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Office of Water

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Method 1625, Revision B: Semivolatile Organic Compounds by Isotope Dilution GC/MS

APPENDIX A TO PART 136
METHOD 1625 REVISION B—SEMIVOLATILE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS

1. Scope and Application

1.1 This method is designed to determine the semivolatile toxic organic pollutants associated with the 1976 Consent Decree and additional compounds amenable to extraction and analysis by capillary column gas chromatography-mass spectrometry (GC/MS).

1.2 The chemical compounds listed in Tables 1 and 2 may be determined in municipal and industrial discharges by this method. The method is designed to meet the survey requirements of Effluent Guidelines Division (EGD) and the National Pollutants Discharge Elimination System (NPDES) under 40 CFR Part 136.1. Any modifications of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR Parts 136.4 and 136.5.

1.3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The limits listed in Tables 3 and 4 represent the minimum quantity that can be detected with no interferences present.

1.4 The GC/MS portions of this method are for use only by analysts experienced with GC/MS or under the close supervision of such qualified persons. Laboratories unfamiliar with analyses of environmental samples by GC/MS should run the performance tests in Reference 1 before beginning.

2. Summary of Method

2.1 Stable isotopically labeled analogs of the compounds of interest are added to a one liter wastewater sample. The sample is extracted at pH 12-13, then at pH <2 with methylene chloride using continuous extraction techniques. The extract is dried over sodium sulfate and concentrated to a volume of 1 mL. An internal standard is added to the extract, and the extract is injected into the gas chromatograph (GC). The compounds are separated by GC and detected by a mass spectrometer (MS). The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a compound (qualitative analysis) is performed by comparing the GC retention time and background corrected characteristic spectral masses with those of authentic standards.

2.3 Quantitative analysis is performed by GC/MS using extracted ion current profile (EICP) areas. Isotope dilution is used when labeled compounds are available; otherwise, an internal standard method is used.

2.4 Quality is assured through reproducible calibration and testing of the extraction and GC/MS systems.

3. Contamination and Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms and spectra. All materials shall be demonstrated to be free from interferences under the conditions of analysis by running method blanks initially and with each sample lot (samples started through the extraction process on a given eight hour shift, to a maximum of 20). Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Glassware and, where possible, reagents are cleaned by solvent rinse and baking at 450°C for one hour minimum.

3.2 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the industrial complex or municipality being samples.

4. Safety

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References 2-4.

4.2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzidine benzo(a)anthracene, 3,3′-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, N-nitrosodimethylamine, and b-naphtylamine. Primary standards of these compounds shall be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5. Apparatus and Materials

5.1 Sampling equipment for discrete or composite sampling.

5.1.1 Sample bottle, amber glass, 1.1 L minimum. If amber bottles are not available, samples shall be protected from light. Bottles are detergent water washed, then solvent rinsed or baked at 450°C for one hour minimum before use.

5.1.2 Bottle caps—threaded to fit sample bottles. Caps are lined with Teflon. Aluminum foil may be substituted if the sample is not corrosive. Liners are detergent water washed, then reagent water (Section 6.5) and solvent rinsed, and baked at approximately 200°C for one hour minimum before use.

5.1.3 Compositing equipment—automatic or manual compositing system incorporating glass containers for collection of a minimum 1.1 L. Sample containers are kept at 0-4°C during sampling. Glass or Teflon tubing only shall be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only. Before use, the tubing is thoroughly rinsed with methanol, followed by repeated rinsings with reagent water (Section 6.5) to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.

5.2 Continuous liquid-liquid extractor—Teflon or glass conncecting joints and stopcocks without lubrication (Hershberg-Wolf Extractor) one liter capacity, Ace Glass 6841-10, or equivalent.

5.3 Drying column—15-20 mm i.d. Pyrex chromatographic column equipped with coarse glass frit or glass wool plug.

5.4 Kuderna-Danish (K-D) apparatus

5.4.1 Concentrator tube—10 mL, graduated (Kontes K-570050-1025, or equivalent) with calibration verified. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

5.4.2 Evaporation flask—500 mL (Kontes K-570001-0500, or equivalent), attached to concentrator tube with springs (Kontes K-662750-0012).

5.4.3 Snyder column—three ball macro (Kontes K-503000-0232, or equivalent).

5.4.4 Snyder column—two ball micro (Kontes K-469002-0219, or equivalent).

5.4.5 Boiling chips—approx 10/40 mesh, extracted with methylene chloride and baked at 450°C for one hour minimum.

5.5 Water bath—heated, with concentric ring cover, capable of temperature control ±2°C, installed in a fume hood.

5.6 Sample vials—amber glass, 2-5 mL with Teflon-lined screw cap.

5.7 Analytical balance—capable of weighing 0.1 mg.

5.8 Gas chromatograph—shall have splitless or on-column injection port for capillary column, temperature program with 30°C hold, and shall meet all of the performance specifications in Section 12.

5.8.1 Column—30 ±5 m x 0.25 ±0.02 mm i.d. 5% phenyl, 94% methyl, 1% vinyl silicone bonded phase fused silica capillary column (J & W DB-5, or equivalent).

5.9 Mass spectrometer—70 eV electron impact ionization, shall repetitively scan from 35-450 amu in 0.95-1.00 second, and shall produce a unit resolution (valleys between m/z 441-442 less than 10% of the height of the 441 peak), backgound corrected mass spectrum from 50 ng decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet. The spectrum shall meet the mass-intensity criteria in Table 5 (Reference 5). The mass spectrometer shall be interfaced to the GC such that the end of the capillary column terminates within one centimeter of the ion source but does not intercept the electron or ion beams. All portions of the column which connect the GC to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.10 Data system—shall collect and record MS data, store mass-intensity data in spectral libraries, process GC/MS data, generate reports, and shall compute and record response factors.

5.10.1 Data acquisition—mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.10.2 Mass spectral libraries-user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GC/MS runs for the compounds of interest (Section 7.2).

5.10.3 Data processing—the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GC/MS analysis. Software routines shall be employed to compute retention times and peak areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.10.4 Response factors and multipoint calibrations—the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and multipoint calibration curves (Section 7). Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity. Statistics on initial (Section 8.2) and on-going (Section 12.7) performance shall be computed and maintained.

6. Reagents and Standards

6.1 Sodium hydroxide—reagent grade, 6 N in reagent water.

6.2 Sulfuric acid—reagent grade, 6 N in reagent water.

6.3 Sodium sulfate—reagent grade, granular anhydrous, rinsed with methylene chloride (20 mL/g) and conditioned at 450°C for one hour minimum.

6.4 Methylene chloride—distilled in glass (Burdick and Jackson, or equivalent).

6.5 Reagent water—water in which the compounds of interest and interfering compounds are not detected by this method.

6.6 Standard solutions—purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96% or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark at -20 to -10°C in screw-capped vials with Teflon-lined lids. A mark is placed on the vial at the level of the solution so that solvent evaporation loss can be detected. The vials are brought to room temperature prior to use. Any precipitate is redissolved and solvent is added if solvent loss has occurred.

6.7 Preparation of stock solutions-prepare in methylene chloride, benzene, p-dioxane, or a mixture of these solvents per the steps below. Observe the safety precautions in Section 4. The large number of labeled and unlabeled acid, base/neutral, and Appendix C compounds used for combined calibration (Section 7) and calibration verification (Section 12.5) require high concentrations (approx 40 mg/mL) when individual stock solutions are prepared, so that dilutions of mixtures will permit calibration with all compounds in a single set of solutions. The working range for most compounds is 10-200 µg/mL. Compounds with a reduced MS response may be prepared at higher concentrations.

6.7.1 Dissolve an appropriate amount of assayed reference material in a suitable solvent. For example, weigh 400 mg naphthalene in a 10 mL ground glass stoppered volumetric flask and fill to the mark with benzene. After the naphthalene is completely dissolved, transfer the solution to a 15 mL vial with Teflon-lined cap.

6.7.2 Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Quality control check samples that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.7.3 Stock standard solutions shall be replaced after six months, or sooner if comparison with quality control check samples indicates a change in concentration.

6.8 Labeled compound spiking solution—from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution at a concentration of 200 µg/mL, or at a concentration appropriate to the MS response of each compound.

6.9 Secondary standard—using stock solutions (Section 6.7), prepare a secondary standard containing all of the compounds in Tables 1 and 2 at a concentration of 400 µg/mL, or higher concentration appropriate to the MS response of the compound.

6.10 Internal standard solution—prepare 2,2′-difluorobiphenyl (DFB) at a concentration of 10 mg/mL in benzene.

6.11 DFTPP solution—prepare at 50 µg/mL in acetone.

6.12 Solutions for obtaining authentic mass spectra (Section 7.2)—prepare mixtures of compounds at concentrations which will assure authentic spectra are obtained for storage in libraries.

6.13 Calibration solutions—combine 0.5 mL of the solution in Section 6.8 with 25, 50, 125, 250, and 500 µL of the solution in Section 6.9 and bring to 1.00 mL total volume each. This will produce calibration solutions of nominal 10, 20, 50, 100, and 200 µg/mL of the pollutants and a constant nominal 100 µg/mL of the labeled compounds. Spike each solution with 10 µL of the internal standard solution (Section 6.10). These solutions permit the relative response (labeled to unlabeled) to be measured as a function of concentration (Section 7.4).

6.14 Precision and recovery standard—used for determination of initial (Section 8.2) and on-going (Section 12.7) precision and recovery. This solution shall contain the pollutants and labeled compounds at a nominal concentration of 100 µg/mL.

6.15 Stability of solutions—all standard solutions (Sections 6.8-6.14) shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standards will remain acceptable if the peak area at the quantitation mass relative to the DFB internal standard remains within ±15% of the area obtained in the initial analysis of the standard.

7. Calibration

7.1 Assemble the GC/MS and establish the operating conditions in Table 3. Analyze standards per the procedure in Section 11 to demonstrate that the analytical system meets the detection limits in Tables 3 and 4, and the mass-intensity criteria in Table 5 for 50 ng DFTPP.

7.2 Mass spectral libraries—detection and identification of compounds of interest are dependent upon spectra stored in user created libraries.

7.2.1 Obtain a mass spectrum of each pollutant, labeled compound, and the internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. That only a single compound is present is determined by examination of the spectrum. Fragments not attributable to the compound under study indicate the presence of an interfering compound.

7.2.2 Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to “enhance” the spectrum may eliminate distortion, but may also eliminate authentic masses or introduce other distortion.

7.2.3 The authentic reference spectrum is obtained under DFTPP tuning conditions (Section 7.1 and Table 5) to normalize it to spectra from other instruments.

7.2.4 The spectrum is edited by saving the five most intense mass spectral peaks and all other mass spectral peaks greater than 10% of the base peak. This edited spectrum is stored for reverse search and for compound confirmation.

7.3 Analytical range—demonstrate that 20 ng anthracene or phenanthrene produces an area at m/z 178 approx one-tenth that required to exceed the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required, and to diagnose instrument sensitivity problems (Section 15.4). The 20 µg/mL calibration standard (Section 6.13) can be used to demonstrate this performance.

7.3.1 Polar compound detection—demonstrate that unlabeled pentachlorophenol and benzidine are detectable at the 50 µg/mL level (per all criteria in Section 13). The 50 µg/mL calibration standard (Section 6.13) can be used to demonstrate this performance.

