A Procedure for Sampling and Analysis of Air for Energetics and Related Compounds

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Abstract

A procedure for the sampling and analysis of energetics and related compounds in the atmosphere is described. The basic procedure consists of the collection of air samples using sampling cartridges containing XAD-2 resin, extraction of the resin with isoamyl acetate, and an analysis of the extract using gas chromatography with electron capture detection. Modifications and additions to this procedure are discussed, such as the use of a prefilter before the resin sampler to collect particulates and the use of a mass selective detector to analyze for some propellant compounds of interest or for quantitative confirmation purposes. Two differing sizes of samplers are evaluated according to the air volumes required for collection. The procedure is tested through the analysis of spiked resin samples, which had air pulled through them for periods of time corresponding with the required sampling volumes. This procedure has application toward the measurement of energetic residues in atmospheres resulting from weapons testing and operations during training exercises involving munitions.

Introduction

The quantitative measurement of the residual amounts of energetics and related compounds in the environment has been routinely performed for over three decades. There are numerous methods used to analyze soils and waters for nitroaromatics, nitramines, and other compounds related to U.S. munitions (1–6). The U.S. Army has used many of these methods in the course of environmental monitoring to protect the health and safety of soldiers and the general population. It has also relied on these methods to measure soil and water contamination from explosives during environmental cleanup operations. The procedures generally involve gas chromatographic (GC) and high-performance liquid chromatographic (HPLC) analyses, but there are also thin-layer chromatography and immunoassay methods that are useful as field screening tests (7–8).

Additionally, there are methods used to monitor selected compounds such as trinitrotoluene (TNT) and dinitrotoluenes in workplace atmospheres (9–10). This monitoring is used to ensure that munitions workers are not exposed to harmful levels of these compounds. However, there has been little done toward environmental air monitoring for energetics other than the specific case of stack emissions produced during weapons destruction by incineration. The primary impetus for stack monitoring has been to determine destruction efficiencies associated with the processes used to burn the munitions feedstocks. The measurement of energetic and related compounds in the general atmosphere from a health-risk standpoint has become an issue only in the last few years.

The U.S. Army has recognized the need to perform air monitoring for energetics, partially because of public concern about air-quality issues in areas near U.S. military reservations. There are operations during weapons testing and training that are potentially capable of putting measurable quantities of energetics and related compounds into the atmosphere. As a result, the Army Center for Health Promotion and Preventive Medicine (USACHPPM) has determined the need to modify current air-sampling methodologies and analytical techniques to provide monitoring efforts for a suite of explosives compounds, including those commonly analyzed for by soil and water methods. The list of compounds of concern includes the nitroaromatics (such as TNT, tetryl, and their precursors and breakdown products) and nitramines (such as hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)). There are also other propellant compounds of occasional concern, including nitroglycerin, dibutyl- and dioctyl-phthalates, diphenylamine, and pentaerythritol tetranitrate (PETN).

There are U.S. Environmental Protection Agency (EPA) air-sampling procedures that employ sampling devices containing XAD-2 resin to trap polynuclear aromatic hydrocarbons from ambient air and semivolatile organic hazardous compounds in stack emissions (11,12). USACHPPM has successfully used modifications of several types pertaining to the XAD-2 sampling trains for the collection of stack emissions for energetic residues. We decided therefore to investigate the use of glass cartridges packed with XAD-2 resin for general atmospheric sampling for the energetics and propellant compounds. Preliminary tests were conducted using PS-1 cartridges manufactured for EPA Air Toxics Method TO-13 for polynuclear aromatic hydrocarbons, and a field study was successfully performed using these cartridges. Recently, newly designed cartridges have been employed. These

cartridges are somewhat more robust during shipping and handling than the original types and are compatible with the sampling requirements of the U.S. Army during weapons testing. These cartridges are of two types: the first being a modification of the original PS-1 design used for high-volume sampling (Figure 1A) and the second a smaller two-section cartridge designed for shorter test intervals (Figure 1B).

