METHOD 8095

EXPLOSIVES BY GAS CHROMATOGRAPHY

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method may be used to determine the concentrations of various explosives in water and soil using capillary column gas chromatography with an electron capture detector (GC/ECD). The compounds are nitroaromatics, nitramines, and nitrate esters, which are used as explosives, are byproducts of the manufacture of explosives, or are the transformation products of explosives. The method has also been successfully used to determine the commonly found explosives in acetonitrile extracts from soil prepared by the extraction procedure in Method 8330. The following RCRA compounds have been determined by this method.

Compound	Abbreviation	CAS Number*
2-Amino-4,6-dinitrotoluene	2-Am-DNT	35572-78-2
4-Amino-2,6-dinitrotoluene	4-Am-DNT	1946-51-0
3,5-Dinitroaniline	3,5-DNA	618-87-1
1,3-Dinitrobenzene	1,3-DNB	99-65-0
2,4-Dinitrotoluene	2,4-DNT	121-14-2
2,6-Dinitrotoluene	2,6-DNT	606-20-2
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
Nitrobenzene	NB	98-95-3
Nitroglycerine	NG	55-63-0
2-Nitrotoluene	2-NT	88-72-2
3-Nitrotoluene	3-NT	99-08-1
4-Nitrotoluene	4-NT	99-99-0
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Pentaerythritoltetranitrate	PETN	78-11-5
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4
2,4,6-Trinitrophenylmethylnitramine	Tetryl	479-45-8
2,4,6-Trinitrotoluene	2,4,6-TNT	118-96-7

* Chemical Abstracts Service Registry number.

1.2 The method is capable of detecting the target compounds in a range from 0.003 to 0.5 µg/L and is capable of quantitative analysis in a range of 0.03 to 5 µg/L, depending on the sensitivity of the analyte to electron capture detection. Sensitivity data are highly matrix dependent and may not always be achievable, and are therefore provided as guidance only.

1.3 This method requires special precautions in the operation of the gas chromatograph because of the thermal lability of many of these compounds, especially the nitramines.

1.4 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, 5000, and 8000) for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under the supervision of, analysts appropriately experienced in the use of GC/ECD, skilled in the interpretation of chromatograms, and trained in the handling of environmental samples that may contain explosives. Also, each analyst must demonstrate the ability to generate acceptable results with this method.

NOTE: Refer to Sec. 5.0 for additional information on safety.

2.0 SUMMARY OF METHOD

2.1 Samples are extracted using either the solid-phase extraction techniques provided in Method 3535 (aqueous samples) or the ultrasonic extraction techniques described in Method 8330 (solid samples). Other sample preparation methods may used provided that the analyst demonstrates their applicability for the intended use. No further concentration of the extract is performed unless lower detection limits are needed.

2.2 Extracts are injected into the heated inlet of a gas chromatograph equipped with an electron capture detector. The analytes are resolved on a short wide-bore fused-silica capillary column coated with polydimethylsiloxane.

3.0 DEFINITIONS

Refer to Chapter One and the manufacturer's instructions for definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Four for general guidance on the cleaning of glassware. Also refer to Methods 3500, 3600, and 8000 for a discussion of interferences.

4.2 In addition to nitrogenated organics, the ECD will respond to other electrophores such as halogenated and oxygenated compounds. Interference by phthalate esters introduced during sample preparation can pose a major problem. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents prior to use.

4.3 The injection port liner must be deactivated to prevent adsorption of several analytes. After several injections of sample extracts, deposition of non-volatile residues may result in peak tailing and a decline in the response for HMX. Each time the septum is replaced, the injection port liner must be deactivated again or a commercially-available deactivated liner must be used. Analysts should expect to replace or deactivate the liner after every 50 injections, unless data demonstrating acceptable performance can be generated for HMX and all other analytes of interest.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 The target analytes for this method are explosive materials. Analysts must be trained in proper handling techniques for explosive-containing material in order to appropriately use this method, or supervised by those who have received such training. Caution should be exercised during the sampling of possible explosive-contaminated material. Solid material must be carefully inspected prior to extraction and care should be employed when handling the analytical standard neat material of the explosives. Observe the precautions described in the warning in Sec. 7.4.1 regarding drying the neat materials at ambient temperatures. Additional caution should be exercised if the material has a dry appearance or a grayish look that is characteristic of raw munitions.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 Gas chromatograph (GC) -- An analytical system complete with gas chromatograph, equipped with a temperature-programmable oven, an electron capture detector (ECD), and suitable for on-column injection. If a split-splitless injection port is used, deactivated direct injection port liners must be used to avoid degradation of the nitramines (especially HMX). Other necessary accessories include syringes, analytical columns, gases, and recorder/integrator or data system.

NOTE: It is recommended that deactivated direct injection port liners be purchased from a commercial vendor (Restek #20964, #20965, #20966, or equivalent).

6.2 GC columns -- The columns listed in this section were the columns used in developing the method. The analyst should select one primary and one confirmation column from either the recommendations listed below or based on other sources. The listing of these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Laboratories may use these columns or other columns provided that they document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

6.2.1 Recommended primary columns

6.2.1.1 6 m x 0.53-mm ID fused-silica, coated with 5% diphenyl - 95% dimethylsiloxane (HP-5, or equivalent), 1.5-µm film thickness.

6.2.1.2 6 m x 0.53-mm ID fused-silica, coated with 100% polydimethyl-siloxane (DB-1 or equivalent), 1.5-µm film thickness.

6.2.2 Recommended confirmatory columns

6.2.2.1 6 m x 0.53-mm ID fused-silica, coated with 100% trifluoropropyl methylpolysiloxane (Restek RTX-200 or equivalent), 1.5-µm film thickness.

6.2.2.2 6 m x 0.53-mm ID fused-silica, coated with 50% cyanopropyl-methyl - 75% phenyl methylpolysiloxane (Restek RTX-225 or equivalent), 1.5-µm film thickness.

6.3 Autosampler with ability to refrigerate vials (HP 6890 or equivalent) -- The use of an autosampler is optional, and injections may be made manually. However, if an autosampler is employed, it MUST be able to refrigerate the vials to #6 EC to avoid the degradation of the analytes of interest while the vials are sitting in the autosampler tray.

6.4 Refrigerated circulating bath (Neslab Endocal or equivalent) -- For use with the refrigerated autosampler in Sec. 6.3. Must be capable of maintaining the autosampler tray at a temperature of #6 EC.