7.4 Calibration with isotope dilution—isotope dilution is used when (1) labeled compounds are available, (2) interferences do not preclude its use, and (3) the quantitation mass extracted ion current profile (EICP) area for the compound is in the calibration range. If any of these conditions preclude isotope dilution, internal standard methods (Section 7.5 or 7.6) are used.

7.4.1 A calibration curve encompassing the concentration range is prepared for each compound to be determined. The relative response (pollutant to labeled) vs concentration in standard solutions is plotted or computed using a linear regression. The example in Figure 1 shows a calibration curve for phenol using phenol-d5 as the isotopic diluent. Also shown are the ±10% error limits (dotted lines). Relative Reponse (RR) is determined according to the procedures described below. A minimum of five data points are employed for calibration.

7.4.2 The relative response of a pollutant to its labeled analog is determined from isotope ratio values computed from acquired data. Three isotope ratios are used in this process:

R_x = the isotope ratio measured for the pure pollutant.
R_y = the isotope ratio measured for the labeled compound.
R_m = the isotope ratio of an analytical mixture of pollutant and labeled compounds.

The m/z's are selected such that $R_x > R_y$. If R_m is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by internal or external standard methods.

7.4.3 Capillary columns usually separate the pollutant-labeled pair, with the labeled compound eluted first (Figure 2). For this case, R_x = [area m_1/z]/1, at the retention time of the pollutant (RT_2). R_y = 1/[area m_2/z, at the retention time of the labeled compound RT_1). R_m = [area at m_1/z (at RT_2)]/[area at RT_1)], as measured in the mixture of the pollutant and labeled compounds (Figure 2), and RR = R_m.

7.4.4 Special precautions are taken when the pollutant-labeled pair is not separated, or when another labeled compound with interfering spectral masses overlaps the pollutant (a case which can occur with isomeric compounds). In this case, it is necessary to determine the respective contributions of the pollutant and labeled compounds to the respective EICP areas. If the peaks are separated well enough to permit the data system or operator to remove the contributions of the compounds to each other, the equations in Section 7.4.3 apply. This usually occurs when the height of the valley between the two GC peaks at the same m/z is less than 10% of the height of the shorter of the two peaks. If significant GC and spectral overlap occur, RR is computed using the following equation:

$RR = (R_y - R_m)(R_x + 1)/(R_m - R_x)(R_y + 1)$, where R_x is measured as shown in Figure 3A, R_y is measured as shown in Figure 3B, and R_m is measured as shown in Figure 3C. For example, R_x = 46100/4780 = 9.644, R_y = 2650/43600 = 0.0608, R_m = 49200/48300 = 1.019. amd RR = 1.114.

7.4.5 To calibrate the analytical system by isotope dilution, analyze a 1.0 µL aliquot of each of the calibration standards (Section 6.13) using the procedure in Section 11. Compute the RR at each concentration.

7.4.6 Linearity—if the ratio of relative response to concentration for any compound is constant (less than 20% coefficient of variation) over the five point calibration range, and averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the five point calibration range.

7.5 Calibration by internal standard—used when criteria for istope dilution (Section 7.4) cannot be met. The internal standard to be used for both acid and base/neutral analyses is 2,2′-difluorobiphenyl. The internal standard method is also applied to determination of compounds having no labeled analog, and to measurement of labeled compounds for intra-laboratory statistics (Sections 8.4 and 12.7.4).

7.5.1 Response factors—calibration requires the determination of response factors (RF) which are defined by the following equation:

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

A_s = the area of the characteristic mass for the compound in the daily standard.

A_{is} = the area of the characteristic mass of the internal standard (µg/mL).

C_{is} = the concentration of the internal standard (µg/mL).

C_s = the concentration of the compound in the daily standard (µg/mL).

7.5.1.1 The response factor is determined for at least five concentrations appropriate to the response of each compound (Section 6.13); nominally, 10, 20, 50, 100, and 200 µg/mL. The amount of internal standard added to each extract is the same (100 µg/mL) so that C_{is}

remains constant. As/Ais is plotted vs. Cs/Cis for each compound in the standard (C_s) to produce a calibration curve.* .

7.5.1.2 Linearity—if the response factor (RF) for any compound is constant (less than 35% coefficient of variation) over the five point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the five point range.

7.6 Combined calibration—by using calibration solutions (Section 6.13) containing the pollutants, labeled compounds, and the internal standard, a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (Section 12.5) by analyzing the 100 0g/mL calibration standard (Section 6.13). Recalibration is required only if calibration verification (Section 12.5) criteria cannot be met.

8. Quality Assurance/Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in Section 8.5.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in Section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (Section 15).

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*This equation corrects an error made in the original method publication (49 FR 43234, October 26, 1984). This correction will be formalized through a rulemaking in FY97.

8.1.5 The laboratory shall, on an on-going basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (Section 6.14) that the analysis system is in control. These procedures are described in Sections 12.1, 12.5, and 12.7.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in Section 8.4.

8.2 Initial precision and accuracy—to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

8.2.1 Extract, concentrate, and analyze two sets of four 1 L aliquots (eight aliquots total) of the precision and recovery standard (Section 6.14) according to the procedure in Section 10.

8.2.2 Using results of the first set of four analyses, compute the average recovery ($\bar{X}$) in µg/mL and the standard deviation of the recovery (s) in 0g/µL for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and $\bar{X}$ with the corresponding limits for initial precision and accuracy in Table 8. If s and $\bar{X}$ for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, system performance is unacceptable for that compound.

NOTE: The large number of compounds in Table 8 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:

8.2.4 Using the results of the second set of four analyses, compute s and $\bar{X}$ for only those compounds which failed the test of the first set of four analyses (Section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compoulds fail again, the analysis system is not performing properly for these compounds. In this event, correct the problem and repeat the entire test (Section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Analyze each sample according to the method in Section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (Section 7.5).

8.3.3 Compare the labeled compound recovery for each compound with the corresponding limits in Table 8. If the recovery of any compounds falls outside its warning limit, method performance is unacceptable for that compound in that sample. Therefore, the sample is complex and is to be diluted and reanalyzed per Section 15.4.

8.4 As part of the QA program for the laboratory, method accuracy for wastewater samples shall be assessed and records shall be maintained. After the analysis of five wastewater samples for which the labeled compounds pass the tests in Section 8.3, compute the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p) for the labeled compounds only. Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. For example, if $\bar{P}$ = 90% and s_p = 10%, the accuracy interval is expressed as 70-100%. Update the accuracy assessment for each compound on a regular basis (e.g., after each 5-10 new accuracy measurements).

8.5 Blanks—reagent water blanks are analyzed to demonstrate freedom from contamination.

8.5.1 Extract and concentrate a blank with each sample lot (samples started through the extraction process on the same eight hour shift, to a maximum of 20 samples). Analyze the blank immediately after analysis of the precision and recovery standard (Section 6.14) to demonstrate freedom from contamination.

8.5.2 If any of the compounds of interest (Tables 1 and 2) or any potentially interfering compound is found in a blank at greater than 10 µg/L (assuming a response factor of 1 relative to the internal standard for compounds not listed in Tables 1 and 2), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for calibration (Section 7), calibration verification (Section 12.5), and for initial (Section 8.2) and on-going (Section 12.7) precision and recovery should be identical, so that the most precise results will be obtained. The GC/MS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analysis of semi-volatiles by this method.

8.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when internal or external standard methods are used.

9. Sample Collection, Preservation, and Handling

9.1 Collect samples in glass containers following conventional sampling practices (Reference 7). Composite samples are collected in refrigerated glass containers (Section 5.1.3) in accordance with the requirements of the sampling program.

9.2 Maintain samples at 0-4°C from the time collectimn until extraction. If residual chlorine is present, add 80 mg sodium thiosulfate per liter of water. EPA Methods 330.4 and 330.5 may be used to measure residual chlorine (Reference 8).

9.3 Begin sample extraction within seven days of collection, and analyze all extracts within 40 days of extraction.

10. Sample Extraction and Concentration (See Figure 4)

10.1 Labeled compound spiking-measure 1.00 ± 0.01 L of sample into a glass container. For untreated effluents, and samples which are expected to be difficult to extract and/or concentrate, measure an additional 10.0 ± 0.1 mL and dilute to a final volume of 1.00 ± 0.01 L with reagent water in a glass container.

10.1.1 For each sample or sample lot (to a maximum of 20) to be extracted at the same time, place three 1.00 ± 0.10 L aliquots of reagent water in glass containers.

10.1.2 Spike 0.5 mL of the labeled compound spiking solution (Section 6.8) into all samples and one reagant water aliquot.

10.1.3 Spike 1.0 mL of the precision and recovery standard (Section 6.14) into the two remaining reagent water aliquots.

10.1.4 Stir and equilibrate all solutions for one to two hours.

10.2 Base/neutral extraction—place 100-150 mL methylene chloride in each continuous extractor and 200-300 mL in each distilling flask.

10.2.1 Pour the sample(s), blank, and standard aliquots into the extractors. Rinse the glass containers with 50-100 mL methylene chloride and add to the respective extractor.

10.2.2 Adjust the pH of the waters in the extractors to 12-13 with 6N NaOH while monitoring with a pH meter. Begin the extraction by heating the flask until the methylene chloride is boiling. When properly adjusted, one to two drops of methylene chloride per second will fall from the condensor tip into the water. After one to two hours of extraction, test the pH and readjust to 12-13 if required. Extract for 18-24 hours.

10.2.3 Remove the distilling flask, estimate and record the volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7-10 cm anhydrous sodium sulfate. Rinse the distilling flask with 30-50 mL of methylene chloride and pour through the drying column. Collect the solution in a 500 mL K-D evaporator flask equipped with a 10 mL concentrator tube. Seal, label as the base/neutral fraction, and concentrate per Sections 10.4 to 10.5.

10.3 Acid extraction—adjust the pH of the waters in the extractors to 2 or less using 6 N sulfuric acid. Charge clean distilling flasks with 300-400 mL of methylene chloride.

Test and adjust the pH of the waters after the first one to two hours of extraction. Extract for 18-24 hours.

10.3.1 Repeat Section 10.2.3, except label as the acid fraction.

10.4 Concentration—concentrate the extracts in separate 500 mL K-D flasks equipped with 10 mL concentrator tubes.

10.4.1 Add one to two clean boiling chips to the flask and attach a three-ball macro Snyder column. Prewet the column by adding approximately 1 mL of methylene chloride through the top. Place the K-D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid has reached an apparent volume of 1 mL, remove the K-D apparatus from the bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of methylene chloride. A 5 mL syringe is recommended for this operation.

10.4.2 For performance standards (Sections 8.2 and 12.7) and for blanks (Section 8.5), combine the acid and base/neutral extracts for each at this point. Do not combine the acid and base/neutral extracts for samples.

10.5 Add a clean boiling chip and attach a two-ball micro Snyder column to the concentrator tube. Prewet the column by adding approx 0.5 mL methylene chloride through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature as required to complete the concentration in 5-10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid reaches an apparent volume of approx 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the micro Snyder column and rinse its lower joint into the concentrator tube with approx 0.2 mL of methylene chloride. Adjust the final volume to 1.0 mL.