The analytical approach has generally been to use a modification of existing USACHPPM GC procedures for the nitro-containing compounds. These procedures use electron-capture detection and have been used for many years in our laboratories to reliably quantitate these compounds from a variety of matrices (3,4,10,13). GC using a mass selective detector (MSD) was chosen as the most expedient means of analyzing for the phthalate esters and diphenylamine, because all can be done in a single GC run. The XAD-2 resin was solvent desorbed with isoamyl acetate in order to place the analytes into solution prior to analysis. Isoamyl acetate has proven to be an excellent solvent for the compounds of interest. It also provides superior response and reproducibility with the electron-capture detector compared with other solvents tried (such as acetonitrile). Finally, it helps to minimize chromatographic problems that can arise with moisture-laden samples because it is not water miscible (and thus does not retain the water).

The sampling cartridges, the chromatographic and analytical procedures used to analyze for the compounds of concern, and the test results from the spike studies conducted with the samplers will be described.

Experimental

Air-sampling cartridges

The initial testing was done using PS-1 sampling cartridges packed with 55 g of XAD-2 resin. Subsequent tests with these samplers used 50 g rather than 55 g of the XAD-2, primarily as a matter of convenience. These cartridges are designed such that the glass cartridge contains a metal screen at the bottom to retain the resin during sampling, and the resin is sandwiched above the screen between two sections of glass wool. Design specifications for these cartridges vary, but the basic size of the inside sampling bed is 4 × 2.25 inches. The actual sampler and its calibration and use has been described elsewhere (11).

The modified sampling cartridges were manufactured by Ace Glass Inc. (Vineland, NJ). The larger size being tested still employed 50 g of XAD-2 resin, but the resin was retained by a metal screen/metal mesh combination at both ends of the cartridge, with some glass wool at the outlet end only. The glass cartridge body and contents were held together using Teflon end fittings. The smaller size cartridge used two 10-g sections of XAD-2 resin separated by glass wool. It also contained metal screens and mesh at both ends and glass wool between the screen/mesh and resin at the outlet end of the cartridge. The inner diameter of the glass body was smaller, but the cartridge was similar to the large one in its use of metal screens and Teflon end fittings (as shown in Figure 1B). The second section of the smaller cartridge was used as a back-up to measure breakthrough. If more than 20% of the total of an analyte was found in this section, then the cartridge was considered to have been oversampled. Both types of cartridges were compatible with the sampling devices used with the original PS-1 cartridges and were used in the same fashion.

The XAD-2 resin used for packing the cartridges was a styrene–divinylbenzene porous polymer. It was purchased from Restek Corporation (Bellefonte, PA) under the name "Ultra Clean XAD-2 Resin". It was found to be sufficiently clean because it did not require further purification for application toward energetic sampling. It was noted, however, that its appearance varied between different lots of the material. This did not seem to affect the resin's adsorbent properties, but it had other effects (as will be described).

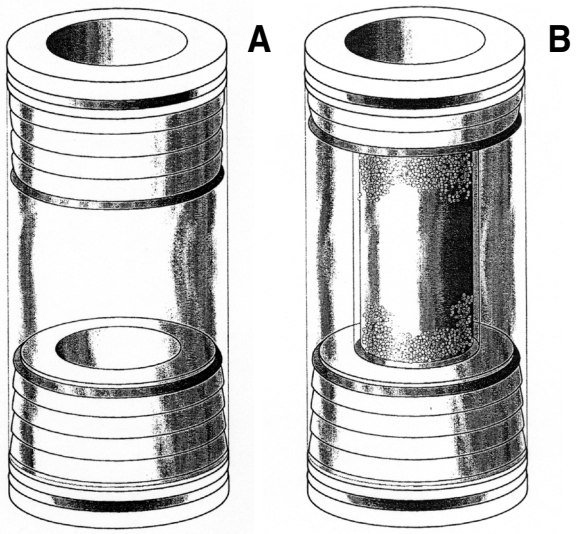

Figure 1. Cartridge designs for XAD-2 samplers used with energetics sampling in air: (A) 50-g cartridge with a modified PS-1 design used for high-volume sampling and (B) 10-g two-section XAD-2 cartridge used for shorter time sampling.