6.5 Disposable cartridge filters, 0.45-micron (Millex SR or equivalent).

6.6 Disposable syringes, Plastipak, 3-mL (or equivalent).

6.7 Vacuum desiccator, glass.

6.8 Volumetric Flasks, Class A

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade or pesticide grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents 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 that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination or causing interferences. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

7.2 Extraction/exchange solvents

Samples should be extracted using a solvent system that gives optimum, reproducible recovery of the analytes of interest from the sample matrix, at the concentrations of interest. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed, *including* those specifically listed in this method, the analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

All solvents should be pesticide quality or equivalent. Solvents may be degassed prior to use.

7.2.1 Acetonitrile (CH_3CN)

7.2.2 Acetone (CH_3COCH_3)

7.2.3 2-Propanol ($(CH_3)_2CHOH$)

7.2.4 Methanol (CH_3OH)

7.3 Standard solutions

The following sections describe the preparation of stock, intermediate, and working standards for the compounds of interest. This discussion is provided as an example, and other approaches and concentrations of the target compounds may be used, as appropriate for the intended application. See Method 8000 for additional information on the preparation of calibration standards.

CAUTION: Calibration standards are commercially available from several sources including Supelco, AccuStandard and Radian, as both solutions and neat materials. It is **highly recommended** that commercially-prepared stock standard solutions be obtained rather than neat materials be handled.

7.4 Stock standard solution (1000 mg/L) -- Can be purchased as certified solutions or may be prepared from pure standard analytical reference material. Store purchased standards in the dark at #6 EC, or as recommended by the standard manufacturer. It is **highly recommended** that commercially-prepared stock standard solutions be obtained rather than

pure explosives and propellant material be handled. However, if the laboratory routinely handles these types of compounds, the compounds may be prepared as follows:

7.4.1 Dry about 0.15 g of the standard for each solid analyte to a constant weight in a vacuum desiccator in the dark at ambient temperature.

WARNING: HMX, RDX, Tetryl, PETN, and 2,4,6-TNT are explosives and the neat material must be handled carefully. HMX, RDX, and Tetryl neat materials are shipped under water. Drying at ambient temperature in a vacuum desiccator takes several days. DO NOT DRY AT ELEVATED TEMPERATURES!

CAUTION: The NG standard is a solution and should not be dried.

7.4.2 Place about 0.100 g (weighed to 0.0001 g) of a single analyte into a 100-mL volumetric flask and fill to volume with acetonitrile. The NG standard is a solution of NG in acetone, which should be diluted with acetonitrile.

7.4.3 Invert the flask several times until the analyte is dissolved. Store this stock solution in a refrigerator at #6 EC in the dark. Stock solutions may be used for up to one year.

7.4.4 Calculate the concentration of the stock solution from the actual weight used (nominal concentration = 1000 mg/L).

7.5 Preparation of intermediate and working standard solutions

7.5.1 The actual analytes used in the calibration mixture should be tailored to what is expected at the site being investigated. Calibration standard A, in Sec. 7.5.2, contains the analytes that are most commonly found. If the analysis of all method analytes is necessary, then the standards should be prepared as described in Sec. 7.5.2. The known co-eluting pairs for the two recommended primary GC columns are listed below:

GC Column	Co-eluting Compounds
HP-5	DNA and 4-Am-DNT
DB-1	DNA and 4-Am-DNT RDX and PETN

Any combination of analytes other than those listed in Sec. 7.5.2. should avoid having these pairs in the same standard. These coeluting compounds may be reported as the total of two compounds, unless one of the compounds is known not to be present at a site.

7.5.2 Prepare two intermediate standard solutions by combining appropriate volumes of the various stock solutions. The ECD response is dependent on the number of nitro groups. The response is greatest for 2,4,6-TNT and least for the nitrotoluenes (see Table 2). These solutions should be stored in a freezer at # -10 EC.

Intermediate Stock Solution A		Intermediate Stock Solution B	
Analyte	Concentration (µg/L)	Analyte	Concentration (µg/L)
1,3-DNB	1000	NB	5000
2,6-DNT	1000	3-NT	5000

2,4-DNT	1000	2-NT	5000
1,3,5-TNB	1000	4-NT	5000
2,4,6-TNT	1000	NG	5000
RDX	1000	PETN	5000
4-Am-DNT	1000	3,5-DNA	1000
2-Am-DNT	1000		
Tetryl	1000		
HMX	1000		

7.5.3 Prepare at least five calibration standard solutions covering a range from 0.5 to 10 µg/L for solution A, and 2.5 to 50 µg/L for solution B, or other project-specific range, by diluting the two intermediate stock solutions with acetonitrile. The calibration standards should be stored in a freezer at # -10 EC, and may be used for up to 30 days. Suggested concentrations for solution A are 0.5, 1.0, 2.0, 5.0, 10.0 µg/L and for solution B are 2.5, 5.0, 10, 25, 50 µg/L. It may be necessary or expedient to extend the calibration concentrations higher and/or lower depending on the linear calibration range of the specific detector in use. However, the analyst must also ensure that the calibration curve extends low enough to include all project-specific limits of quantitation.

CAUTION: The analytes 1,3,5-TNB, 2,4,6-TNT, and tetryl are unstable at low concentrations in some brands of acetonitrile. If the response for these analytes declines with time, another brand of acetonitrile should be used.

7.6 Preparation of surrogate spiking solution

The analyst should monitor the performance of the extraction and analytical system, as well as the effectiveness of the method in dealing with each sample matrix, by spiking each sample, standard, and reagent water blank with one or two surrogates (i.e., analytes not expected to be in the sample but having properties similar to the target analytes). Suggested surrogates are 2,5-DNT and 3,4-DNT. It is recommended that both be used. This will minimize quantitation problems caused by interference peaks since one peak should be resolved sufficiently for accurate quantitation. 2-Methyl-4-nitroaniline has also been suggested as a possible surrogate compound. Other surrogates may be used, provided that the analyst can demonstrate and document performance appropriate for the data quality needs of the particular application. Prepare a surrogate standard concentration of 0.25 g/L. Spike 1 liter of reagent water with 1.0 mL of this standard. This will result in a concentration of 0.05 g/L in the final 5.0 mL acetonitrile eluate (membrane filter method) assuming 100% recovery. Store standards in the dark at # -10 EC, or as recommended by the standard manufacturer.

