TECHNICAL NOTE

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Optimization of Solid-Phase Microextraction (SPME) for the Recovery of Explosives from Aqueous and Post-Explosion Debris Followed by Gas and Liquid Chromatographic Analysis*

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ABSTRACT: Solid-phase microextraction (SPME) has been evaluated for the recovery of explosives residues from aqueous samples and real post-explosion solid debris samples and optimized using gas chromatography with an electron capture detector (GC-ECD) and high-performance liquid chromatography with ultraviolet detection (HPLC-UV). A modified SPME/HPLC interface utilizing dual six-port valves allowed for independent optimization of SPME desorption and injection variables that provided improved chromatographic resolution and sensitivity. A unique combination of cyano and octadecyl columns resulted in the complete separation of the 14 explosives in EPA method 8330 mixture using HPLC with good quantitative results. At the optimum SPME conditions, the limits of detection (LOD) were found to be of 5 ng/mL to 16 ng/mL of explosives in water and 10 μg/kg to 40 μg/kg of explosives from soil. The technique has been successfully applied to the analysis of real post-explosion debris and can be adapted for use in the field utilizing portable chromatographic instruments.

KEYWORDS: forensic science, criminalistics, explosives, post-explosion debris, solid-phase microextraction, high-performance liquid chromatography interface, chromatography explosives residue

The trace analysis of explosives is of major importance in both forensic and environmental applications (1). Analytical techniques for the detection of explosives at the picogram level have been developed for the analysis of samples from post-explosion debris (2,3), or from the illicit transportation of explosives related to international terrorism or other criminal activities (4). For these applications, the matrix often interferes significantly with the detection of the explosives that are present at trace levels. In this respect, several analytical techniques using explosive-specific detectors, such as gas chromatography-electron capture/photoionization detector (GC-ECD/PID) (5), gas chromatography-thermal energy analyzer (GC-TEA) (2,3,6–8), and high-performance liquid chromatography electrochemical detector (HPLC-ECD) (9,10), have been developed and offer acceptable performance in the detection of explosive substances. However, the analytical techniques for the analysis of explosives developed to date still face challenges such as efficient sample preparation and cleanup for samples extracted from complicated matrices and the selection of the best detector for these analytes of interest (8).

The conventional sample preparation methods for extracting explosives from water are liquid-liquid extraction (LLE) (11–13) and solid-phase extraction (SPE) (8,13–15). Although LLE is a commonly used method, the disadvantages include the formation of emulsions, different extraction efficiencies for various compounds, the use of relatively large amounts of solvent, relatively low recoveries, and the amount of time required for use. SPE is extensively used for the concentration of organic compounds on a cartridge and subsequent elution with an adequate solvent. SPE can be relatively expensive, however, with the cartridges usually disposed of after one extraction. The entire analysis can be lengthy with a series of stages, including conditioning, retention, rinse, and elution. In addition, the extraction methods use organic solvents that pose a potential threat to the environment and human health, as well as being rather expensive in their disposal. Solid-phase microextraction (SPME) is a rapid, simple, sensitive, and solvent-free extraction technique, introduced and described in detail by Pawliszyn and co-workers (16–21). The mechanism of SPME involves exposing a fused silica fiber that has been coated with a stationary phase to an aqueous solution containing organic analytes. The analytes partition into the stationary phase until an equilibrium has been reached. The fiber is then removed from the solution and the analytes are thermally desorbed into the injector of a gas chromatograph, or solvent-desorbed via a SPME/HPLC interface connected to an HPLC system. Compared with SPE, SPME uses a coated fused-silica fiber of cylindrical geometry, which can be considered a special format of SPE, allowing fast mass transfer during the adsorption and desorption processes. Thus, SPME preserves all of the advantages of SPE while eliminating the disadvantages of plugging and the use of solvents (17).

In the present study, we evaluate the optimum parameters for the analysis of explosives in aqueous solutions using SPME/GC-ECD and SPME/HPLC-UV. The feasibility of applying this technique is also evaluated by investigating limits of detection, the possibility of

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quantitative analysis, and the precision of the methods for common organic explosives including aromatic nitro compounds (C-NO_2), nitramine compounds (C-N-NO_2), and nitrated esters (C-O-NO_2).