Recovery tests

The ability of the XAD-2 cartridges to retain the compounds of concern while large volumes of air were passed through them was tested. Solutions containing known amounts of the analytes in acetonitrile were spiked into the front part of the resin within a cartridge (in the case of a two-section cartridge the front section was spiked). The cartridges were placed in a PS-1 sampling apparatus, and clean ambient air was pulled through in the same way as it is generally done with actual sampling in the field. The cartridges were then returned to the laboratory for the analysis and evaluation of analyte retention.

Analytical procedures

The XAD-2 from sampled cartridges was transferred to 250-mL glass bottles with Teflon-lined caps. The two section cartridges used one bottle per section. Isoamyl acetate (Aldrich, Milwaukee, WI) was added to the containers to desorb the analytes of interest from the resin. The nominal amounts used were 100 mL for the large size samples and 25 mL for the small size samples. Usually, these amounts were sufficient to cover all the resin in a jar, but occasionally they had to be increased to 125 mL and 40 mL,

respectively, as a result of the excessive swelling of the resin when placed in the solvent. The reason for this was not known but appeared to vary with the lot of resin used to pack the cartridge. The jars or vials were agitated for 2 h on a platform-type shaker in order to ensure adequate resin–solvent contact, then allowed to sit for at least 18 h in a refrigerator at 5°C. A portion of the solvent extract was subsequently placed in an autosampler vial prior to analysis for energetics and propellant compounds.