10.6 Transfer the concentrated extract to a clean screw-cap vial. Seal the vial with a Teflon-lined lid, and mark the level on the vial. Label with the sample number and fraction, and store in the dark at -20 to -10°C until ready for analysis.

11. GC/MS Analysis

11.1 Establish the operating conditions given in Table 3 or 4 for analysis of the base/neutral or acid extracts, respectively. For analysis of combined extracts (Section 10.4.2), use the operating conditions in Table 3.

11.2 Bring the concentrated extract (Section 10.6) or standard (Sections 6.13 through 6.14) to room temperature and verify that any precipitate has redissolved. Verify the level on the extract (Sections 6.6 and 10.6) and bring to the mark with solvent if required.

11.3 Add the internal standard solution (Section 6.10) to the extract (use 1.0 µL of solution per 0.1 mL of extract) immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction. Mix thoroughly.

11.4 Inject a volume of the standard solution or extract such that 100 ng of the internal standard will be injected, using on-column or splitless injection. For 1 mL extracts, this volume will be 1.0 µL. Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes. Stop data collection after the benzo(ghi)perylene or pentachlorophenol peak elutes for the base/neutral or acid fraction, respectively. Return the column to the initial temperature for analysis of the next sample.

12. System and Laboratory Performance

12.1 At the beginning of each eight hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all pollutants and labeled compounds. For these tests, analysis of the 100 µg/mL calibration standard (Section 6.13) shall be used to verify all performance criteria. Adjustment and/or recalibration (per Section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, and precision and recovery standards be analyzed.

12.2 DFTPP spectrum validity—inject 1 µL of the DFTPP solution (Section 6.11) either separately or within a few seconds of injection of the standard (Section 12.1) analyzed at the beginning of each shift. The criteria in Table 5 shall be met.

12.3 Retention times—the absolute retention time of 2,2′-difluorobiphenyl shall be within the range of 1078-1248 seconds and the relative retention times of all pollutants and labeled compounds shall fall within the limits given in Tables 3 and 4.

12.4 GC resolution—the valley height between anthracene and phenanthrene at m/z 178 (or the analogs at m/z 188) shall not exceed 10% of the taller of the two peaks.

12.5 Calibration verification—compute the concentration of each pollutant (Tables 1 and 2) by isotope dilution (Section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (Section 7.5). Compute the concentration of the labeled compounds by the internal standard method. These concentrations are computed based on the calibration data determined in Section 7.

12.5.1 For each pollutant and labeled compound being tested, compare the concentration with the calibration verification limit in Table 8. If all compounds meet the acceptance criteria, calibration has been verified and analysis of blanks, samples, and precision and recovery standards may proceed. If, however, any compound fails, the measurement system is not performing properly for that compound. In this event, prepare a fresh calibration standard or correct the problem causing the failure and repeat the test (Section 12.1), or recalibrate (Section 7).

12.6 Multiple peaks-each compound injected shall give a single, distinct GC peak.

12.7 On-going precision and accuracy

12.7.1 Analyze the extract of one of the pair of precision and recovery standards (Section 10.1.3) prior to analysis of samples from the same lot.

12.7.2 Compute the concentration of each pollutant (Tables 1 and 2) by isotope dilution (Section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (Section 7.5). Compute the concentration of the labeled compounds by the internal standard method.

12.7.3 For each pollutant and labeled compound, compare the concentration with the limits for on-going accuracy in Table 8. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, system performance is unacceptable for that compound.

NOTE: The large number of compounds in Table 8 present a substantial probability that one or more will fail when all compounds are analyzed. To determine if the extraction/concentration system is out of control or if the failure is caused by probability, proceed as follows:

12.7.3.1 Analyze the second aliquot of the pair of precision and recovery standard (Section 10.1.3).

12.7.3.2 Compute the concentration of only those pollutants or labeled compounds that failed the previous test (Section 12.7.3). If these compounds now pass, the extraction/concentration processes are in control and analysis of blanks and samples may proceed. If, however, any of the same compounds fail again, the extraction/concentration processes are not being performed properly for these compounds. In this event, correct the problem, re-extract the sample lot (Section 10) and repeat the on-going precision and recovery test (Section 12.7).

12.7.4 Add results which pass the specifications in Section 12.7.2 to initial and previous on-going data. Update QC charts to perform a graphic representation of continued laboratory performance (Figure 5). Develop a statement of laboratory accuracy for each pollutant and labeled compound by calculating the average percent recovery (R) and the standard deviation of percent recovery (s_r). Express the accuracy as a recovery interval from R-2s_r to R+2s_r. For example, if R = 95% and s_r = 5%, the accuracy is 85-105%.

13. Qualitative Determination

13.1 Qualititative determination is accomplished by comparison of data from analysis of a sample or blank with data from analysis of the shift standard (Section 12.1) and with data stored in the spectral libraries (Section 7.2.4). Identification is confirmed when spectra and retention times agree per the criteria below.

13.2 Labeled compounds and pollutants having no labeled analog

13.2.1 The signals for all characteristic masses stored in the spectral library (Section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.2.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (one-half to two times) for all masses stored in the library.

13.2.3 The retention time relative to the nearest eluted internal standard shall be within ±15 scans or ±15 seconds, whichever is greater of this difference in the shift standard (Section 12.1).

13.3 Pollutants having a labled analog

13.3.1 The signals for all characteristic masses stored in the spectral library (Section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.3.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

13.3.3 The retention time difference between the pollutant and its labeled analog shall agree within ±6 scans or ±6 seconds (whichever is greater) of this difference in the shift standard (Section 12.1).

13.4 Masses present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the experimental mass spectrum is contaminated, an experienced spectrometrist (Section 1.4) is to determine the presence or absence of the compound.

14. Quantitative Determination

14.1 Isotope dilution—by adding a known amount of a labeled compound to every sample prior to extraction, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon extraction, concentration, and gas chromatography. Relative response (RR) values for mixtures are used in conjunction with calibration curves described in Section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the phenol example given in Figure 1 (Section 7.4.1), RR would be equal to 1.114. For this RR value, the phenol calibration curve given in Figure 1 indicates a concentration of 27 µg/mL in the sample extract (C_{ex}).

14.2 Internal standard—compute the concentration in the extract using the response factor determined from calibration data (Section 7.5) and the following equation:

$$C_{ex}\ (\mu g/L) = \frac{(A_s)\ (C_{is})}{(A_{is})\ (RF)}$$

where:

C_{ex} = the concentration of the compound in the extract and the other terms are as defined in Section 7.5.1.

14.3 The concentration of the pollutant in water is computed using the volumes of the original water sample (Section 10.1) and the final extract volume (Section 10.5), as follows:

$$\text{Concentration in water}\ (\mu g/L) = \frac{(C_{ex})\ (V_{ex})}{V_s}$$

where:

V_{ex} = the extract volume in mL.
V_s = the sample volume in liters.

14.4 If the EICP area at the quantitiation mass for any compound exceeds the calibration range of the system, the extract of the dilute aliquot (Section 10.1) is analyzed by isotope dilution; otherwise, the extract is diluted by a factor of 10, 9 µL of internal standard solution (Section 6.10) are added to a 1.0 mL aliquot, and this diluted extract is analyzed by the internal standard method (Section 14.2). Quantify each compound at the highest concentration level within the calibration range.

14.5 Report results for all pollutants and labeled compounds (Tables 1 and 2) found in all standards, blanks, and samples in µg/L, to three significant figures. Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation mass is within the calibration range (Section 14.4) and the labeled compound recovery is within the normal range for the method (Section 15.4).

15. Analysis of Complex Samples

15.1 Untreated effluents and other samples frequently contain high levels (>1000 µg/L) of the compounds of interest, interfering compounds, and/or polymeric materials. Some samples will not concentrate to 1 mL (Section 10.5); others will overload the GC column and/or mass spectrometer.

15.2 Analyze the dilute aliquot (Section 10.1) when the sample will not concentrate to 1.0 mL. If a dilute aliquot was not extracted, and the sample holding time (Section 9.3) has not been exceeded, dilute an aliquot of the sample with reagent water and re-extract (Section 10.1); otherwise, dilute the extract (Section 14.4) and analyze by the internal standard method (Section 14.2).

15.3 Recovery of internal standard—the EICP area of the internal standard should be within a factor of two of the area in the shift standard (Section 12.1). If the absolute areas of the labeled compounds are within a factor of two of the respective areas in the shift standard, and the internal standard area is less than one-half of its respective area, then internal standard loss in the extract has occurred. In this case, use one of the labeled compounds (perferably a polynuclear aromatic hydrocarbon) to compute the concentration of a pollutant with no labeled analog.

15.4 Recovery of labeled compounds—in most samples, labeled compound recoveries will be similar to those from reagent water (Section 12.7). If the labeled compound recovery is outside the limits given in Table 8, the dilute extract (Section 10.1) is analyzed as in Section 14.4. If the recoveries of all labeled compounds and the internal standard are low (per the criteria above), then a loss in instrument sensitivity is the most likely cause. In this case, the 100 µg/mL calibration standard (Section 12.1) shall be analyzed and calibration verified (Section 12.5). If a loss in sensitivity has occurred, the instrument shall be repaired, the performance specifications in Section 12 shall be met, and the extract reanalyzed. If a loss in instrument sensitivity has not occurred, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes.

16. Method Performance

16.1 Interlaboratory performance for this method is detailed in References 9 and 10.

16.2 A chromatogram of the 100 µg/mL acid/base/neutral calibration standard (Section 6.13) is shown in Figure 6.

References

1. "Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories" USEPA, EMSL/Cincinnati, OH 45268, EPA-600/4-80-025 (April 1980).

2. "Working with Carcinogens," DHEW, PHS, CDC, NIOSH, Publication 77-206, (August 1977).

3. "OSHA Safety and Health Standards, General Industry" OSHA 2206, 29 CFR Part 1910 (January 1976).

4. "Safety in Academic Chemistry Laboratories," ACS Committee on Chemical Safety (1979).

5. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry Systems," J.W. Eichelberger, L.E. Harris, and W.L. Budde. Anal. Chem., *47,* 955 (1975).

6. "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL/Cincinnati, OH 45268, EPA-600/4-79-019 (March 1979).

7. “Standard Practice for Sampling Water,” ASTM Annual Book of Standards, ASTM, Philadelphia, PA, 76 (1980).

8. “Methods 330.4 and 330.5 for Total Residual Chlorine,” USEPA, EMSL/ Cincinnati, OH 45268, EPA 600/4-70-020 (March 1979).

9. Colby, B.N., Beimer, R.G., Rushneck, D.R., and Telliard, W.A. “Isotope Dilution Gas Chromatography-Mass Spectrometry for the determination of Priority Pollutants in Industrial Effluents.” USEPA, Effluent Guidelines Division, Washington, DC 20460 (1980).

10. “Inter-laboratory Validation of US Environmental Protection Agency Method 1625,” USEPA, Effluent Guidelines Division, Washington, DC 20460 (June 15, 1984).