Experimental

Materials

All solvents, including methanol, acetonitrile, and water (Fisher Scientific, Fair Lawn, NJ) in use were HPLC grade. The single explosive standards including 2-nitrotoluene (2-NT), 3-nitrotoluene (3-NT), 4-nitrotoluene (4-NT), nitrobenzene (NB), 1,3-dinitrobenzene (1,3-DNB), 2,4-dinitrotoluene (2,4-DNT), 2,4,6-trinitrotoluene (2,4,6-TNT), 4-amino-2, 6-dinitrotoluene (4-A-2, 6-DNT), 2-amino-4, 6-dinitrotoluene (2-A-4, 6-DNT), 2,4,6-N-tetranitro-N-methyaniline (Tetryl), 1,3,5-trinitro-1,3,5-triazacyclohexane (RDX), nitroglycerin (NG), ethylene glycol dinitrate (EGDN), and pentaerythritol tetranitrate (PETN), were purchased from Radian International LLC (Austin, TX). EPA 8330 explosive standard mixtures (including 1,3,5,7-Tetranitro-1,3,5,7-tetrazacyclooctane (HMX), 1,3,5-trinitrobenzene (1,3,5-TNB), RDX, 1,3-DNB, 2,4,6-TNT, Tetryl, NB, 2,4-DNT, 2,6-dinitrotoluene (2,6-DNT), 2-A-4,6-DNT, 4-A-2,6-DNT, 2-NT, 3-NT, 4-NT) were obtained from Supelco Inc. (Bellefonte, PA). Dilutions of the explosive mixtures were prepared using acetonitrile followed by spiking some amounts of a 25% NaCl aqueous solution.

The real post-explosion residues were collected from the resulting craters after detonations of 5 g quantities of dynamite and C-4 (ICI Explosives, Byron, GA) conducted by the Miami-Dade Police Department Bomb Squad (Miami, FL). Prior to detonation, 2 g samples of range soil were collected and analyzed as a blank. After detonation, 2 g samples of soil from the resulting crater were collected and washed with 5.0 mL of HPLC grade acetonitrile in a clean glass jar manually shaken for 15 min, allowed to settle for 10 min and filtered through a 0.45 μm filter. Seventy-five microliters of the filtered solution were added to 7.5 mL of water (containing 25% NaCl) and extracted by SPME as described below.

SPME/GC-ECD Procedure

A 65 μm film thickness CW/DVB (polyethyene glycol/polydivinybenzene SPME fiber (Supelco, Bellefonte, PA) was first conditioned in a GC injector at 260°C for 30 min. The SPME fiber was immersed into aqueous solutions containing the explosives after adding 25% NaCl with agitation (1000 rpm) for 30 min. The SPME fiber was then retracted into the SPME needle and the assembly was removed from the solution. The needle was inserted into a 0.75 mm SPME Injection Sleeve (Supelco, Inc., Bellefonte, PA) installed in a Hewlett-Packard 5890 II gas chromatograph equipped with an electron capture detector (Wilmington, DE), interfaced to a Peak Simple Data System (SRI, Torrance, CA). The plunger was depressed for 5 min to desorb and transfer the explosives into GC capillary column (DB-5MS, 30 m × 0.25 mm ID, 0.25 μm film) (J&W Scientific, Folsom, CA). Injection was performed in the splitless mode with the split turned on 5 min after the injection. The fiber assembly was cleaned between injections by allowing the fiber to remain in the heated injector of GC for ca. 5 min after the splitter was turned on. Injector (desorption) temperature and ECD detector temperature were isothermally held at 220 and 240°C, respectively. The carrier gas was helium (He, flow rate 57 cm/s, inlet pressure 20 psi). The make-up gas for ECD detector was nitrogen (N_2, flow rate 55 mL/min). The column temperature was held at 60°C for 1 min, then ramped at 12°C/min up to 240°C and held for 9 min.