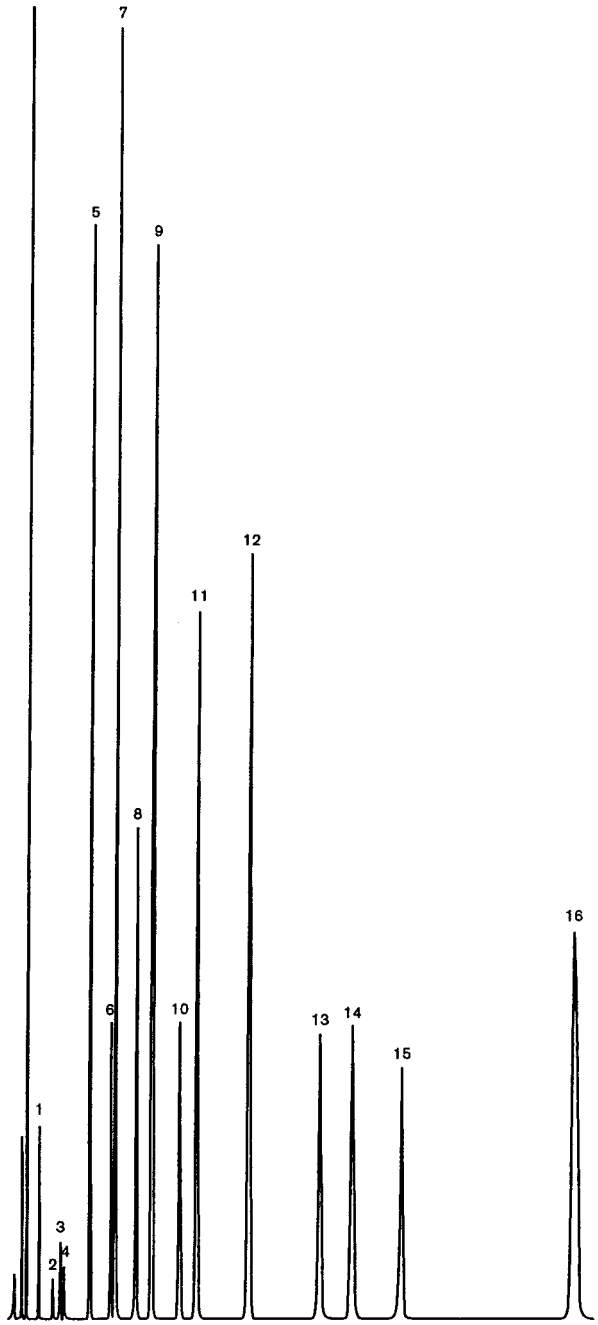

Figure 2. Chromatogram for energetics analysis on a 7-m, 0.53-mm-i.d., 1.0-µm film DB-1 column with electron-capture detection: (1) nitrobenzene, RT = 1.57; (2) 2-nitrotoluene, RT = 2.03; (3) 3-nitrotoluene, RT = 2.29; (4) 4-nitrotoluene, RT = 2.41; (5) nitroglycerin, RT = 3.26; (6) 1,3-dinitrobenzene, RT = 3.97; (7) 2,6-dinitrotoluene, RT = 4.13; (8) 2,4-dinitrotoluene, RT = 4.84; (9) 3,4-dinitrotoluene, RT = 5.37; (10) 1,3,5-trinitrobenzene, RT = 6.32; (11) 2,4,6-TNT, RT = 6.88; (12) RDX, RT = 8.62; (13) 4-amino-2,6-dinitrotoluene, RT = 11.04; (14) 2-amino-4,6-dinitrotoluene, RT = 12.14; (15) tetryl, RT = 13.80; and (16) HMX, RT = 19.56.

Energetic stock standards were purchased as 1.0-mg/mL solutions. All of them were available from AccuStandard Inc. (New Haven, CT) except nitroglycerin, which was purchased from Cerilliant (Austin, TX). The diphenylamine and phthalate esters were available as neat materials from Aldrich. Working standards in isoamyl acetate were prepared from the stock standards (or neat materials). The energetic and nitroglycerin standards ranged from 0.01 to 2.0 µg/mL (0.02 to 4.0 µg/mL for HMX), and the standards for MSD analysis were from 0.5 to 5.0 µg/mL.

The chromatographic analyses for the energetics and nitroglycerin were conducted using Agilent Technologies (Wilmington, DE) Model 6890 GCs equipped with electron-capture detectors. Chromatographic runs (shown in Figure 2) were typically made using a DB-1 column (J&W Scientific, Folsom, CA) (0.53-mm i.d., 1.0-µm film thickness) cut to 7 m in length. The GC oven was temperature programmed from 80°C at 15°C/min to 140°C, then to 170°C at 3°C/min, and finally to 200°C at 5°C/min and held for 3.0 min. The helium carrier gas was programmed from 2.0 psig (held for 13.0 min) to 4.0 psig at a rate of 150 psig/min and held. The dilution gas was nitrogen at 30 mL/min. The injection-port temperature was set at 225°C, and the injection-port liner was a Silcosleeve (Restek) with a Silcosteel seal used in splitless mode. The Ni-63 electron capture detector temperature was 250°C. Data processing was done using Turbochrom (PE Nelson, Shelton, CT). An Agilent Model 7673A autosampler was used to make the injections (the injection volume was 1.0 µL).

Analyses for the phthalate esters and diphenylamine were done using an Agilent Technologies 5792 MSD interfaced with an Agilent 5890 GC. The analytical column was an RTX-5ms column (Restek) (0.25-mm i.d., 0.25-µm film thickness) that was 30 m in length. The GC oven was temperature programmed from 80°C at 30°/min to 260°C and then held for 6.0 min. The helium carrier gas was set to a constant pressure of 14 psi. The injection-port temperature was set at 275°C, and the injection-port liner was a Silcosleeve with a Silcosteel seal used in splitless mode. The GC–MSD interface temperature was 260°C. An Agilent Model 7673 autosampler was used to make the injections (the injection volume was 3.0 µL). The detector was scanned from *m/z* 45 to 300 after a 5-min solvent delay. Data processing was done using Agilent ChemStation software. Figure 3 shows a typical chromatogram of the three analytes.