Table 1—Base/Neutral Extractable Compounds

Compound	STORET	CAS registry	EPA-EGD	NPDES
Acenaphthene	34205	83-32-9	001 B	001 B
Acenaphthylene	34200	208-96-8	077 B	002 B
Anthracene	34220	120-12-7	078 B	003 B
Benzidine	39120	92-87-5	005 B	004 B
Benzo(a)anthracene	34526	56-55-3	072 B	005 B
Benzo(b)fluoranthene	34230	205-99-2	074 B	007 B
Benzo(k)fluoranthene	34242	207-08-9	075 B	009 B
Benzo(a)pyrene	34247	50-32-8	073 B	006 B
Benzo(ghi)perylene	34521	191-24-2	079 B	008 B
Biphenyl (Appendix C)	81513	92-52-4	512 B	......
Bis(2-chloroethyl)ether	34273	111-44-4	018 B	011 B
Bis(2-chloroethyoxy)methane	34278	111-91-1	043 B	010 B
Bis(2-chloroisopropyl)ether	34283	108-60-1	042 B	012 B
Bis(2-ethylhexyl)phthalate	39100	117-81-7	066 B	013 B
4-bromophenyl phenyl ether	34636	101-55-3	041 B	014 B
Butyl benzyl phthalate	34292	85-68-7	067 B	015 B
n-C10 (Appendix C)	77427	124-18-5	517 B	......
n-C12 (Appendix C)	77588	112-40-2	506 B	......
n-C14 (Appendix C)	77691	629-59-4	518 B	......
n-C16 (Appendix C)	77757	544-76-3	519 B	......
n-C18 (Appendix C)	77804	593-45-3	520 B	......
n-C20 (Appendix C)	77830	112-95-8	521 B	......
n-C22 (Appendix C)	77859	629-97-0	522 B	......
n-C24 (Appendix C)	77886	646-31-1	523 B	......
n-C26 (Appendix C)	77901	630-01-3	524 B	......
n-C28 (Appendix C)	78116	630-02-4	525 B	......
n-C30 (Appendix C)	78117	638-68-6	526 B	......
Carbazole (4c)	77571	86-74-8	528 B	......
2-chloronaphthalene	34581	91-58-7	020 B	016 B
4-chlorophenyl phenyl ether	34641	7005-72-3	040 B	017 B

Table 1—Base/Neutral Extractable Compounds

Compound	STORET	CAS registry	EPA-EGD	NPDES
Chrysene	34320	218-01-9	076 B	018 B
P-cymene (Appendix C)	77356	99-87-6	513 B	......
Dibenzo(a,h)anthracene	34556	53-70-3	082 B	019 B
Dibenzofuran (Appendix C and 4c)	81302	132-64-9	505 B	......
Dibenzothiophene (Synfuel)	77639	132-65-0	504 B	......
Di-n-butyl phthalate	39110	84-74-2	068 B	026 B
1,2-dichlorobenzene	34536	95-50-1	025 B	020 B
1,3-dichlorobenzene	34566	541-73-1	026 B	021 B
1,4-dichlorobenzene	34571	106-46-7	027 B	022 B
3,3′-dichlorobenzidine	34631	91-94-1	028 B	023 B
Diethyl phthalate	34336	84-66-2	070 B	024 B
2,4-dimethylphenol	34606	105-67-9	034 A	003 A
Dimethyl phthalate	34341	131-11-3	071 B	025 B
2,4-dinitrotoluene	34611	121-14-2	035 B	027 B
2,6-dinitrotoluene	34626	606-20-2	036 B	028 B
Di-n-octyl phthalate	34596	117-84-0	069 B	029 B
Diphenylamine (Appendix C)	77579	122-39-4	507 B	......
Diphenyl ether (Appendix C)	77587	101-84-8	508 B	......
1,2-diphenylhydrazine	34346	122-66-7	037 B	030 B
Fluoranthene	34376	206-44-0	039 B	031 B
Fluorene	34381	86-73-7	080 B	032 B
Hexachlorobenzene	39700	118-74-1	009 B	033 B
Hexachlorobutadiene	34391	87-68-3	052 B	034 B
Hexachloroethane	34396	67-72-1	012 B	036 B
Hexachlorocyclopentadiene	34386	77-47-4	053 B	035 B
Indeno(1,2,3-cd)pyrene	34403	193-39-5	083 B	037 B
Isophorone	34408	78-59-1	054 B	038 B
Naphthalene	34696	91-20-3	055 B	039 B
B-naphthylamine (Appendix C)	82553	91-59-8	502 B	......
Nitrobenzene	34447	98-95-3	056 B	040 B
N-nitrosodimethylamine	34438	62-75-9	061 B	041 B
N-nitrosodi-n-propylamine	34428	621-64-7	063 B	042 B
N-nitrosodiphenylamine	34433	86-30-3	062 B	043 B
Phenanthrene	34461	85-01-8	081 B	044 B
Phenol	34694	108-95-2	065 A	010 A
a-Picoline (Synfuel)	77088	109-06-89	503 B	......
Pyrene	34469	129-00-0	084 B	045 B
styrene (Appendix C)	77128	100-42-5	510 B	......
a-terpineol (Appendix C)	77493	98-55-5	509 B	......
1,2,3-trichlorobenzene (4c)	77613	87-61-6	529 B	......
1,2,4-trichlorobenzene	34551	120-82-1	008 B	046 B

Table 2—Acid Extractable Compounds

Compound	STORET	CAS registry	EPA-EGD	NPDES
4-chloro-3-methylphenol	34452	59-50-7	022 A	008 A
2-chlorophenol	34586	95-57-8	024 A	001 A
2,4-dichlorophenol	34601	120-83-2	031 A	002 A
2,4-dinitrophenol	34616	51-28-5	059 A	005 A
2-methyl-4,6-dinitrophenol	34657	534-52-1	060 A	004 A
2-nitrophenol	34591	88-75-5	057 A	006 A
4-nitrophenol	34646	100-02-7	058 A	007 A
Pentachlorophenol	39032	87-86-5	064 A	009 A
2,3,6-trichlorophenol (4c)	77688	93-37-55	530 A	......
2,4,5-trichlorophenol (4c)	......	95-95-4	531 A	......
2,4,6-trichlorophenol	34621	88-06-2	021 A	011 A

Table 3—Gas Chromatography of Base/Neutral Extractable Compounds

EGD No.[1]	Compound	Retention time			Detection limit[2] (µg/L)
		Mean (sec)	EGD Ref	Relative	
164	2,2′-difluorobiphenyl (int std)	1163	164	1.000-1.000	10
061	N-nitrosodimethylamine	385	164	ns	50
603	alpha picoline-d7	417	164	0.326-0.393	50
703	alpha picoline	426	603	1.006-1.028	50
610	styrene-d5	546	164	0.450-0.488	10
710	styrene	549	610	1.002-1.009	10
613	p-cymene-d14	742	164	0.624-0.652	10
713	p-cymene	755	613	1.008-1.023	10
265	phenol-d5	696	164	0.584-0.613	10
365	phenol	700	265	0.995-1.010	10
218	bis(2-chloroethyl)ether-d8	696	164	0.584-0.607	10
318	bis(2-chloroethyl)ether	704	218	1.007-1.016	10
617	n-decane-d22	698	164	0.585-0.615	10
717	n-decane	720	617	1.022-1.038	10
226	1,3-dichlorobenzene-d4	722	164	0.605-0.636	10
326	1,3-dichlorobenzene	724	226	0.998-1.008	10
227	1,4-dichlorobenzene-d4	737	164	0.601-0.666	10
327	1,4-dichlorobenzene	740	227	0.997-1.009	10
225	1,2-dichlorobenzene-d4	758	164	0.632-0.667	10
325	1,2-dichlorobenzene	760	225	0.995-1.008	10
242	bis(2-chloroisopropyl)ether-d12	788	164	0.664-0.691	10
342	bis(2-chloroisopropyl)ether	799	242	1.010-1.016	10
212	hexachloroethane-13C	819	164	0.690-0.717	10
312	hexachloroethane	823	212	0.999-1.001	10
063	N-nitrosodi-n-propylamine	830	164	ns	20
256	nitrobenzene-d5	845	164	0.706-0.727	10

Table 3—Gas Chromatography of Base/Neutral Extractable Compounds

EGD No.[1]	Compound	Retention time			Detection limit[2] (µg/L)
		Mean (sec)	EGD Ref	Relative	
356	nitrobenzene	849	256	1.002-1.007	10
254	isophorone-d8	881	164	0.747-0.767	10
354	isophorone	889	254	0.999-1.017	10
234	2,4-dimethyl phenol-d3	921	164	0.781-0.803	10
334	2,4-dimethylphenol	924	234	0.999-1.003	10
043	bis(2-chloroethoxy)methane	939	164	ns	10
208	1,2,4-trichlorobenzene-d3	955	164	0.813-0.830	10
308	1,2,4-trichlorobenzene	958	208	1.000-1.005	10
255	naphthalene-d8	963	164	0.819-0.836	10
355	naphthalene	967	255	1.001-1.006	10
609	alpha-terpineol-d3	973	164	0.829-0.844	10
709	alpha-terpineol	975	609	0.998-1.008	10
606	n-dodecane-d26	953	164	0.730-0.908	10
706	n-dodecane	981	606	0.986-1.051	10
529	1,2,3-trichlorobenzene	1003	164	ns	10
252	hexachlorobutadiene-13C4	1005	164	0.856-0.871	10
352	hexachlorobutadiene	1006	252	0.999-1.002	10
253	hexachlorocyclopentadiene-13C4	1147	164	0.976-0.986	10
353	hexachlorocyclopentadiene	1142	253	0.999-1.001	10
220	2-chloronaphthalene-d7	1185	164	1.014-1.024	10
320	2-chloronaphthalene	1200	220	0.997-1.007	10
518	n-tetradecane	1203	164	ns	10
612	Biphenyl-d10	1205	164	1.016-1.027	10
712	Biphenyl	1195	612	1.001-1.006	10
608	Diphenyl ether-d10	1211	164	1.036-1.047	10
708	Diphenyl ether	1216	608	0.997-1.009	10
277	Acenaphthylene-d8	1265	164	1.080-1.095	10
377	Acenaphthylene	1247	277	1.000-1.004	10
271	Dimethyl phthalate-d4	1269	164	1.083-1.102	10
371	Dimethyl phthalate	1273	271	0.998-1.005	10
236	2,6-dinitrotoluene-d3	1283	164	1.090-1.112	10
336	2,6-dinitrotoluene	1300	236	1.001-1.005	10
201	Acenaphthene-d10	1298	164	1.107-1.125	10
301	Acenaphthene	1304	201	0.999-1.009	10
605	Dibenzofuran-d8	1331	164	1.134-1.155	10
705	Dibenzofuran	1335	605	0.998-1.007	10
602	Beta-naphthylamine-d7	1368	164	1.163-1.189	50
702	Beta-naphthylamine	1371	602	0.996-1.007	50
280	Fluorene-d10	1395	164	1.185-1.214	10
380	Fluorene	1401	281	0.999-1.008	10
240	4-chlorophenyl phenyl ether-d5	1406	164	1.194-1.223	10
340	4-chlorophenyl phenyl ether	1409	240	0.990-1.015	10
270	Diethyl phthalate-d4	1409	164	1.197-1.229	10
370	Diethyl phthalate	1414	270	0.996-1.006	10

