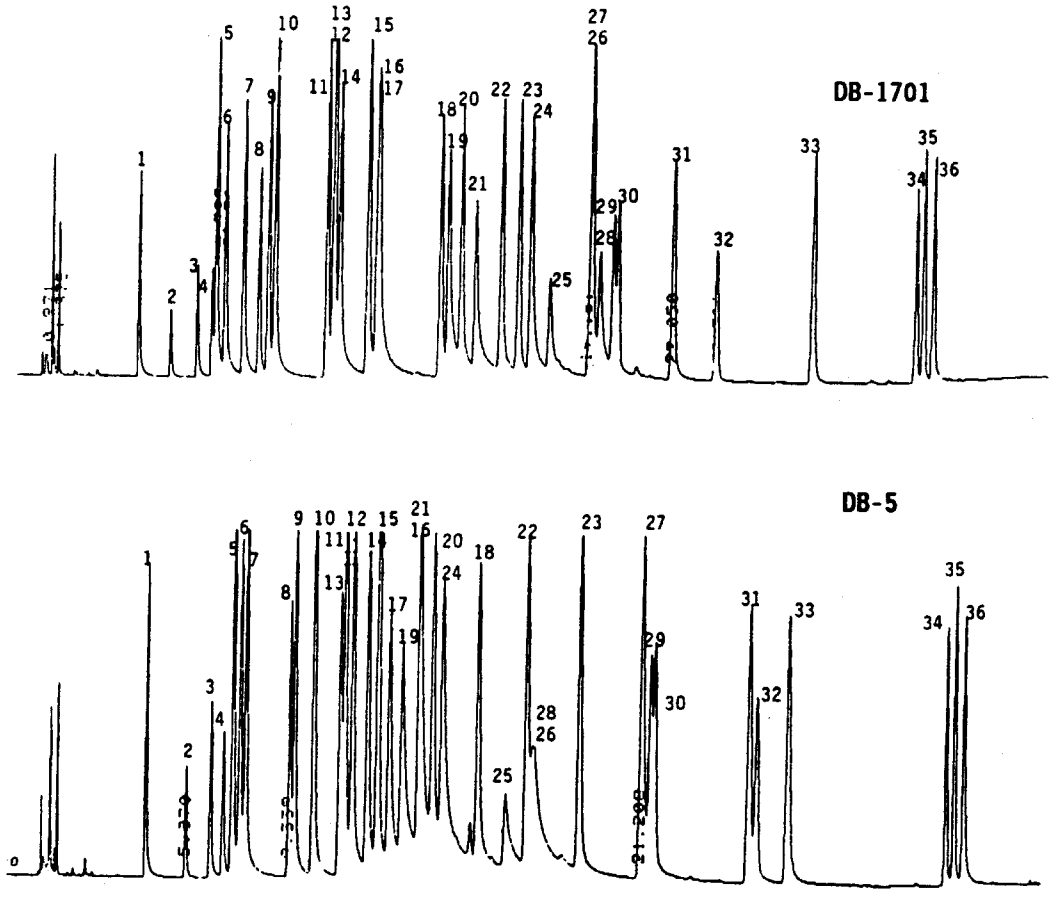


FIGURE 2

RECOVERY OF NITROAROMATICS - MIX 1

See Table 4 for operating conditions.