Results and Discussion

Sampling

All recovery tests were done using spiked cartridges. We recognize that the ideal way to evaluate the cartridges would have been to sample atmospheres containing known concentrations of the target analytes, but unfortunately this was not an option. It would be difficult (if not impossible) to generate stable atmospheres of a known vapor concentration for many of the compounds. The situation was further complicated by the requirement to sample very large air volumes (a small test chamber would be inadequate for such testing). Fortunately, the ability of XAD-2 to trap ener-

getics has been demonstrated by actual field sampling. XAD-2 resin has been successfully used by USACHPPM over the last dozen years to sample for some of the target compounds of this study. It has also been used for gas sampling during the testing of proprietary methodology used for the destruction of chemical munitions. The multisection sampling tubes used in both instances were of a different design than the cartridges described in this study, but the resin was the same. Recent field-sampling events using the cartridges described in this study have also shown that the resin is effective in trapping the energetics with minimal breakthrough. We are confident that these spiking tests provide an adequate further indication of the utility of these cartridges for energetic trapping and retention.

The recovery results for seven replicate tests on the smaller cartridges are presented in Table I. These cartridges had 2.6 to 2.7 m^3 of air pulled through them. Similar tests on the larger cartridges (but with 130 m^3 of air) are shown in Table II.

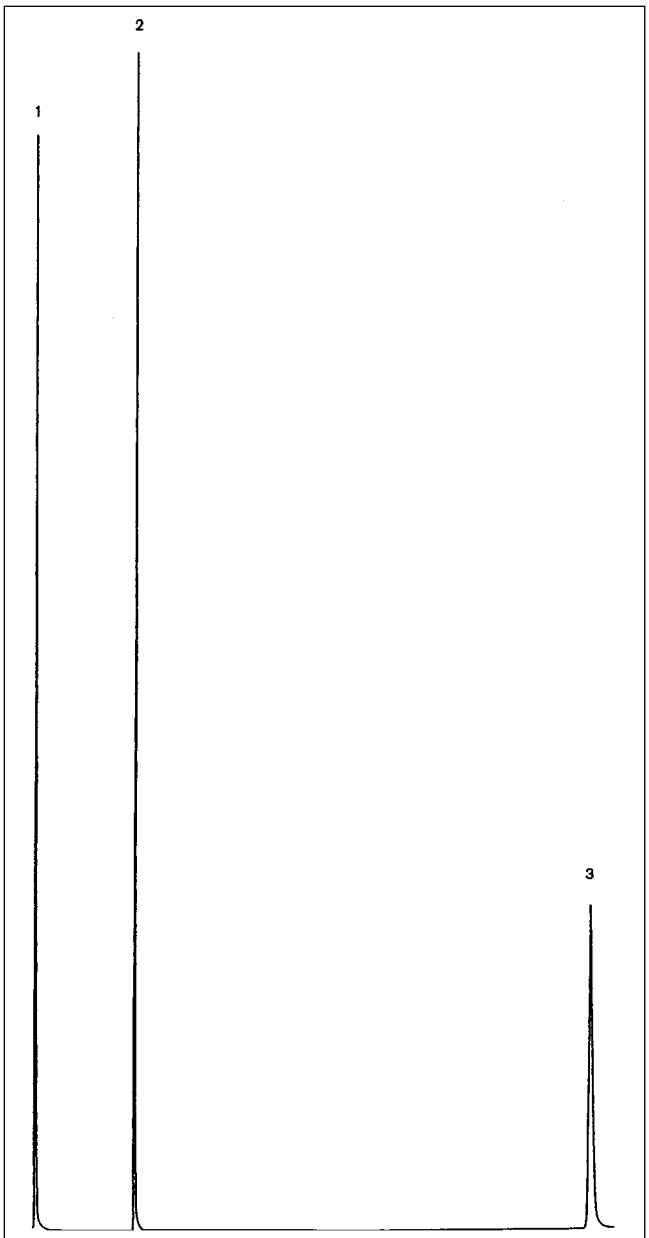

Figure 3. Total ion chromatogram for propellant analysis of 5.0 µg/mL diphenylamine and phthalate esters on a 30-m, 0.25-mm-i.d., 0.25-µm film RTX-5MS column with mass selective detection: (1) diphenylamine, RT = 5.25; (2) di-*n*-butylphthalate, RT = 6.35; and (3) dioctylphthalate, RT = 11.55.

Table I. Tests Using a Small Cartridge Containing Two 10-g XAD-2 Resin Sections*

Compound	%Recovery	%Relative standard deviation