7.7 Preparation of matrix spiking solutions

It is recommended that two matrix spiking solutions be prepared in acetonitrile such that the concentration in the sample is:

0.2 µg/L for 1,3-DNB, 2,6-DNT, 2,4-DNT, 1,3,5-TNB, 2,4,6-TNT, RDX, 4-Am-DNT, 2-Am-DNT, tetryl, and DNA

1.0 µg/L for NB, 3-NT, 2-NT, 4-NT, NG, and PETN

2.0 µg/L for HMX

All target analytes for a given project should be included in the matrix spiking solutions. Because RDX and PETN co-elute on the DB-1 column, these analytes should be in separate spiking solutions (A and B) when the DB-1 column is employed. Follow the same guidance outlined in Secs. 7.5.1 and 7.5.2 to determine what analytes to include in which matrix spiking solution. It would be expected that the same analytes in the calibration standard(s) are also present in the matrix spike standard(s). Store standards in the dark at # -10 EC, or as recommended by the standards manufacturer.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See the introductory material to Chapter Four, "Organic Analytes."

8.2 Sample extracts should be stored in the dark at #6 EC.

8.3 Soil samples may be contaminated, and should therefore be considered hazardous and handled accordingly. See Sec. 5.0 for additional safety considerations.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Refer to Method 8000 for specific determinative method QC procedures. Refer to Method 3500 for QC procedures to ensure the proper operation of the various sample preparation techniques. If an extract cleanup procedure is performed, refer to Method 3600 for the appropriate QC procedures. Any more specific QC procedures provided in this method will supersede those noted in Methods 8000, 3500, or 3600.

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

9.4 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. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. The laboratory must also repeat the demonstration of proficiency

whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000 for information on how to accomplish a demonstration of proficiency.

9.4.1 It is suggested that the spiking solution used to prepare the samples for this demonstration of proficiency contain each analyte of interest at the concentrations listed for the matrix spike in Sec. 7.7.

9.4.2 Calculate the average recovery and the standard deviation of the recoveries of the analytes in each of the four QC reference samples. Refer to Method 8000 for procedures for evaluating method performance.

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis. When new reagents or chemicals are received, the laboratory should monitor the preparation and/or analysis blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

9.6 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, method sensitivity). At a minimum, this should include 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 when surrogates are used. Any method blanks, matrix spike samples, and replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.6.1 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, laboratories may use a matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair. Consult Method 8000 for information on developing acceptance criteria for the MS/MSD

Note: The compounds in solution A (see Sec. 7.5.2) are more commonly found at arsenals that work with the finished product. Therefore, unless the site is expected to contain compounds found in solution B, use the compounds in solution A to prepare routine matrix spike/matrix spike duplicate pairs (see Sec. 7.7).

9.6.2 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 appropriate. 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. Consult Method 8000 for information on developing acceptance criteria for the LCS.

9.6.3 Also see Method 8000 for the details on carrying out sample quality control procedures for preparation and analysis. In-house method performance criteria for evaluating method performance should be developed using the guidance found in Method 8000.

9.7 Surrogate recoveries

If surrogates are used, the laboratory should evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000 for information on evaluating surrogate data and developing and updating surrogate limits. Procedures for evaluating the recoveries of multiple surrogates and the associated corrective actions should be defined in an approved project plan.

9.8 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.

10.0 CALIBRATION AND STANDARDIZATION

See Sec. 11.3 for information on calibration and standardization.

11.0 PROCEDURE

11.1 Sample extraction

Procedures for sample extraction of aqueous and solid samples are provided elsewhere in this manual. Methods other than those recommended below may be used, provided that they can be demonstrated to perform adequately for the intended application.

11.1.1 Consult Method 3535 for the solid-phase extraction procedures utilizing either SPE cartridges or SPE disks.

11.1.2 Consult Method 8330 for an ultrasonic procedure for the extraction of soil and solid waste samples.

CAUTION: When performing ultrasonic extraction of explosives, it is important to keep the ultrasonic bath at or below ambient laboratory temperature, in order to minimize the thermal degradation of the analytes of interest. This may be accomplished by several means, including by placing a cooling coil in the bath.

11.1.3 Store sample extracts in the freezer until analysis.

11.2 Sample analysis by GC/ECD

11.2.1 Sample extracts must be kept refrigerated at all times, in order to minimize the degradation of the analytes of interest. If an autosampler is used, it must be capable of refrigerating the vials (see Sec. 6.3) at #6 EC. When using an autosampler, allow the sample extracts to equilibrate to the temperature of the autosampler tray before beginning analyses. If manual injections are used, keep the extracts refrigerated until just before the injection is made.

11.2.2 Deactivate the GC column if the GC has not been used during the past day or so (see Sec. 11.3.4).

11.2.3 Perform initial calibration (Sec. 11.3.6) or calibration verification (Sec. 11.3.7). Beginning an analysis run with calibration verification is acceptable if the calibration verification criterion can still be met. Subsequent calibration verification includes the analysis of additional calibration standards interspersed with sample extracts (see Sec. 11.3.7). Consult Method 8000 for approaches to initial calibration and calibration verification.

11.2.4 Inject about 1 µL of the sample extract into the GC and record the exact volume injected. The same GC operating conditions used for the initial calibration must be employed for the analysis of samples.

11.2.5 Sample injections may continue for as long as the calibration verification standards and the standards interspersed with the samples meet instrument QC criteria (see Sec. 11.3.7).

11.3 Calibration of GC

11.3.1 The GC column should be baked at the injection port temperature until the baseline is stable. (The injection port temperature should not be set higher then the maximum column temperature recommended by the column manufacturer.)

NOTE: Because of the thermal lability of some of these analytes, reliable quantitation is very dependent on the condition of the GC system. The injection port and column should always be cleaned prior to performing the initial calibration. This is especially true for the nitramines and for HMX in particular.

11.3.2 Prepare calibration standards as described in Sec. 7.5. All standards should be kept refrigerated at *all* times, *including* while the standards are in the autosampler. If a refrigerated autosampler is used, it should be maintained at #6 EC. If manual injections are used, keep the standards refrigerated until just before the injection is made.

CAUTION: The compounds 1,3,5-TNB, 2,4,6-TNT and tetryl are unstable at low concentrations in some brands of acetonitrile. If the responses for these analytes decline with time, another brand of acetonitrile should be used.