SPME/HPLC-UV Procedure

For SPME/HPLC coupling, the extraction procedure was the same as that used for SPME/GC-ECD except that a CW/TPR (polyethylene glycol/template polydivinybenzene resin) fiber (Supelco, Inc., Bellefonte, PA) was used. The main difference between the SPME/HPLC interface and the SPME/GC interface is the desorption procedure. Solvent desorption was used for the HPLC method instead of thermal desorption used for the GC method. A modified SPME/HPLC interface using dual six-port valves (Valco, Houston, TX) and a 200 μL inner volume SPME desorption chamber (Supelco, Inc., Bellefonte, PA) depicted in Fig. 1*a,b,c* was used. The SPME CW/TPR fiber was conditioned

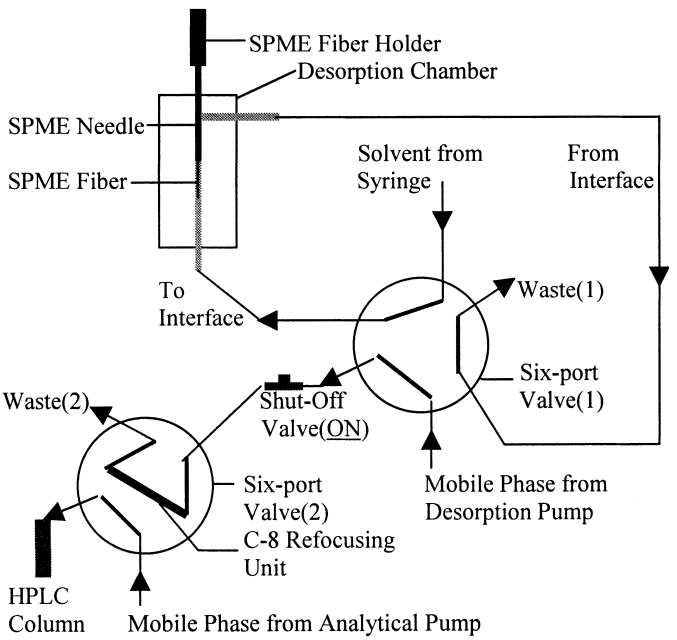

FIG. 1*a—Static desorption—Valve (1) and Valve (2) at "loading position."*

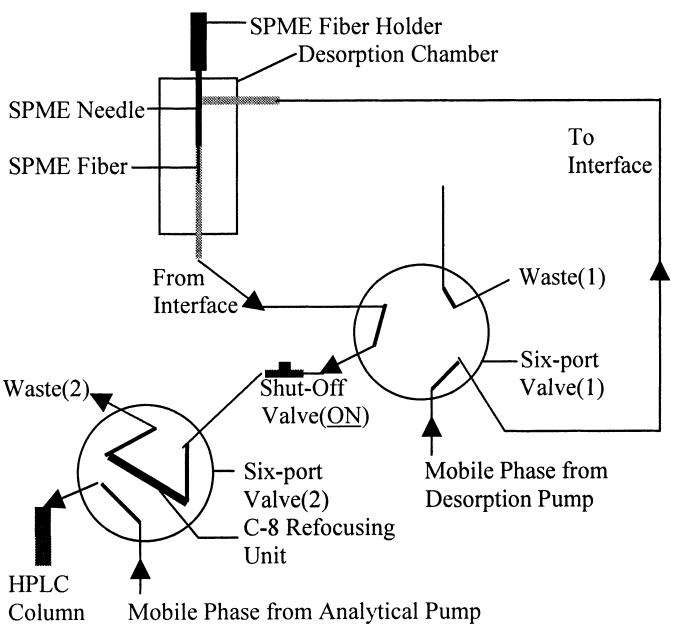

FIG. 1*b—Sample loading—Valve (1) at "injection position" and Valve (2) at "loading position."*