2,6-Dinitrotoluene	95	18.8
2,4-Dinitrotoluene	93	18.1
3,4-Dinitrotoluene†	100	21.9
2,4,6-TNT	101	14.5
RDX	125	17.3
HMX	118	14.3
2-Nitrotoluene	77	16.9
3-Nitrotoluene	94	5.0
4-Nitrotoluene	95	19.3
Nitrobenzene	85	15.6
1,3-Dinitrobenzene	93	17.7
1,3,5-Trinitrobenzene	94	16.5
4-Amino-2,6-dinitrotoluene	102	15.5
2-Amino-4,6-dinitrotoluene	109	14.3
Tetryl	100	18.3
Nitroglycerin	125	17.8
Diphenylamine	91	8.3
Di-*n*-butylphthalate	113	8.4
Dioctylphthalate	109	8.2

* 2.6–2.7 m^3 volume sampled. Seven spikes at 15 µg (energetics) or 75 µg (propellants).
† Surrogate compound. Four replicates were tested.

Table II. Tests Using a Modified PS-1 Cartridge Containing One 50-g XAD-2 Resin Section*

Compound	%Recovery	%Relative standard deviation
2,6-Dinitrotoluene	88	8.1
2,4-Dinitrotoluene	89	7.2
3,4-Dinitrotoluene†	87	6.2
2,4,6-TNT	91	6.9
RDX	101	5.2
HMX	107	17.7
2-Nitrotoluene	99	13.9
3-Nitrotoluene	103	18.8
4-Nitrotoluene	114	17.2
Nitrobenzene	89	9.1
1,3-Dinitrobenzene	87	7.4
1,3,5-Trinitrobenzene	85	7.9
4-Amino-2,6-dinitrotoluene	93	6.6
2-Amino-4,6-dinitrotoluene	103	5.1
Tetryl	96	11.2
Nitroglycerin	104	16.0
Diphenylamine	88	3.9
Di-*n*-butylphthalate	100	3.5
Dioctylphthalate	106	3.3

* 130 m^3 volume sampled. Seven spikes at 100 µg (energetics) or 500 µg (propellants).
† Surrogate compound. Six spikes were done for this compound.

The recoveries were acceptable and no breakthrough was observed in any of the tests of the small cartridges. The effectiveness of desorbing the resin via shaking was tested by multiple extractions of spiked resins. One hour appeared to be sufficient to recover all the analytes, but two hours (followed by standing overnight) is recommended to ensure for full recovery. The shakeout procedure was much simpler than the Sohxlet procedure used for desorbing XAD-2 with methylene chloride as mandated in the EPA organics procedures (11,12). A Sohxlet extraction using isoamyl acetate would be difficult to conduct because of the high boiling temperature of the solvent. Extraction using methylene chloride is not recommended because it is a poor solvent for the nitramine compounds.