11.3.3 Establish the GC operating conditions appropriate for the GC column being utilized and the target analytes listed in the project plan. Optimize the instrumental conditions for resolution of the target analytes and sensitivity. Suggested operating conditions are given below for the columns recommended in Sec. 6.2. Table 1 presents examples of analyte retention times at different linear velocities for some of the primary and confirmation columns. Figures 1 through 3 illustrate the potential effects of flow rate

on the separation and retention times of the target analytes. Use these figures as guidance in selecting the appropriate linear velocity for the target analytes in the project plan. If all target analytes are to be included, then the lowest linear velocity is recommended.

NOTE: Once established, the same operating conditions must be used for both calibrations and sample analyses.

11.3.3.1 Suggested GC operating conditions for the recommended primary columns

HP-5 column	
Injection Port Temperature:	250 EC
Injection volume:	1 µL
Carrier gas:	Hydrogen (flow rate 15 mL/min)
Makeup gas:	Nitrogen (flow rate 30 mL/min)
Detector temperature:	300 EC
Temperature Program	
Initial temperature:	100 EC for 2 min
1st temperature ramp:	10 EC per min to 200 EC
2nd temperature ramp:	20 EC per min to 250 EC
Final hold:	5.5 min

DB-1 column	
Injection Port Temperature:	250 EC
Injection volume:	1 µL
Carrier gas:	Hydrogen (linear velocity 40-125 cm/sec)
Makeup gas:	Nitrogen (flow rate 38 mL/min)
Detector temperature:	300EC
Temperature Program	
Initial temperature:	100EC for 2 min
1st temperature ramp:	10 EC per min to 200 EC
2nd temperature ramp:	20 EC per min to 250 EC
Final hold:	5 min

NOTE: Peak resolution is greatest at low linear velocity, but GC response for some analytes is greatest at high linear velocity (see Table 2). The linear velocity should be chosen based on the objectives of the analysis. A mid-range linear velocity of about 80 cm/s will be suitable for most analyses unless nitrobenzene and the nitrotoluenes are to be included and solvent peak broadening interferes.

NOTE: Other carrier gases used routinely with an ECD are acceptable. However, the use of hydrogen provides the best peak resolution. The retention times, chromatograms, and data presented in this method were developed with hydrogen.

11.3.3.2 Suggested GC operating conditions for the recommended confirmation columns

RTX-200 column (low linear velocity option)	
Injection Port Temperature:	250 EC
Injection volume:	1 µL
Carrier gas:	Hydrogen (linear velocity 40 cm/sec)
Detector temperature:	290 EC
Temperature Program	
Initial temperature:	100 EC for 1.2 min
1st temperature ramp:	5 EC per min to 140 EC
2nd temperature ramp:	1 EC per min to 160 EC
3rd temperature ramp:	20 EC per min to 250 EC

RTX-200 column (high linear velocity option)	
Injection Port Temperature:	270 EC
Injection volume:	1 µL
Carrier gas:	Hydrogen (linear velocity 122 cm/sec)
Detector temperature:	290 EC
Temperature Program	
Initial temperature:	150 EC for 1 min
Temperature ramp:	20 EC per min to 250 EC and hold

RTX-225	
Injection Port Temperature:	220 EC
Injection volume:	1 µL
Carrier gas:	Hydrogen (linear velocity 108 cm/sec)
Detector temperature:	250 EC
Temperature Program	
Initial temperature:	100 EC for 2 min
Temperature ramp:	10 EC per min to 220 EC
Final hold:	8 min

11.3.4 Because of the low concentration of standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more.

Therefore, the GC column should be primed (or deactivated) by injecting a standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

11.3.5 A 1-µL injection volume of each calibration standard is recommended.

11.3.6 Initial calibration

Inject each of the calibration standards, in order from lowest to highest concentration, and obtain peak heights or peak areas for each analyte. Follow the calibration procedures outlined in Method 8000. Normally, since this is an ECD method, external standard calibration is used along with the linear calibration model. However, other calibration models may be used, as appropriate. Calculate calibration factors (CFs) and ensure that all target analytes meet the recommended calibration criterion of #20% RSD, or other criteria appropriate for the specific project.

11.3.7 Calibration verification

11.3.7.1 See Method 8000 for detailed instructions on calibration verification. Include a mid-level calibration standard after each group of 20 samples in the analysis sequence as a calibration check. (It is *recommended* that a calibration standard be included after every 10 samples to minimize the number of repeat injections). Injections of method blanks, matrix spike samples, and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, need not be counted in the total. If all target analytes are included in a given project, it will be necessary to inject two calibration standards in order to verify the calibration of all the analytes. If the injection of both calibration standards is necessary, alternate the two calibration standards by injecting one set after the first ten samples and the second set after the second 10 samples.

NOTE: As mentioned in Sec. 7.5.1, two calibration standards are used because DNA and 4-Am-DNT coelute on both the HP-5 and the DB-1 columns and RDX and PETN co-elute on the DB-1 column. These coeluting compounds may be reported as the total of two compounds, unless one of the compounds is known not to be present at a site.

11.3.7.2 If there has been a break in analyses since initial calibration was performed, begin by injecting a mid-level calibration standard containing the target analytes. As noted above, the injection of two calibration standards may be needed. Ensure that the recommended calibration verification criterion of ±20% is met before beginning the injection of samples. If the results for the verification standard are close to the control limits, it is highly recommended that injection port and column maintenance be performed and the verification standard be successfully reanalyzed prior to proceeding with sample injections. This should be done to avoid having to reinject all samples if the next calibration verification standard exceeds the criterion. This is especially true if HMX is a target analyte and is close to exceeding control limits.

11.3.7.3 The calibration factors for the verification should be within ±20% of the mean calibration factors from the initial calibration (see Sec. 11.0 of Method 8000 for more details). When this calibration verification standard falls out

of this acceptance window, the laboratory should stop analyses and take corrective action as outlined in Method 8000, and recalibrate, if necessary.

11.3.7.4 If quantitation is accomplished using an internal standard, internal standards must be evaluated for acceptance. The measured area of the internal standard must be no more than 50 percent different from the average area calculated during calibration. When the internal standard peak area is outside the limit, all samples that fall outside the QC criterion must be reanalyzed or the data must be treated as estimated.

11.3.8 Retention time windows -- Absolute retention times are used for compound identification. Retention time windows are crucial to the identification of target compounds, and should be established by one of the approaches described in Method 8000.

11.3.8.1 Before establishing the retention time windows, make sure the gas chromatographic system is operating within optimum conditions.

11.3.8.2 The widths of the retention time windows are defined as described in Method 8000. However, the experience of the analyst should weigh heavily in the interpretation of the chromatograms.