Table 3—Gas Chromatography of Base/Neutral Extractable Compounds

EGD No.[1]	Compound	Retention time			Detection limit[2] (µg/L)
		Mean (sec)	EGD Ref	Relative	
619	n-hexadecane-d34	1447	164	1.010-1.478	10
719	n-hexadecane	1469	619	1.013-1.020	10
235	2,4-dinitrotoluene-d3	1359	164	1.152-1.181	10
335	2,4-dinitrotoluene	1344	235	1.000-1.002	10
237	1,2-diphenylhydrazine-d8	1433	164	1.216-1.248	20
337	1,2-diphenylhydrazine[3]	1439	237	0.999-1.009	20
607	Diphenylamine-d10	1437	164	1.213-1.249	20
707	Diphenylamine	1439	607	1.000-1.007	20
262	N-nitrosodiphenylamine-d6	1447	164	1.225-1.252	20
362	N-nitrosodiphenylamine[4]	1464	262	1.000-1.002	20
041	4-bromophenyl phenyl ether	1498	164	1.271-1.307	10
209	Hexachlorobenzene-13C6	1521	164	1.288-1.327	10
309	Hexachlorobenzene	1522	209	0.999-1.001	10
281	Phenanthrene-d10	1578	164	1.334-1.380	10
520	n-octadecane	1580	164	ns	10
381	Phenanthrene	1583	281	1.000-1.005	10
278	Anthracene-d10	1588	164	1.342-1.388	10
378	Anthracene	1592	278	0.998-1.006	10
604	Dibenzothiophene-d8	1559	164	1.314-1.361	10
704	Dibenzothiophene	1564	604	1.000-1.006	10
528	Carbazole	1650	164	ns	20
621	n-eicosane-d42	1655	164	1.184-1.662	10
721	n-eicosane	1677	621	1.010-1.021	10
268	Di-n-butyl phthalate-d4	1719	164	1.446-1.510	10
368	Di-n-butyl phthalate	1723	268	1.000-1.003	10
239	Fluoranthene-d10	1813	164	1.522-1.596	10
339	Fluoranthene	1817	239	1.000-1.004	10
284	Pyrene-d10	1844	164	1.523-1.644	10
384	Pyrene	1852	284	1.001-1.003	10
205	Benzidine-d8	1854	164	1.549-1.632	50
305	Benzidine	1853	205	1.000-1.002	50
522	n-docosane	1889	164	ns	10
623	n-tetracosane-d50	1997	164	1.671-1.764	10
723	n-tetracosane	2025	612	1.012-1.015	10
067	Butylbenzyl phthalate	2060	164	ns	10
276	Chrysene-d12	2081	164	1.743-1.837	10
376	Chrysene	2083	276	1.000-1.004	10
272	Benzo(a)anthracene-d12	2082	164	1.735-1.846	10
372	Benzo(a)anthracene	2090	272	0.999-1.007	10
228	3,3′-dichlorobenzidine-d6	2088	164	1.744-1.848	50
328	3,3′-dichlorobenzidine	2086	228	1.000-1.001	50
266	Bis(2-ethylhexyl)phthalate-d4	2123	164	1.771-1.880	10
366	Bis(2-ethylhexyl)phthalate	2124	266	1.000-1.002	10
524	n-hexacosane	2147	164	ns	10

Table 3—Gas Chromatography of Base/Neutral Extractable Compounds

EGD No.[1]	Compound	Retention time			Detection limit[2] (µg/L)
		Mean (sec)	EGD Ref	Relative	
269	di-n-octyl phthalate-d4	2239	164	1.867-1.982	10
369	di-n-octyl phthalate	2240	269	1.000-1.002	10
525	n-octacosane	2272	164	ns	10
274	Benzo(b)fluoranthene-d12	2281	164	1.902-2.025	10
354	Benzo(b)fluoranthene	2293	274	1.000-1.005	10
275	Benzo(k)fluoranthene-d12	2287	164	1.906-2.033	10
375	Benzo(k)fluoranthene	2293	275	1.000-1.005	10
273	Benzo(a)pyrene-d12	2351	164	1.954-2.088	10
373	Benzo(a)pyrene	2350	273	1.000-1.004	10
626	N-triacontane-d62	2384	164	1.972-2.127	10
726	N-triacontane	2429	626	1.011-1.028	10
083	Indeno(1,2,3-cd)pyrene	2650	164	ns	20
082	Dibenzo(a,h)anthracene	2660	164	ns	20
279	Benzo(ghi)perylene-d12	2741	164	2.187-2.524	20
379	Benzo(ghi)perylene	2750	279	1.001-1.006	20

[1]Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
[2]This is a minimum level at which the entire GC/MS system must give recognizable mass spectra (background corrected) and acceptable calibration points.
[3]Detected as azobenzene.
[4]Detected as diphenylamine.
ns = specification not available at time of release of method.
Column: 30 ±2 m x 0.25 ±0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary.
Temperature program: five minutes at 30°C; 30-280°C at 8°C per minute; isothermal at 280°C until benzo(ghi)perylene elutes.
Gas velocity: 30 ±5 cm/sec.

Table 4—Gas Chromatography of Acid Extractable Compounds

EGD No.[1]	Compound	Retention time: Mean (sec)	EGD Ref	Relative	Detection limit[2] (µg/L)
164	2,2′-difluorobiphenyl (int std)	1163	164	1.000-1.000	10
224	2-chlorophenol-d4	701	164	0.587-0.618	10
324	2-chlorophenol	705	224	0.997-1.010	10
257	2-nitrophenol-d4	898	164	0.761-0.783	20
357	2-nitrophenol	900	257	0.994-1.009	20
231	2,4-dichlorophenol-d3	944	164	0.802-0.822	10
331	2,4-dichlorophenol	947	231	0.997-1.006	10
222	4-chloro-3-methylphenol-d2	1086	164	0.930-0.943	10
322	4-chloro-3-methylphenol	1091	222	0.998-1.003	10
221	2,4,6-trichlorophenol-d2	1162	164	0.994-1.005	10
321	2,4,6-trichlorophenol	1165	221	0.998-1.004	10
531	2,4,5-trichlorophenol	1170	164	ns	10
530	2,3,6-trichlorophenol	1195	164	ns	10
259	2,4-dinitrophenol-d3	1323	164	1.127-1.149	50
359	2,4-dinitrophenol	1325	259	1.000-1.005	50
258	4-nitrophenol-d4	1349	164	1.147-1.175	50
358	4-nitrophenol	1354	258	0.997-1.006	50
260	2-methyl-4,6-dinitrophenol-d2	1433	164	1.216-1.249	20
360	2-methyl-4,6-dinitrophenol	1435	260	1.000-1.002	20
264	Pentachlorophenol-13C6	1559	164	1.320-1.363	50
364	Pentachlorophenol	1561	264	0.998-1.002	50

[1]Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
[2]This is a minimum level at which the entire GC/MS system must give recognizable mass spectra (background corrected) and acceptable calibration points.
ns = specification not available at time of release of method.
Column: 30 ±2m x 0.25 ±0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary.
Temperature program: five minutes at 30°C; 8°C/min to 250°C or until pentachlorophenol elutes.
Gas velocity: 30 ±5 cm/sec.

Table 5—DFTPP Mass Intensity Specifications

Mass	Intensity required
51	30-60 percent of mass 198.
68	Less than 2 percent of mass 69.
70	Less than 2 percent of mass 69.
127	40-60 percent of mass 198.
197	Less than 1 percent of mass 198.
199	5-9 percent of mass 198.
275	10-30 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present and less than mass 443.
442	40-100 percent of mass 198.
443	17-23 percent of mass 442.

Table 6—Base/Neutral Extractable Compound Characteristic Masses

Compound	Labeled analog	Primary m/z
Acenaphthene	d10	154/164
Acenaphthylene	d8	152/160
Anthracene	d10	178/188
Benzidine	d8	184/192
Benzo(a)anthracene	d12	228/240
Benzo(b)fluoranthene	d12	252/264
Benzo(k)fluoranthene	d12	252/264
Benzo(a)pyrene	d12	252/264
Benzo(ghi)perylene	d12	276/288
Biphenyl	d10	154/164
Bis(2-chloroethyl)ether	d8	93/101
Bis(2-chloroethoxy)methane	......	93
Bis(2-chloroisopropyl)ether	d12	121/131
Bis(2-ethylhexyl)phthalate	d4	149/153
4-bromophenyl phenyl ether	......	248
Butyl benzyl phthalate	......	149
n-C10	d22	55/66
n-C12	d26	55/66
n-C14	......	55
n-C16	d34	55/66
n-C18	......	55
n-C20	d42	55/66
n-C22	......	55
n-C24	d50	55/66
n-C26	......	55
n-C28	......	55
n-C30	d62	55/66
Carbazole	d8	167/175
2-chloronaphthalene	d7	162/169

Table 6—Base/Neutral Extractable Compound Characteristic Masses

Compound	Labeled analog	Primary m/z
4-chlorophenyl phenyl ether	d5	204/209
Chrysene	d12	228/240
p-cymene	d14	114/130
Dibenzo(a,h)anthracene	......	278
Dibenzofuran	d8	168/176
Dibenzothiophene	d8	184/192
Di-n-butyl phthalate	d4	149/153
1,2-dichlorobenzene	d4	146/152
1,3-dichlorobenzene	d4	146/152
1,4-dichlorobenzene	d4	146/152
3,3′-dichlorobenzidine	d6	252/258
Diethyl phthalate	d4	149/153
2,4-dimethylphenol	d3	122/125
Dimethyl phthalate	d4	163/167
2,4-dinitrotoluene	d3	164/168
2,6-dinitrotoluene	d3	165/167
Di-n-octyl phthalate	d4	149/153
Diphenylamine	d10	169/179
Diphenyl ether	d10	170/180
1,2-diphenylhydrazine[1]	d10	77/82
Fluoranthene	d10	202/212
Fluorene	d10	166/176
Hexachlorobenzene	13C6	284/292
Hexachlorobutadiene	13C4	225/231
Hexachloroethane	13C	201/204
Hexachlorocyclopentadiene	13C4	237/241
Ideno(1,2,3-cd)pyrene	......	276
Isophorone	d8	82/88
Naphthalene	d8	128/136
B-naphthylamine	d7	143/150
Nitrobenzene	d5	123/128
N-nitrosodimethylamine	......	74
N-nitrosodi-n-propylamine	......	70
N-nitrosodiphenylamile[2]	d6	169/175
Phenanthrene	d10	178/188
Phenol	d5	94/71
a-picoline	d7	93/100
Pyrene	d10	202/212
Styrene	d5	104/109
a-terpineol	d3	59/62
1,2,3-trichlorobenzene	d3	180/183
1,2,4-trichlorobenzene	d3	180/183

[1]Detected as azobenzene.
[2]Detected as diphenylamine.