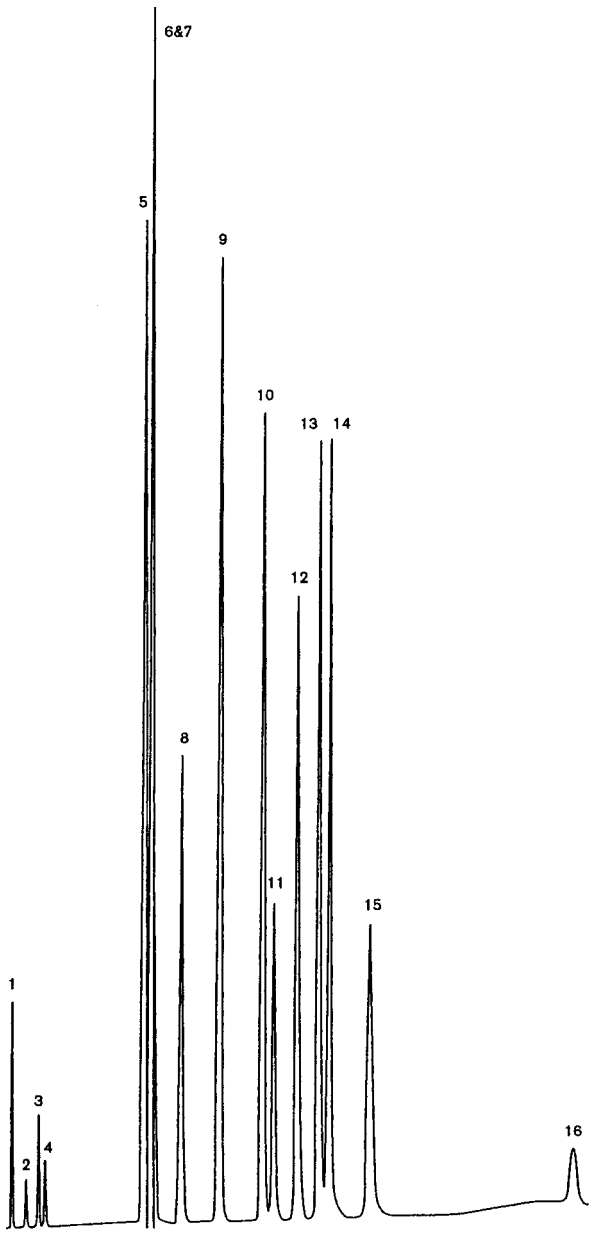

Figure 4. Chromatogram for energetics verification analysis on a 9-m, 0.53-mm i.d., 1.0-µm film DB-210 column with electron capture detection: (1) nitrobenzene, RT = 1.58; (2) 2-nitrotoluene, RT = 1.86; (3) 3-nitrotoluene, RT = 2.12; (4) 4-nitrotoluene, RT = 2.25; (5) 2,6-dinitrotoluene, RT = 4.17; (6) nitroglycerin, RT = 4.31; (7) 1,3-dinitrobenzene, RT = 4.39; (8) 2,4-dinitrotoluene, RT = 4.89; (9) 3,4-dinitrotoluene, RT = 5.66; (10) 2,4,6-TNT, RT = 6.53; (11) 1,3,5-trinitrobenzene, RT = 6.75; (12) 4-amino-2,6-dinitrotoluene, RT = 7.21; (13) 2-amino-4,6-dinitrotoluene, RT = 7.65; (14) RDX, RT = 7.86; (15) tetryl, RT = 8.68; and (16) HMX, RT = 12.67.