11.4 GC analysis

11.4.1 Compare the retention time of each analyte in the calibration standard with the absolute retention time windows established in Sec. 11.3.8. As described in Method 8000, the center of the absolute retention time window for each analyte is its retention time in the mid-concentration standard analyzed during the initial calibration. Each analyte in each standard must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard does result in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

11.4.2 Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window. Each tentative identification should be confirmed using either a second GC column of dissimilar stationary phase or using another technique such as GC/MS or HPLC/UV (see Method 8000, Sec. 11.0).

11.4.3 For the options on reporting confirmation results, see Sec. 11.0 of Method 8000.

11.4.4 For guidance on verification of external standard calibrations, see Sec. 11.3.7 of this method and see Method 8000. When performing external standard calibration and a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent misquantitations and possible false negative results. Reinjection of the sample extracts may be needed. More frequent analyses of standards will minimize the number of sample extracts that would have to be reinjected if the QC limits are violated for the standard analysis.

However, if the standard analyzed <u>after</u> a group of samples exhibits a response for an analyte that is <u>above</u> the acceptance limit, i.e., >20%, and the analyte was <u>not</u> detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the verification standard has demonstrated that

the analyte would have been detected if it were present. In contrast, if an analyte above the QC limits was detected in a sample extract, then reinjection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 20% *below* the initial calibration response, then reinjection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present (i.e., a false negative result).

11.4.5 The target compounds in this method are known to decompose if the injection port of the GC becomes activated. Therefore, when a calibration verification standard fails, the analyst must evaluate whether the calibration has drifted out of the acceptable range due to normal calibration drift or active sites in the injection port causing degradation of the target compounds. Degradation of the target compounds would be expected to lead to lower responses.

When reviewing the results of the calibration verification standard, the analyst should keep in mind the number of samples run and/or whether some very dirty samples may have contaminated the injection port. As noted in Sec. 4.3, the injection port liner must be deactivated again or a commercially-available deactivated liner should be inserted each time the septum is replaced or at least after every 50 injections.

11.4.6 For guidance on calculating results from internal and external standard calibrations, see Method 8000. The nominal final extract volumes are 4.0 mL for the cartridge SPE method and 5.0 mL for the disk SPE method, unless the eluate was concentrated further. For proper quantitation, the appropriate selection of a baseline from which the peak area or height can be determined is needed.

11.4.7 As mentioned in Sec. 7.5.1, DNA and 4-Am-DNT coelute on both the HP-5 and the DB-1 columns and RDX and PETN co-elute on the DB-1 column. These coeluting compounds may be reported as the total of two compounds, unless one of the compounds is known not to be present at a site.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 See Method 8000 for information regarding data analysis and calculations.

12.2 Results need to be reported in units commensurate with their intended use and all dilutions need to be taken into account when computing final results.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 Tables 1 and 2 present example retention times and calibration factors obtained on various GC columns and using several different carrier gas velocities. Example chromatograms are presented in Figures 1 through 3 (Reference 3). These data are provided for guidance purposes only.

13.3 Using the disk and cartridge SPE procedures outlined in Method 3535, single-laboratory lower limits of detection for the analytes in solution A (Sec. 7.5.2) ranged from approximately 0.003 to 0.02 µg/L, based on a spiked sample concentration of 0.01 µg/L. For the analytes in solution B, the lower limits of detection ranged from approximately 0.2 to 0.5 µg/L for the analytes spiked at 1 µg/L, and was approximately 0.06 µg/L for 3,5-DNA, which was spiked at 0.2 µg/L. These data are provided for guidance purposes only.

13.4 Table 3 presents single-laboratory recovery and precision data for the analytes spiked into 500 mL of reagent water. Extraction was by the disk SPE method (see Reference 3). These data are provided for guidance purposes only.

13.5 Single-laboratory lower limits of detection were determined for spiked soil samples prepared in two different soil matrices. Lower limits of detection ranged from approximately 0.7 to 3.5 µg/kg for analytes spiked at 5 µg/kg, and from approximately 10 to 25 µg/kg for analytes spiked at 50 µg/kg. These data are provided for guidance purposes only.

13.6 Table 4 presents single-laboratory recovery and precision data for the analytes spiked into two different soil matrices. Extraction was performed by the procedure described in Method 8330. These data are provided for guidance purposes only.

13.7 Tables 5 and 6 present data on the recovery and repeatability of GC/ECD and HPLC/UV determinations of the analyte concentrations in spiked water samples. The HPLC conditions were those found in Method 8330. The samples were extracted as described in Method 3535 and the extracts were split for GC and HPLC analysis. The analytes in this study represent those most commonly found at arsenals around the country. Data from the disk SPE method are presented in Table 5 and data from the cartridge SPE method are presented in Table 6 (see Reference 2). These data are provided for guidance purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

14.3 This method conforms with the USEPA's pollution prevention goals. The cartridge SPE method uses only 34 mL of acetonitrile per sample and the disk SPE method only uses 45 mL of solvent.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from

hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. M. Hable, C. Stern, C. Asowata, and K. Williams, "The Determination of Nitroaromatics and Nitramines in Ground and Drinking Water by Wide-Bore Capillary Gas Chromatography," *Journal of Chromatographic Science*, 29:131-135 (1991).

2. M. E. Walsh and T. Ranney, "Determination of Nitroaromatic, Nitramine, and Nitrate Ester Explosives in Water using Solid-phase Extraction and Gas Chromatography-electron Capture Detection: Comparison with High-performance Liquid Chromatography," *Journal of Chromatographic Science*, 36, pp. 406-416 (1998).

3. M. E. Walsh and T. Ranney, "Determination of Nitroaromatic, Nitramine, and Nitrate Ester Explosives in Water Using SPE and GC-ECD: Comparison with HPLC," CRREL Report 98-2. U.S. Army Cold Regions Research and Engineering Laboratory, Hanover, NH (1998).