Table 7—Acid Extractable Compound Characteristic Masses

Compound	Labeled analog	Primary m/z
4-chloro-3-methylphenol	d2	107/109
2-chlorophenol	d4	128/132
2,4-dichlorophenol	d3	162/167
2,4-dinitrophenol	d3	184/187
2-methyl-4,6-dinitrophenol	d2	198/200
2-nitrophenol	d4	139/143
4-nitrophenol	d4	139/143
Pentachlorophenol	13C6	266/272
2,3,6-trichlorophenol	d2	196/200
2,4,5-trichlorophenol	d2	196/200
2,4,6-trichlorophenol	d2	196/200

Table 8—Acceptance Criteria for Performance Tests

EGD No.[1]	Compound	Acceptance criteria				
		Initial precision and accuracy, Section 8.2.3		Labeled compound recovery, Sections 8.3 and 14.2 P (percent)	Calibration verification, Section 12.5 (µg/L)	On-going accuracy, Section 11.6 R (µg/L)
		s	X			
301	Acenaphthene	21	79-134	......	80-125	72-144
201	Acenaphthene-d10	38	38-147	20-270	71-141	30-180
377	Acenaphtylene	38	69-186	......	60-166	61-207
277	Acenaphthylene-d8	31	38-146	23-239	66-152	33-168
378	Anthracene	41	58-174	......	60-168	50-199
278	Anthracene-d10	49	31-194	14-419	58-171	23-242
305	Benzidine	119	16-518	......	34-296	11-672
205	Benzidine-d8	269	ns-ns	ns-ns	ns-ns	ns-ns
372	Benzo(a)anthracene	20	65-168	......	70-142	62-176
272	Benzo(a)anthracene-d12	41	25-298	12-605	28-357	22-329
374	Benzo(b)fluoranthene	183	32-545	......	61-164	20-ns
274	Benzo(b)fluoranthene-d12	168	11-577	ns-ns	14-ns	ns-ns
375	Benzo(k)fluoranthene	26	59-143	......	13-ns	53-155
275	Benzo(k)fluoranthene-d12	114	15-514	ns-ns	13-ns	ns-685
373	Benzo(a)pyrene	26	62-195	......	78-129	59-206
273	Benzo(a)pyrene-d12	24	35-181	21-290	12-ns	32-194
379	Benzo(ghi)perylene	21	72-160	......	69-145	58-168
279	Benzo(ghi)perylene-d12	45	29-268	14-529	13-ns	25-303
712	Biphenyl (Appendix C)	41	75-148	......	58-171	62-176
612	Biphenyl-d12	43	28-165	ns-ns	52-192	17-267
318	Bis(2-chloroethyl)ether	34	55-196	......	61-164	50-213
218	Bis(2-chloroethyl)ether-d8	33	29-196	15-372	52-194	25-222

Table 8—Acceptance Criteria for Performance Tests

EGD No.[1]	Compound	Acceptance criteria: Initial precision and accuracy, Section 8.2.3 — s	Acceptance criteria: Initial precision and accuracy, Section 8.2.3 — X	Labeled compound recovery, Sections 8.3 and 14.2 P (percent)	Calibration verification, Section 12.5 (µg/L)	On-going accuracy, Section 11.6 R (µg/L)
043	Bis(2-chloroethoxy)methane*	27	43-153	......	44-228	39-166
342	Bis(2-chloroisopropyl)ether	17	81-138	......	67-148	77-145
242	Bis(2-chloroisopropyl) ether-d12	27	35-149	20-260	44-229	30-169
366	Bis(2-ethylhexyl)phthalate	31	69-220	......	76-131	64-232
266	Bis(2-ethylhexyl)phthalate-d4	29	32-205	18-364	43-232	28-224
041	4-bromophenyl phenyl ether*	44	44-140	......	52-193	35-172
067	Butyl benzyl phthalate*	31	19-233	......	22-450	35-170
717	n-C10 (Appendix C)	51	24-195	......	42-235	19-237
617	n-C10-d22	70	ns-298	ns-ns	44-227	ns-504
706	n-C12 (Appendix C)	74	35-369	......	60-166	29-424
606	n-C12-d26	53	ns-331	ns-ns	41-242	ns-408
518	n-C14 (Appendix C)*	109	ns-985	......	37-268	ns-ns
719	n-C16 (Appendix C)	33	80-162	......	72-138	71-181
619	n-C16-d34	46	37-162	18-308	54-186	28-202
520	n-C18 (Appendix C)*	39	42-131	......	40-249	35-167
721	n-C20 (Appendix C)	59	53-263	......	54-184	46-301
621	n-C20-d42	34	34-172	19-306	62-162	29-198
522	n-C22 (Appendix C)*	31	45-152	......	40-249	39-195
723	n-C24 (Appendix C)	11	80-139	......	65-154	78-142
623	n-C24-d50	28	27-211	15-376	50-199	25-229
524	n-C26 (Appendix C)*	35	35-193	......	26-392	31-212
525	n-C28 (Appendix C)*	35	35-193	......	26-392	31-212
726	n-C30 (Appendix C)	32	61-200	......	66-152	56-215
626	n-C30-d62	41	27-242	13-479	24-423	23-274
528	Carbazole (4c)*	38	36-165	......	44-227	31-188
320	2-chloronaphthalene	100	46-357	......	58-171	35-442
220	2-chloronaphthalene-d7	41	30-168	15-324	72-139	24-204
322	4-chloro-3-methylphenol	37	76-131	......	85-115	62-159
222	4-chloro-3-methylphenol-d2	111	30-174	ns-613	68-147	14-314
324	2-chlorophenol	13	79-135	......	78-129	76-138
224	2-chlorophenol-d4	24	36-162	23-255	55-180	33-176
340	4-chlorophenyl phenyl ether	42	75-166	......	71-142	63-194
240	4-chlorophenyl phenyl ether-d5	52	40-161	19-325	57-175	29-212
376	Chrysene	51	59-186	......	70-142	48-221
276	Chrysene-d12	69	33-219	13-512	24-411	23-290
713	p-cymene (Appendix C)	18	76-140	......	79-127	72-147
613	p-cymene-d14	67	ns-359	ns-ns	66-152	ns-468
082	Dibenzo(a,h)anthracene*	55	23-299	......	13-761	19-340

Table 8—Acceptance Criteria for Performance Tests

EGD No.[1]	Compound	Acceptance criteria: Initial precision and accuracy, Section 8.2.3 — s	Acceptance criteria: Initial precision and accuracy, Section 8.2.3 — X	Labeled compound recovery, Sections 8.3 and 14.2 P (percent)	Calibration verification, Section 12.5 (µg/L)	On-going accuracy, Section 11.6 R (µg/L)
705	Dibenzofuran (Appendix C)	20	85-136	......	73-136	79-146
605	Dibenzofuran-d8	31	47-136	28-220	66-150	39-160
704	Dibenzothiophene (Synfuel)	31	79-150	......	72-140	70-168
604	Dibenzothiophene-d8	31	48-130	29-215	69-145	40-156
368	Di-n-butyl phthalate	15	76-165	......	71-142	74-169
268	Di-n-butyl phthalate-d4	23	23-195	13-346	52-192	22-209
325	1,2-dichlorobenzene	17	73-146	......	74-135	70-152
225	1,2-dichlorobenzene-d4	35	14-212	ns-494	61-164	11-247
326	1,3-dichlorobenzene	43	63-201	......	65-154	55-225
226	1,3-dichlorobenzene-d4	48	13-203	ns-550	52-192	ns-260
327	1,4-dichlorobenzene	42	61-194	......	62-161	53-219
227	1,4-dichlorobenzene-d4	48	15-193	ns-474	65-153	11-245
328	3,3′-dichlorobenzidine	26	68-174	......	77-130	64-185
228	3,3′-dichlorobenzidine-d6	80	ns-562	ns-ns	18-558	ns-ns
331	2,4-dichlorophenol	12	85-131	......	67-149	83-135
231	2,4-dichlorophenol-d3	28	38-164	24-260	64-157	34-182
370	Diethyl phthalate	44	75-196	......	74-135	65-222
270	Diethyl phthalate-d4	78	ns-260	ns-ns	47-211	ns-ns
334	2,4-dimethylphenol	13	62-153	......	67-150	60-156
234	2,4-dimethylphenol-d3	22	15-228	ns-449	58-172	14-242
371	Dimethyl phthalate	36	74-188	......	73-137	67-207
271	Dimethyl phthalate-d4	108	ns-640	ns-ns	50-201	ns-ns
359	2,4-dinitrophenol	18	72-134	......	75-133	68-141
259	2,4-dinitrophenol-d3	66	22-308	ns-ns	39-256	17-378
335	2,4-dinitrotoluene	18	75-158	......	79-127	72-164
235	2,4-dinitrotoluene-d3	37	22-245	10-514	53-187	19-275
336	2,6-dinitrotoluene	30	80-141	......	55-183	70-159
236	2,6-dinitrotoluene-d3	59	44-184	17-442	36-278	31-250
369	Di-n-octyl phthalate	16	77-161	......	71-140	74-166
269	Di-n-octyl phthalate-d4	46	12-383	ns-ns	21-467	10-433
707	Diphenylamine (Appendix C)	45	58-205	......	57-176	51-231
607	Diphenylamine-d10	42	27-206	11-488	59-169	21-249
708	Diphenyl ether (Appendix C)	19	82-136	......	83-120	77-144
608	Diphenyl ether-d10	37	36-155	19-281	77-129	29-186
337	1,2-diphenylhydrazine	73	49-308	......	75-134	40-360
237	1,2-diphenylhydrazine-d10	35	31-173	17-316	58-174	26-200
339	Fluoranthene	33	71-177	......	67-149	64-194
239	Fluoranthene-d10	35	36-161	20-278	47-215	30-187
380	Fluorene	29	81-132	......	74-135	70-151
280	Fluorene-d10	43	51-131	27-238	61-164	38-172

Table 8—Acceptance Criteria for Performance Tests

EGD No.[1]	Compound	Acceptance criteria: Initial precision and accuracy, Section 8.2.3 — s	X	Labeled compound recovery, Sections 8.3 and 14.2 P (percent)	Calibration verification, Section 12.5 (µg/L)	On-going accuracy, Section 11.6 R (µg/L)
309	Hexachlorobenzene	16	90-124	......	78-128	85-132
209	Hexachlorobenzene-13C6	81	36-228	13-595	38-265	23-321
352	Hexachlorobutadiene	56	51-251	......	74-135	43-287
252	Hexachlorobutadiene-13C4	63	ns-316	ns-ns	68-148	ns-413
312	Hexachloroethane	227	21-ns	......	71-141	13-ns
212	Hexachloroethane-13C1	77	ns-400	ns-ns	47-212	ns-563
353	Hexachlorocyclopentadiene	15	69-144	......	77-129	67-148
253	Hexachlorocyclo-pentadiene-13C4	60	ns-ns	ns-ns	47-211	ns-ns
083	Ideno(1,2,3-cd)pyrene*	55	23-299	......	13-761	19-340
354	Isophorone	25	76-156	......	70-142	70-168
254	Isophorone-d8	23	49-133	33-193	52-194	44-147
360	2-methyl-4,6-dinitrophenol	19	77-133	......	69-145	72-142
260	2-methyl-4,6-dinitrophenol-d2	64	36-247	16-527	56-177	28-307
355	Naphthalene	20	80-139	......	73-137	75-149
255	Naphthalene-d8	39	28-157	14-305	71-141	22-192
702	B-naphthylamine (Appendix C)	49	10-ns	......	39-256	ns-ns
602	B-naphthylamine-d7	33	ns-ns	ns-ns	44-230	ns-ns
356	Nitrobenzene	25	69-161	......	85-115	65-169
256	Nitrobenzene-d5	28	18-265	ns-ns	46-219	15-314
357	2-nitrophenol	15	78-140	......	77-129	75-145
257	2-nitrophenol-d4	23	41-145	27-217	61-163	37-158
358	4-nitrophenol	42	62-146	......	55-183	51-175
258	4-nitrophenol-d4	188	14-398	ns-ns	35-287	ns-ns
061	N-nitrosodimethylamile*	198	21-472	......	40-249	12-807
063	N-nitrosodi-n-proplyamine*	198	21-472	......	40-249	12-807
362	N-nitrosodiphenylamine	45	65-142	......	68-148	53-173
262	N-nitrosodiphenylamine-d6	37	54-126	26-256	59-170	40-166
364	Pentachlorophenol	21	76-140	......	77-130	71-150
264	Pentachlorophenol-13C6	49	37-212	18-412	42-237	29-254
381	Phenanthrene	13	93-119	......	75-133	87-126
281	Phenanthrene-d10	40	45-130	24-241	67-149	34-168
365	Phenol	36	77-127	......	65-155	62-154
265	Phenol-d5	161	21-210	ns-ns	48-208	ns-ns
703	a-picoline (Synfuel)	38	59-149	......	60-165	50-174
603	a-picoline-d7	138	11-380	ns-ns	31-324	ns-608
384	Pyrene	19	76-152	......	76-132	72-159
284	Pyrene-d10	29	32-176	18-303	48-210	28-196