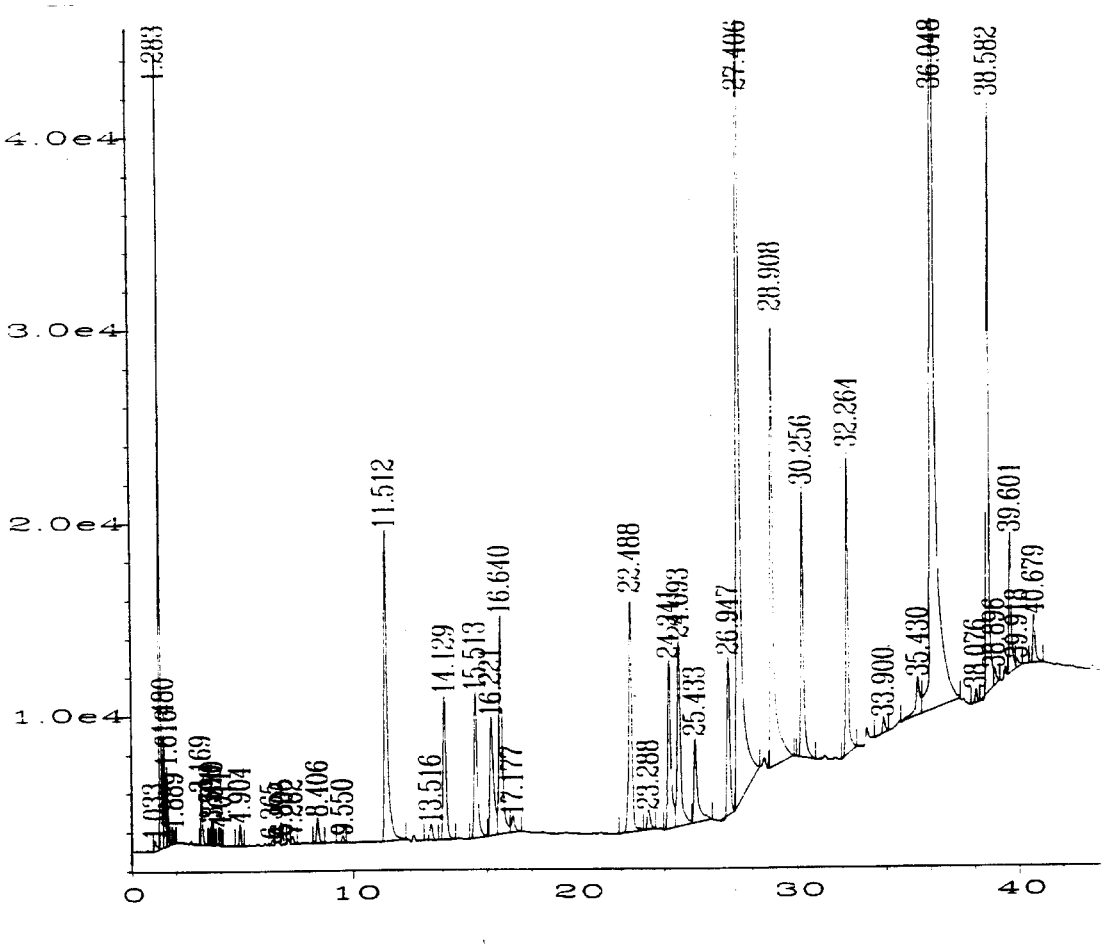
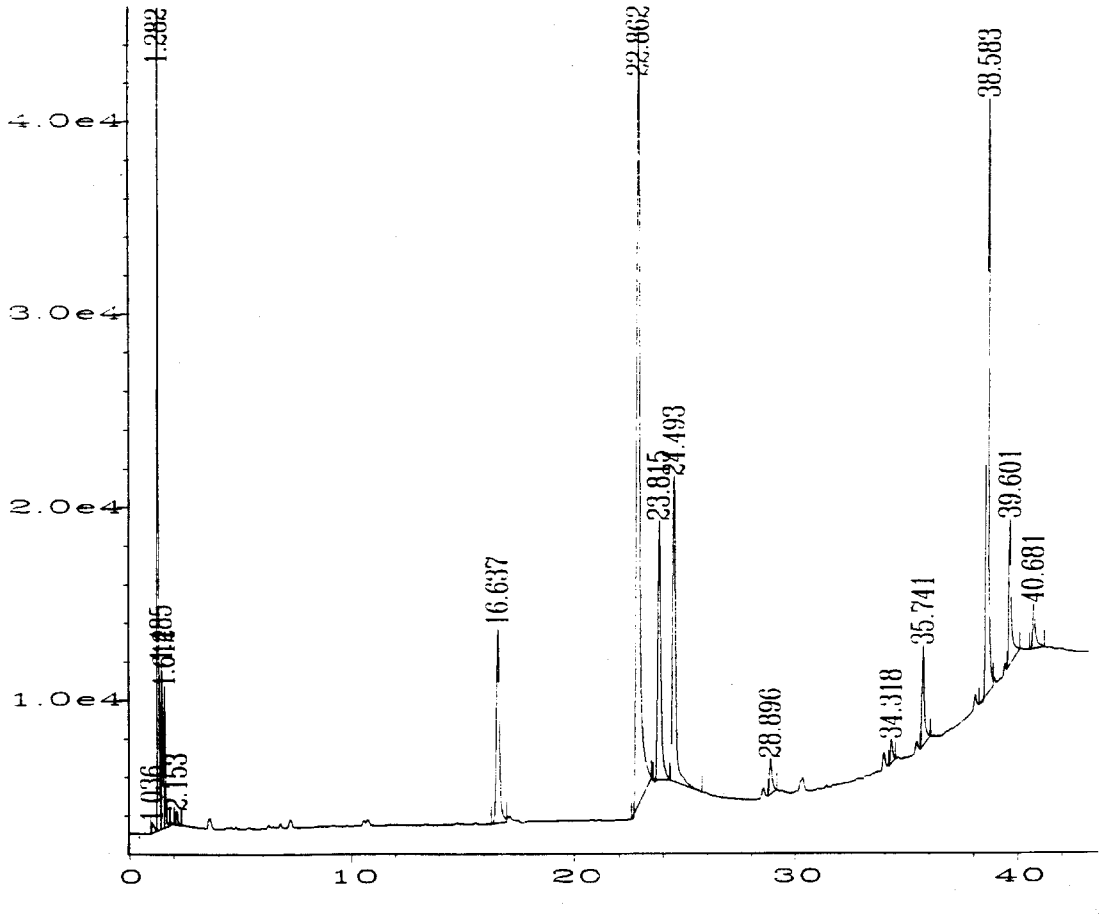
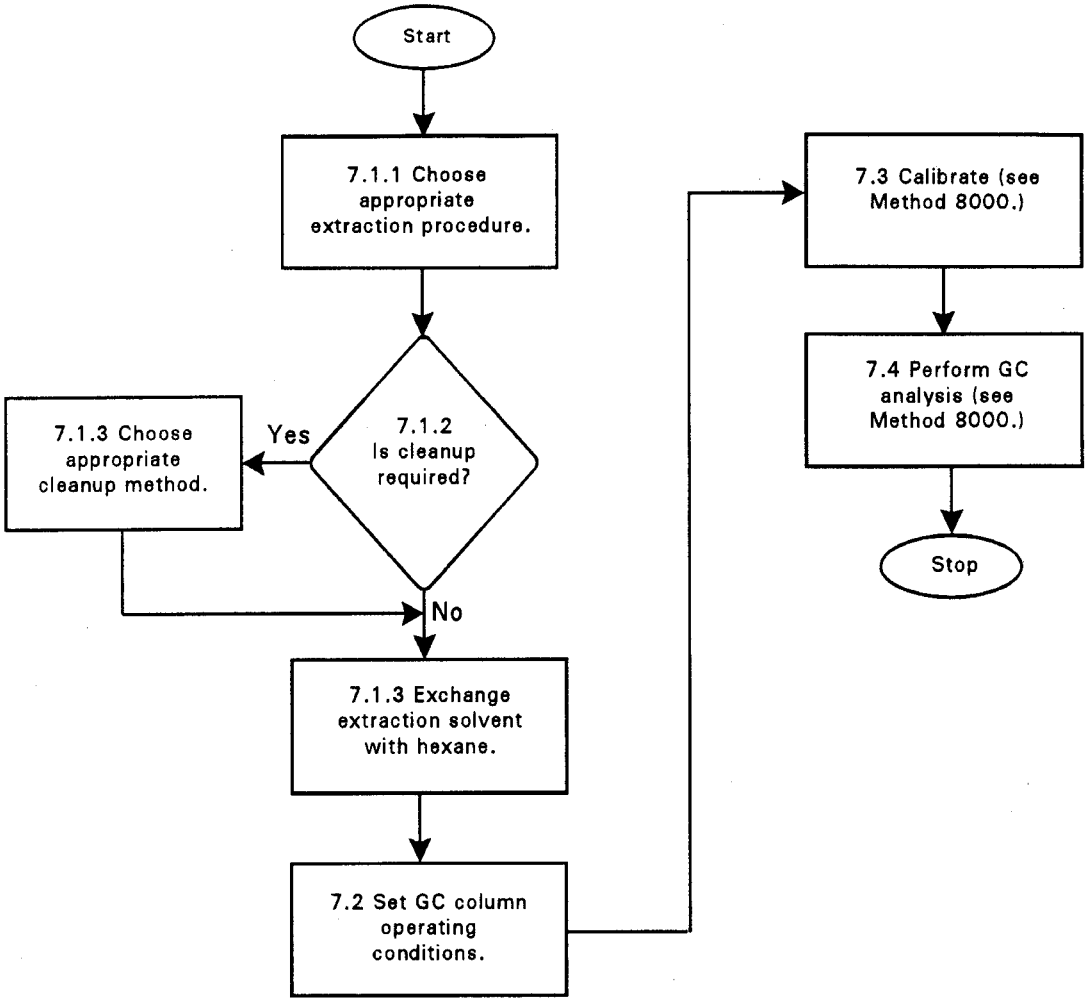


FIGURE 3

RECOVERY OF NITROAROMATICS - MIX 2

See Table 4 for operating conditions.




Start
7.1.1 Choose appropriate extraction procedure.
7.1.2 Is cleanup required?
Yes
7.1.3 Choose appropriate cleanup method.
No
7.1.3 Exchange extraction solvent with hexane.
7.2 Set GC column operating conditions.
7.3 Calibrate (see Method 8000.)
7.4 Perform GC analysis (see Method 8000.)
Stop