17.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

TABLE 1

EXAMPLE RETENTION TIMES FOR VARIOUS GC COLUMNS
OBTAINED AT DIFFERENT CARRIER GAS LINEAR VELOCITIES

	Retention Time (min)				
	DB-1 Column		RTX-200 Column		RTX-225 Column
Compound	LV = 126	LV = 44	LV = 40	LV = 122	LV = 108
NB	0.32	1.38	2.15	ES	ND
2-NT	0.47	2.06	2.78	ES	0.95
3-NT	0.57	2.47	3.40	ES	1.20
4-NT	0.62	2.69	3.72	ES	1.40
NG	1.18	3.84	8.57	0.52	6.25
1,3-DNB	1.84	5.05	9.01	0.63	5.86
2,6-DNT	2.07	5.28	8.51	0.55	5.50
2,4-DNT	2.88	6.12	10.64	0.88	6.51
1,3,5-TNB	4.19	7.42	18.90	1.98	9.99
2,4,6-TNT	4.61	7.82	17.81	1.86	9.51
PETN	5.62	8.79	28.52	2.74	11.57
RDX	5.62	8.83	29.19	2.86	13.66
4-Am-DNT	6.77	9.92	23.80	2.45	12.65
3,5-DNA	6.83	10.07	26.08	2.65	13.32
2-Am-DNT	7.17	10.38	28.57	2.85	13.17
Tetryl	8.05	11.26	32.11	3.54	13.65
HMX	11.21	13.92	not eluted	6.29	not eluted

surr = Surrogate
LV = Linear Velocity in cm/s
ES = Elutes in solvent peak
ND =- Not Determined
Data are taken from Reference 3
Chromatographic conditions are described in Sec. 11.3.3
All data are provided as examples only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

TABLE 2

EXAMPLE CALIBRATION FACTORS OBTAINED AT DIFFERENT CARRIER GAS LINEAR VELOCITIES USING A DB-1 COLUMN AND 50 µg/L SOLUTIONS

	Calibration Factor	
Compound	LV = 126 cm/s	LV = 44 cm/s
2,4,6-TNT	104	109
2,6-DNT	91	92
2-Am-DNT	80	83
RDX	79	52
HMX	75	32
4-Am-DNT	71	76
DNA	69	69
2,4-DNT	58	58
Tetryl	46	59
1,3,5-TNB	45	50
1,3-DNB	30	28
NB	18	9.9
PETN	17	8.1
NG	12	4.5
3-NT	7.5	7.4
2-NT	5.9	3.6
4-NT	2.5	4.5

The calibration factor is calculated as the peak height divided by the concentration of the standard. All data are provided as examples only. Each laboratory must determine calibration factors for their specific application of the method.

Data are taken from Reference 3.

TABLE 3

RECOVERY OF TARGET ANALYTES FROM WATER USING DISK SPE PROCEDURES

Compound	Spike Conc. (µg/L)	Mean Recovery (%)	RSD(%)
1,3-DNB	0.2	99	9.7
2,6-DNT	0.2	93	7.1
2,4-DNT	0.2	104	7.6
1,3,5-TNB	0.2	94	7.7
2,4,6-TNT	0.2	116	8.0
RDX	0.2	88	7.2
4-Am-DNT	0.2	75	11.2
2-AM-DNT	0.2	87	11.6
Tetryl	0.2	95	8.3
DNA	0.2	74	9.2
NB	1	97	7.1
2-NT	1	93	5.1
3-NT	1	92	4.6
4-NT	1	90	5.3
NG	1	92	5.5
PETN	1	99	4.8
HMX	2	79	8.1

Mean recovery from seven replicate 500-mL water samples extracted using Empore SDB-RPS disks and with a final volume of 5 mL of acetonitrile.

Data are taken from Reference 3.
These data are provided for guidance purposes only.

TABLE 4

SINGLE LABORATORY PERFORMANCE DATA FOR EXPLOSIVES IN SOIL

		Ottawa Sand		AEC Soil	
Compound	Spike Level (µg/kg)	Mean Recovery (%)	RSD (%)	Mean Recovery (%)	RSD (%)
1,3-DNB	50	106	2.4	102	3.4
2,6-DNT	50	107	1.8	105	3.6
2,4-DNT	50	108	1.9	105	3.4
TNB	50	126	4.3	91	19.6
TNT	50	120	2.6	107	3.9
RDX	50	118	6.9	93	11.4
4-Am-DNT	50	106	4.8	99	7.1
3,5-DNA	50	111	5.6	100	8.1
2-Am-DNT	50	113	5.2	107	7.3

Seven soil samples were extracted using the approach described in Method 8330.

Sample Prep: 2.00 g soil (AEC or Ottawa Sand) spiked at either 5 or 50 µg/kg, extracted with 10 mL acetonitrile by 18-hour ultrasonic extraction in a water-cooled ultrasonic bath. Filtration of extract through Millex SR filter unit.

These data are provided for guidance purposes only.

TABLE 5

COMPARISON OF GC/ECD AND HPLC/UV DETERMINATIONS
OF SPIKED WATER SAMPLES USING EMPORE SDB-RPS 47-mm SPE DISKS

Compound	Spiked Conc. (µg/L)	Conc. Found (µg/L) Replicate 1	Replicate 2	Mean Rec. (%)	RPD (%)
GC/ECD result					
1,3-DNB	5.06	4.77	4.35	90	9.4
2,6-DNT	5.08	4.88	4.48	92	8.7
2,4-DNT	5.12	4.78	4.50	91	6.1
1,3,5-TNB	5.04	4.33	4.25	85	1.7
2,4,6-TNT	5.01	4.72	4.63	93	1.9
RDX	10.0	9.55	9.32	94	2.4
4-Am-DNT	5.06	4.51	4.28	87	5.1
2-Am-DNT	5.02	5.74	5.22	109	9.5
HMX	50.1	49.7	47.0	96	5.6
HPLC/UV result					
1,3-DNB	5.06	5.45	5.26	106	3.7
2,6-DNT and 2,4-DNT[†]	10.2	10.6	10.2	102	3.9
1,3,5-TNB	5.04	5.62	5.18	107	8.1
2,4,6-TNT	5.01	6.04	5.48	115	9.8
RDX	10.0	10.3	10.3	103	0.2
4-Am-DNT and 2-Am-DNT[†]	10.1	10.8	10.3	105	5.2
HMX	50.1	45.9	46.9	93	2.2

[†] Peaks are not resolved and the results are reported as the total of the two compounds.
Data are taken from Reference 2.
These data are provided for guidance purposes only.