Table 8—Acceptance Criteria for Performance Tests

EGD No.[1]	Compound	Acceptance criteria				
		Initial precision and accuracy, Section 8.2.3		Labeled compound recovery, Sections 8.3 and 14.2 P (percent)	Calibration verification, Section 12.5 (µg/L)	On-going accuracy, Section 11.6 R (µg/L)
		s	X			
710	Styrene (Appendix C)	42	53-221	......	65-153	48-244
610	Styrene-d5	49	ns-281	ns-ns	44-228	ns-348
709	a-terpineol (Appendix C)	44	42-234	......	54-186	38-258
609	a-terpineol-d3	48	22-292	ns-672	20-502	18-339
529	1,2,3-trichlorobenzene (4c)*	69	15-229	......	60-167	11-297
308	1,2,4-trichlorobenzene	19	82-136	......	78-128	77-144
208	1,2,4-trichlorobenzene-d3	57	15-212	ns-592	61-163	10-282
530	2,3,6-trichlorophenol (4c)*	30	58-137	......	56-180	51-153
531	2,4,5-trichlorophenol (4c)*	30	58-137	......	56-180	51-153
321	2,4,6-trichlorophenol	57	59-205	......	81-123	48-244
221	2,4,6-trichlorophenol-d2	47	43-183	21-363	69-144	34-226

[1]Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.

*Measured by internal standard; specification derived from related compound.

ns = no specification; limit is outside the range that can be measured reliably.

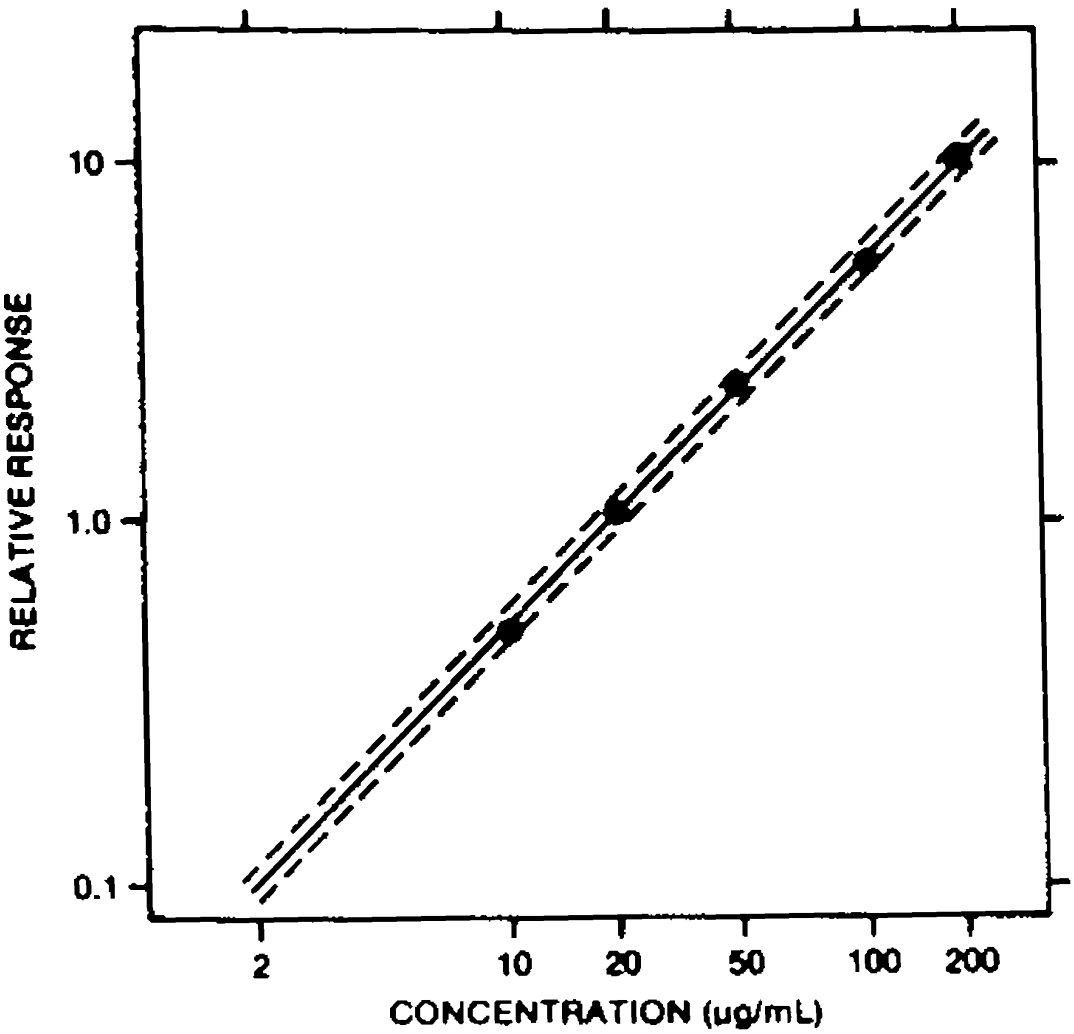

FIGURE 1 Relative Response Calibration Curve for Phenol. The Dotted Lines Enclose a ±10 Percent Error Window.

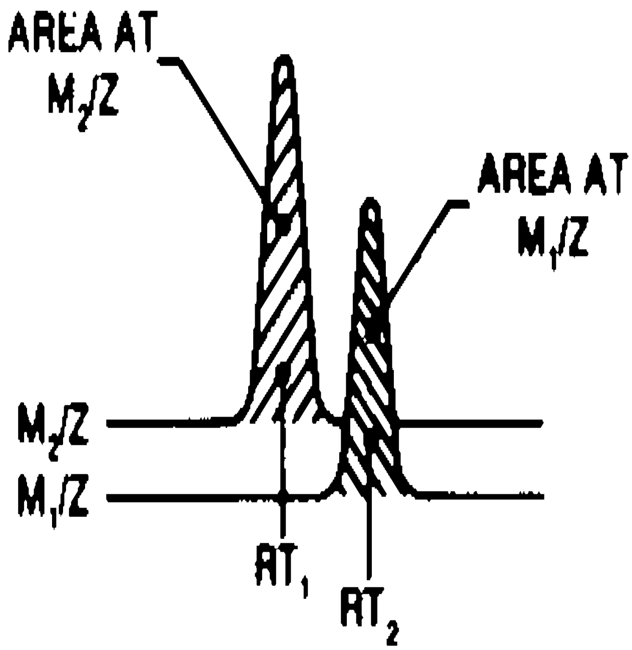

FIGURE 2 Extracted Ion Current Profiles for Chromatographically Resolved Labeled (m_2/z) and Unlabeled (m_1/z) Pairs.

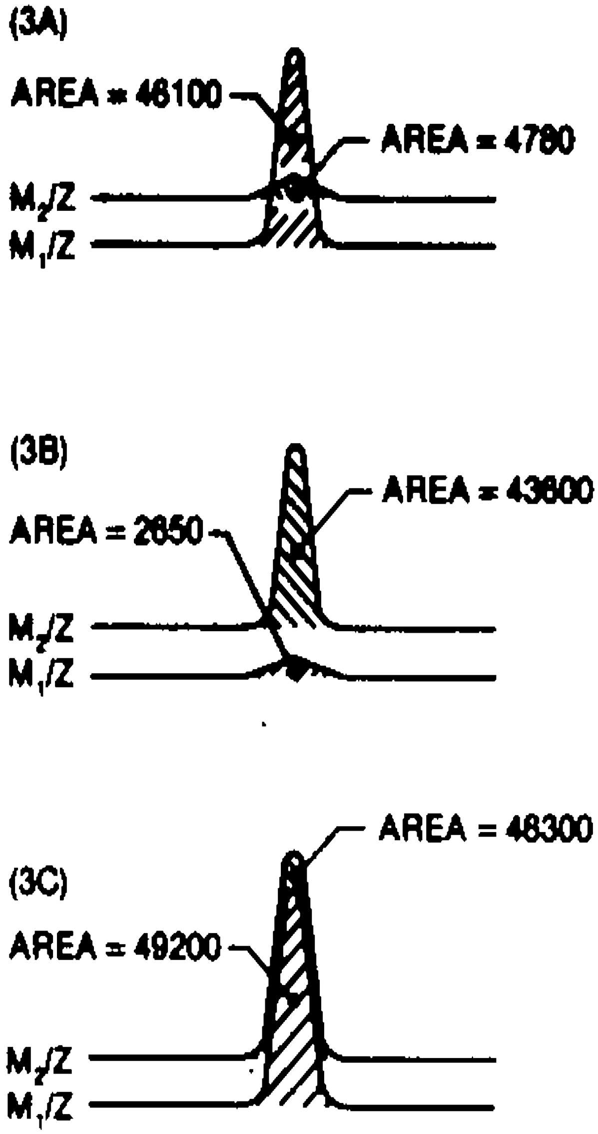

FIGURE 3 Extracted Ion Current Profiles for (3A) Unlabeled Compound, (3B) Labeled Compound, and (3C) Equal Mixture of Unlabeled and Labeled Compounds.