Analysis

The chromatographic procedures used to analyze the resin extracts were not complex for most of the analytes, but several potentially required some adjustment of analytical conditions. The nonpolar (DB-1) primary column we used for energetic analysis was capable of separating all of the analytes of interest except PETN in a relatively short time. Most of the compounds were not subject to interferences when used to analyze XAD-2 extracts from ambient air samples. However, there may be occasional background interferences with the peaks for the nitrotoluene isomers or nitroglycerin depending on the lot of resin, isoamyl acetate used, or both. When necessary, a column containing a different liquid phase was used to quantitate compounds that could not be determined with the primary column. Secondary column analysis was also routinely done in order to verify positive detections on the primary column. A polar DB-210 column (J&W Scientific) was useful for this purpose. The temperature program was from 80°C to 240°C, and the carrier gas (H_2) was programmed from 1.5 to 9 psig. Chromatographic conditions can be varied, but a short column is recommended if HMX verification is required (shown in Figure 4). HMX is very reactive; a fast flow rate and temperature program is required to get it through the polar column before peak degradation begins to occur.

One important factor that must be considered when performing GC analyses for energetic compounds is the use of a clean, properly silanized injection-port liner. Commercially prepared liners such as Silcosleeve are recommended. Peaks for the more reactive compounds (especially HMX and the aminodinitrotoluene isomers) will show distorted peak shapes or disappear entirely if the liner is dirty or not silanized. On-column injections are not recommended with this analysis because reproducibility is not as good as with the splitless injections and column life may be shortened.

A 30-m RTX-5ms column is recommended for the propellant compound analysis on the GC–MSD, but a shorter column (10 m) can be used. The only consideration with a shorter column is the separation of the diphenylamine from the isoamyl acetate solvent (there is not much separation between the two). If any of the later-eluting energetic compounds (trinitrobenzene and subsequent) (Figure 2 shows the elution order for energetics on the DB-1 and RTX-5ms columns) are present in the samples, they may be detected during the propellant compound scan if they are present in high enough concentrations. The earlier compounds elute with the solvent front and HMX is not seen. HMX possibly breaks down either when it contacts the metal parts of the detector or is so slowly eluted from the column that its peak flattens out completely (or both).

The reporting limit for the energetics and nitroglycerin based on the lowest injected standard is 0.4 µg for each compound (0.8 µg for HMX) per cartridge for the small cartridge if 40 mL of the desorbing solvent is used. It is 1.0 µg for each compound (2.0 µg for HMX) for the larger cartridge desorbed with 100 mL. Similarly,

the reporting limits for the propellant compounds are 20 µg and 50 µg, respectively, based on their lowest injected standard.

The propellant compound PETN was not included during the conduct of these tests because it was difficult to separate chromatographically from RDX. The two compounds coelute on both the primary and secondary GC columns that were used during the test analyses. Because RDX is a major component of many U.S. high explosives, it was considered the more important compound to evaluate during these cartridge studies. A subsequent check using other chromatographic columns has shown that a DB-1301 column (J&W Scientific) can separate the two compounds. This column can be used when both RDX and PETN are potentially present in a sample. PETN is structurally related to nitroglycerin and thus is probably similarly collected and retained by the XAD-2 resin. PETN spiked onto XAD-2 and desorbed with isoamyl acetate showed good extraction efficiency, but no tests have been conducted using spike cartridges with air pulled through them.

The XAD-2 resin cartridge was designed to collect the analytes of interest in the vapor state, but probably serves as a particulate trap, also. A prefilter can be placed within the cartridge (or in a separate housing before the cartridge) if differentiation between the two physical forms is desired. A filter sampled separately from the resin should be placed in desorbing solvent soon after collection, because many of the compounds of interest will evaporate or sublimate from a filter. A filter included within a cartridge would just transfer evaporating compounds to the adjoining resin. This is the basis of the design of small sampling tubes containing Tenax resin used in industrial hygiene applications involving air sampling for TNT and other substances (9,13).

Conclusion

A sampling and analytical procedure has been devised and validated for the measurement of energetics and related compounds in the atmosphere. The sampling cartridge is a successful modification of other resin-containing devices used for the collection of semivolatile organic compounds in air. GC with electron-capture detection provides a very sensitive and reliable technique for the quantitation of nitro-compounds collected on the sampling media. Other compounds that may be of interest (such as the propellant components described in this study) can easily be quantitated using GC–MSD.

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