TABLE 6

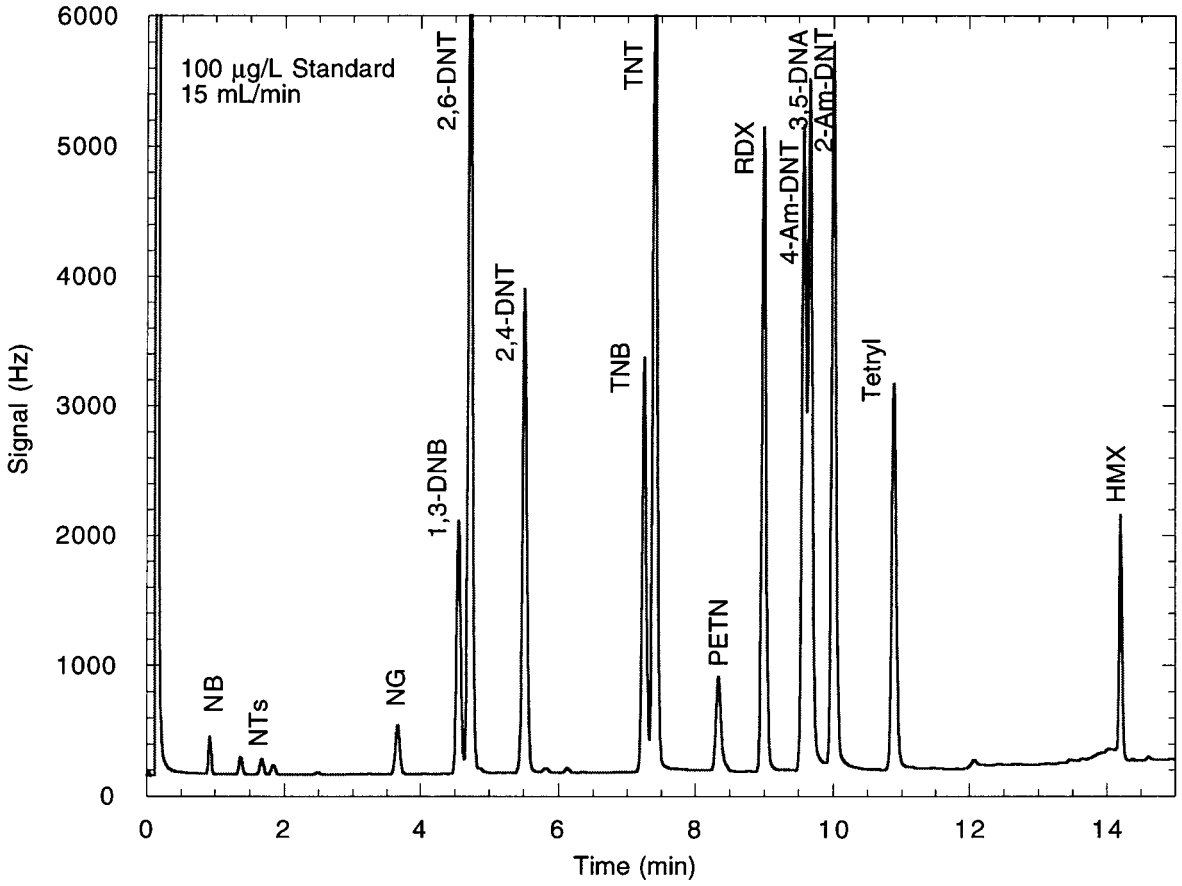
COMPARISON OF GC/ECD AND HPLC/UV DETERMINATIONS OF SPIKED WATER SAMPLES USING WATERS SEP-PAK RDX SPE CARTRIDGES

Compound	Spiked Conc. (µg/L)	Conc. Found (µg/L) Replicate 1	Replicate 2	Mean Rec. (%)	RPD (%)
GC/ECD result					
1,3-DNB	5.06	5.20	4.66	98	11.1
2,6-DNT	5.08	5.29	4.87	100	8.3
2,4-DNT	5.12	5.03	4.80	96	4.6
1,3,5-TNB	5.04	4.92	4.73	96	3.8
2,4,6-TNT	5.01	5.26	5.07	103	3.7
RDX	10.0	10.8	10.6	106	1.8
4-Am-DNT	5.06	5.05	4.58	95	9.6
2-Am-DNT	5.02	5.26	4.85	101	8.1
HMX	50.1	68.8	67.7	136	1.6
HPLC/UV result					
1,3-DNB	5.06	5.76	5.70	113	1.1
2,6-DNT and 2,4-DNT[†]	10.16	11.0	11.0	108	0.3
1,3,5-TNB	5.04	5.71	5.67	113	0.7
2,4,6-TNT	5.01	5.97	5.99	119	0.4
RDX	10.0	12.5	12.1	123	3.3
4-Am-DNT and 2-Am-DNT[†]	10.1	10.6	10.6	105	0.4
HMX	50.1	55.5	56.2	111	1.3

[†] Peaks are not resolved and the results are reported as the total of the two compounds.
Data are taken from Reference 2.
These data are provided for guidance purposes only.