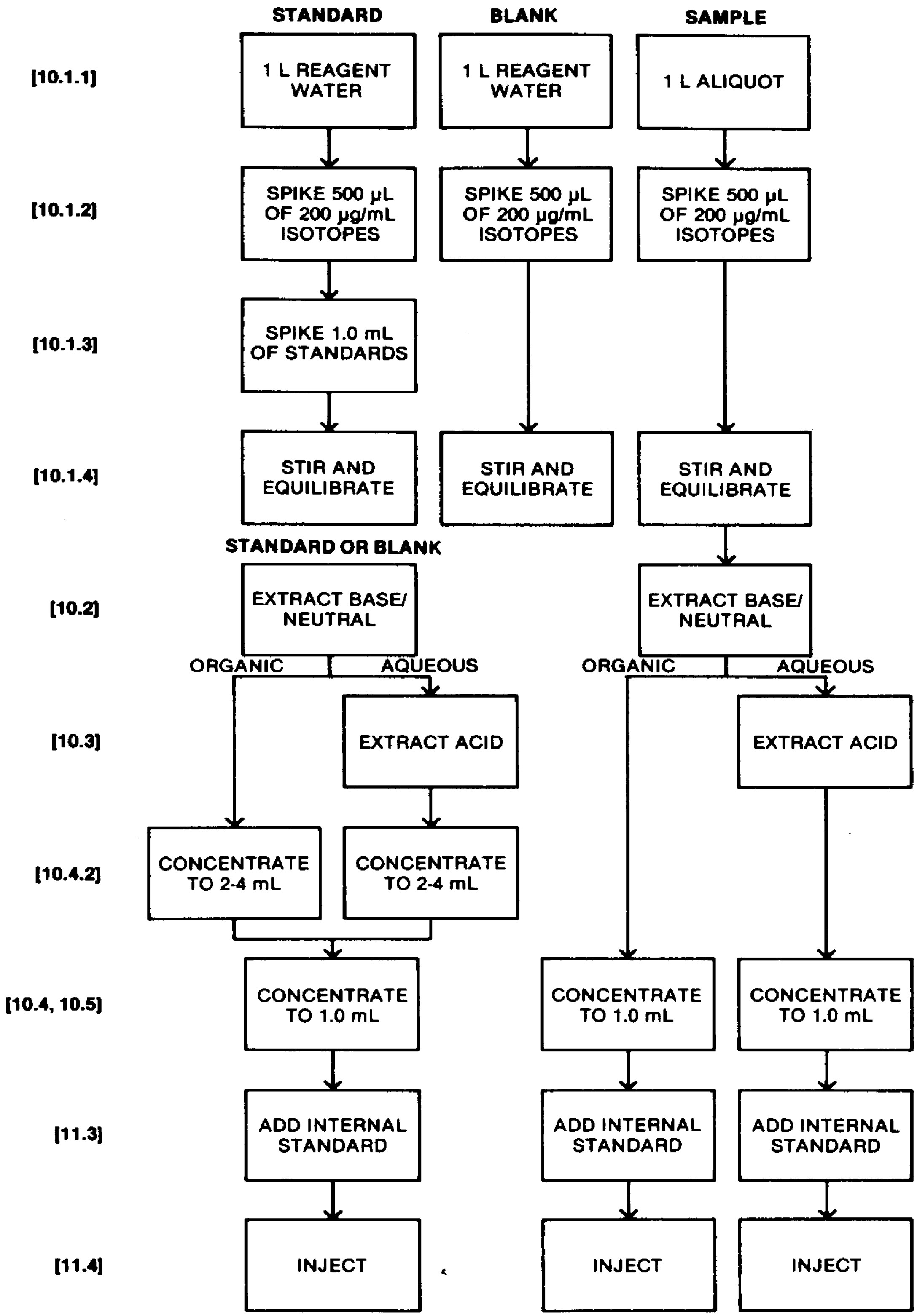

FIGURE 4 Flow Chart for Extraction/Concentration of Precision and Recovery Standard, Blank, and Sample by Method 1625. Numbers in Brackets [ ] Refer to Section Numbers in the Method.

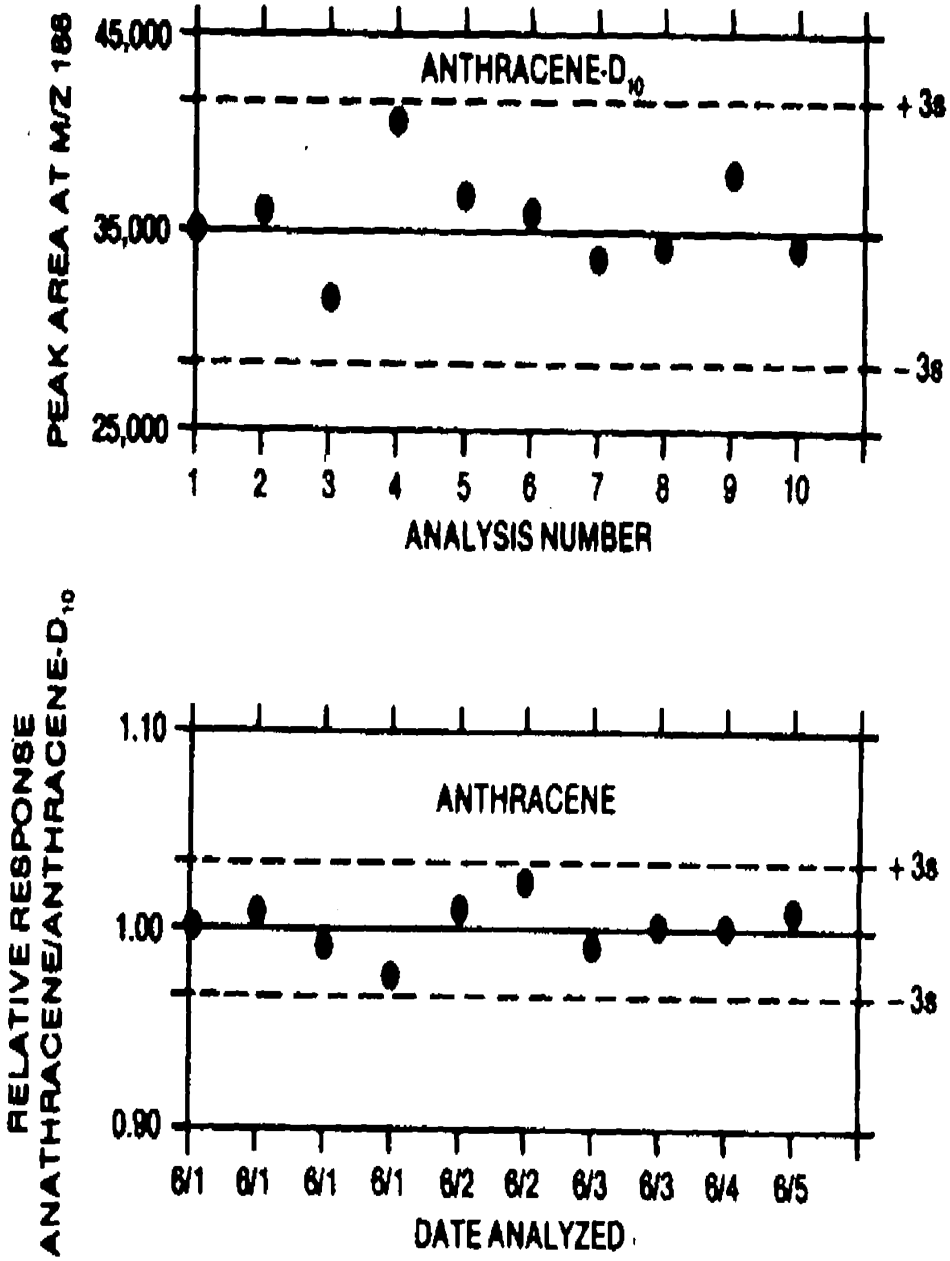

FIGURE 5 Quality Control Charts Showing Area (top graph) and Relative Response of Anthracene to Anthracene-d_{10} (lower graph) Plotted as a Function of Time or Analysis Number.

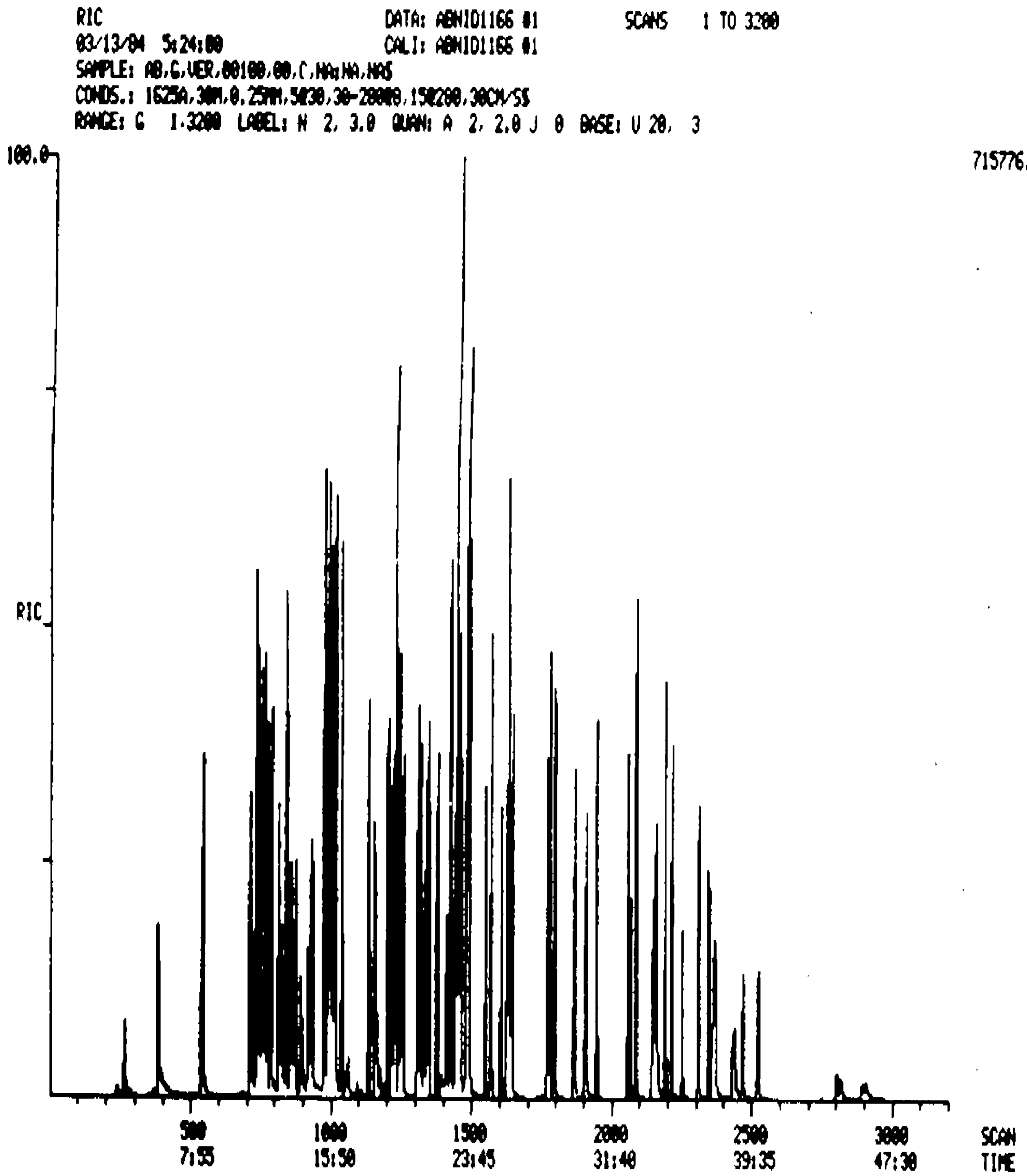

FIGURE 6 Chromatogram of Combined Acid/base/neutral Standard.