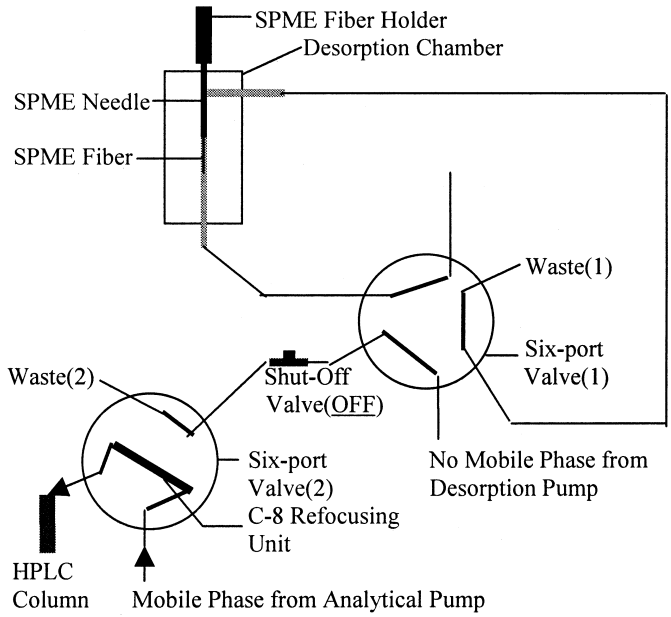

FIG. 1*c—Sample injection—Valve (1) at "loading position" and Valve (2) at "injection position."*

in the SPME desorption chamber with the mobile phase of 1:1 methanol:water at the flow rate of 0.2 mL/min passing through the fiber for 30 min.

Prior to transferring the fiber into the desorption chamber, the six-port valve was placed in the "static position." The fiber was inserted through the ferrule and introduced into the desorption chamber under ambient pressure. Two hundred microliters of a 1:1 methanol:water mixture was injected into the desorption chamber by a syringe to soak the fiber with the solvent for 2 min. The desorbed analytes were then transferred to a C-8 refocusing unit (3.0 cm × 4.6 mm ID, 8 μm, 100Å) (Varian, Harbor City, CA) by the desorption pump (SSI, State College, PA) at a flow rate of 0.2 mL/min for 3 min. The valve was then switched back to the original position (sample injection position) and the analytical pump (Waters, Model 600E, Milford, MA) carried the desorbed analytes from the C-8 refocusing unit to the analytical columns consisting of a combination of a Res-Elut CN column (3.0 cm × 4.6 mm ID, 5 μm) and a Bondesil C-18 column (25 cm × 4.6 mm ID, 5 μm) (Varian, Harbor City, CA) for separation. All experiments were performed at isocratic and constant-flow conditions. The UV detector (Waters, Model 484, Milford, MA) was interfaced to Peak Simple Data System (SRI, Torrance, CA), operated at the wavelength of 220 nm for EGDN, NG, and PETN and 254 nm for all of the other explosives.

Results and Discussion

The fiber coatings of CW/DVB and CW/TPR yielded the highest sensitivity and selectivity for the explosives studied. The amount of explosives extracted by SPME increased with decreasing acetonitrile (CH_3CN):water (H_2O) ratio in aqueous solutions and increasing salt concentration. The CH_3CN:H_2O ratio effect was expected based on the known hydrophobicity of the explosives expressed as octanol/water partition coefficients (K_{ow}) (22). The highest SPME recovery was found from solutions with the lowest acetonitrile concentration. Real post-explosion residues were extracted with a pure acetonitrile rinse followed by dilution into water with a CH_3CN:H_2O of 1:199. The addition of either NaCl or Na_2SO_4 had positive effects on increasing peak areas of extracted explosives and minimizing %RSD; however, NaCl salting proved better than Na_2SO_4 salting at the same ionic strength. This indicates that the salting effect depends on not only the ionic strength of the solution, but also the concentration, the charge(s), and the size of the ions.

Quantitative analysis of the explosive standards proved problematic due to the thermal instability of these high explosives. For the SPME/GC-ECD method, there was no significant chromatographic improvement observed employing oven cryofocusing using liquid CO_2 or increasing the column flow rate to shorten the analysis time. Use of the solvent flush technique improved detection limits over normal injection (25,26), but SPME significantly improved the GC/ECD detection limits as summarized in Table 1. The desorption temperature of 220°C was used to rapidly and efficiently desorb all explosives except RDX and provide excellent chromatographic resolution as seen in Fig. 2. Explosives by their very nature are thermally unstable to varying degrees. This presents an inherent problem when they are analyzed by gas chromatography, especially for explosives with very low vapor pressures such as PETN and RDX. Nitroaromatics, which normally have fairly high vapor pressure, are more stable than nitramines and nitrate esters, since the C-NO_2 bond is more stable than the C-N-NO_2 or C-O-NO_2 bond. The deflagration temperatures (at which sudden decomposition takes place) of TNT, RDX, PETN, NG, and EGDN are shown in Table 2. The thermal instability of organic explosives must be carefully considered in developing optimal conditions for their analysis by GC (7,11,27,28).