FIGURE 1

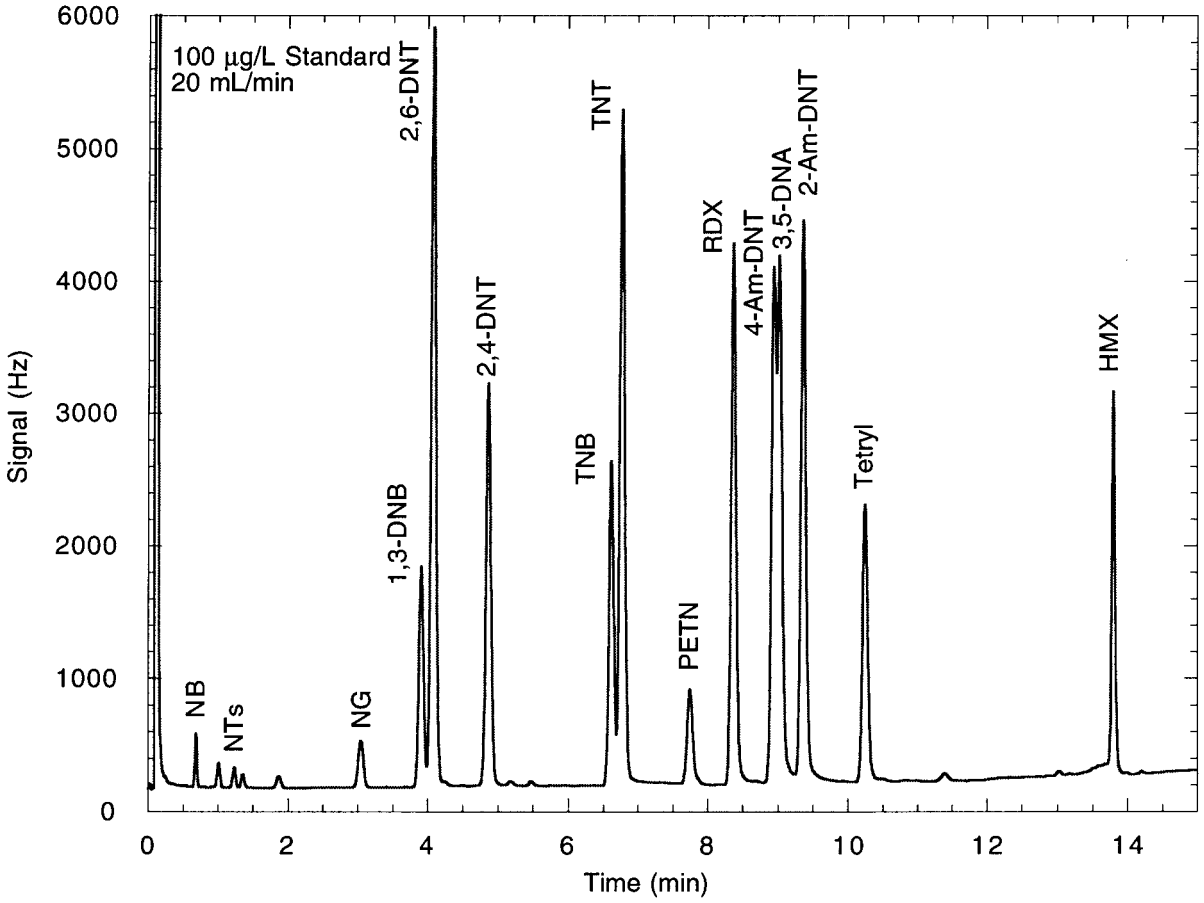
CHROMATOGRAM OF A 100 µg/L STANDARD ON AN HP-5 GC COLUMN
AT A FLOW RATE OF 15 mL/MIN HYDROGEN CARRIER GAS



GC operating conditions are listed in Sec. 11.3.3.1.

FIGURE 2

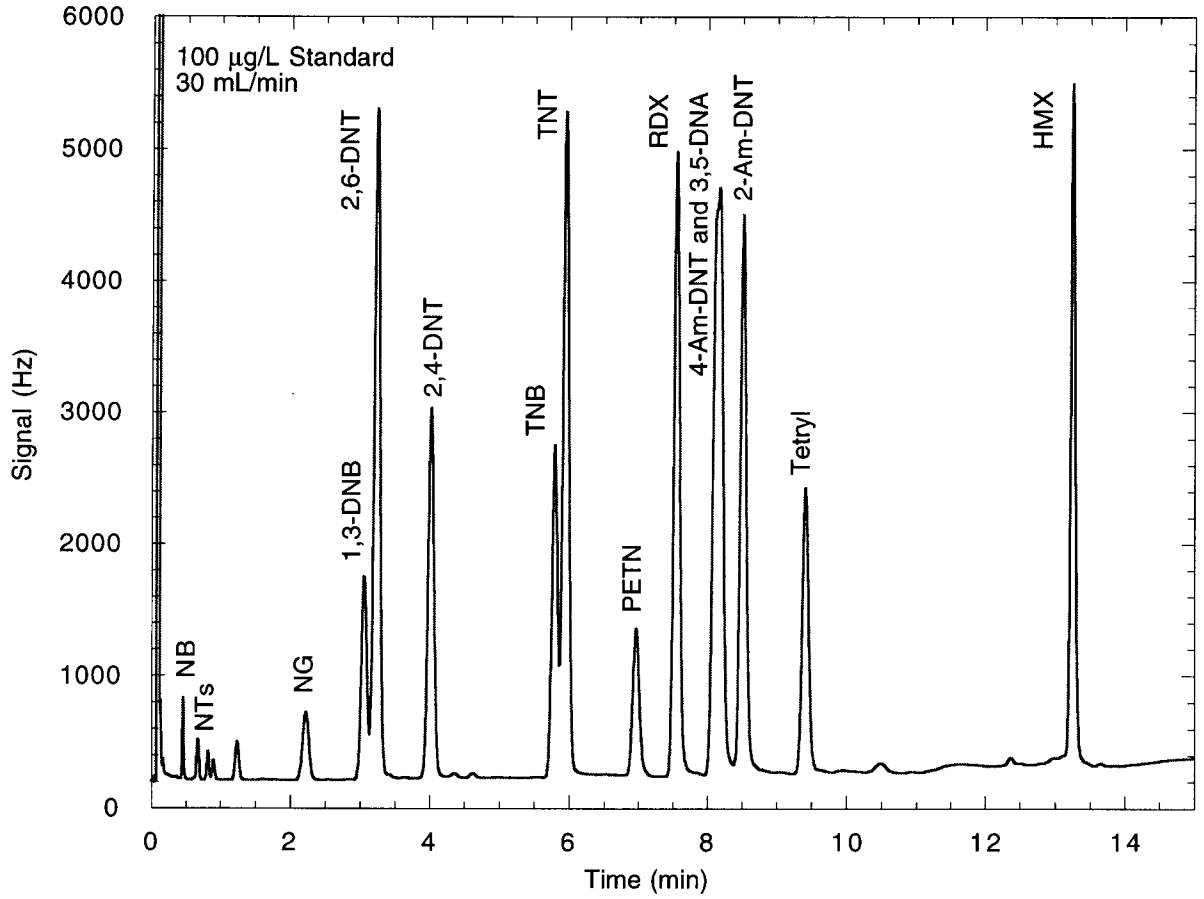
CHROMATOGRAM OF A 100 µg/L STANDARD ON AN HP-5 GC COLUMN
AT A FLOW RATE OF 20 mL/MIN HYDROGEN CARRIER GAS



GC operating conditions are listed in Sec. 11.3.3.1. Note the effect of the flow rate on the separation of 4-Am-DNT and 3,5-DNA , as well as on the retention time of HMX.

FIGURE 3

CHROMATOGRAM OF A 100 µg/L STANDARD ON AN HP-5 GC COLUMN
AT A FLOW RATE OF 30 mL/MIN HYDROGEN CARRIER GAS



GC operating conditions are listed in Sec. 11.3.3.1. Note the effect of the flow rate on the separation of 4-Am-DNT and 3,5-DNA , as well as on the retention time of HMX.