The equilibrium vapor pressures of the common explosives at standard conditions of temperature and pressure are generally very low and vary substantially. For example, there are five orders of magnitude difference in the vapor pressure of EGDN and RDX as seen in Table 2 which shows the equilibrium vapor pressures of TNT, RDX, PETN, and NG at 1 atm and various temperatures (1,7,11,27,29–31). Vapor pressure gives a general indication of the retention order in gas chromatographic separations with the EGDN eluting first and RDX eluting late in the separation, as seen in Fig. 2. Recoveries of explosive standards spiked on soil samples were estimated by comparing the peak areas using the SPME/GC method at the optimum conditions from the simulated soil sample with that from the explosive standard mixture, and from 52.1% (EGDN) to 76.2% (2,4-DNT) were obtained. Detection limits of 5 ng/mL to 16 ng/mL explosives in water and 10 μg/kg to 40 μg/kg explosives in soil were achieved.

Modification of the conventional SPME/HPLC interface (23) (shown in Fig. 1) provided improved desorption efficiency of SPME/HPLC (24) and the combination of cyano and octadecyl columns yielded excellent resolution for all of the explosives, including HMX, as seen in Fig. 3. The SPME/HPLC method also yielded excellent quantitative results with correlation coefficients of 0.9985 for TNT and 0.9971 for RDX for concentrations from 10 ng/mL to 1000 ng/mL.

Finally, the SPME/GC-ECD and SPME/HPLC-UV techniques were successfully applied to the analysis real post-explosion debris samples as illustrated in Figs. 4–7 for TNT and RDX for detonations of 5 g quantities of each explosive. The significant thermal decomposition of RDX relative to TNT using the GC method is evident comparing the GC-ECD chromatograms (Figs. 4 and 5) using identical scales for the response axis (for comparative purposes) to the HPLC-UV chromatograms (Figs. 6 and 7) also plotted with the

TABLE 1—*Comparison of detection limits and RSDs (for five replicate samples) of explosives by gas and liquid chromatographic methods.*

	Detection Limits (ng/mL) (S/N>3)				
	GC/ECD			LC/UV	
	Direct Injection				
Explosives	Normal	Solvent-Flush	SPME/GC	Direct Injection	SPME/HPLC
HMX****	N/A	N/A	N/A	3.8 (2.6) [a]	1.2 (2.7) [a]
RDX***	175	130 (5.0)	0.61 (5.2)	2.9 (2.3) [a]	1.1 (2.5) [a]
1,3,5-TNB	48	39 (3.7)	0.18 (2.1)	2.6 (1.1) [a]	0.6 (1.2) [a]
Tetryl**	65	43 (4.0)	0.25 (4.2)	3.2 (2.8) [a]	1.3 (2.9) [a]
1,3-DNB*	60	44 (2.7)	0.22 (2.7)	2.3 (1.2) [a]	0.8 (1.2) [a]
TNT	28	22 (1.7)	0.09 (1.7)	2.9 (1.2) [a]	1.1 (1.3) [a]
4-A-2,6-DNT*	38	30 (1.7)	0.10 (1.6)	2.9 (1.6) [a]	1.2 (1.6) [a]
NB	67	53 (3.3)	0.24 (3.5)	3.9 (2.7) [a]	1.2 (2.7) [a]
2-A-4,6-DNT*	35	29 (1.4)	0.09 (1.4)	3.4 (2.1) [a]	1.2 (1.9) [a]
2,6-DNT*	34	27 (1.9)	0.09 (1.5)	4.4 (3.0) [a]	1.3 (2.9) [a]
2,4-DNT*	36	29 (1.5)	0.10 (1.7)	3.4 (1.9) [a]	1.2 (1.7) [a]
2-NT*	64	55 (3.2)	0.24 (.34)	8.0 (3.7) [a]	1.8 (3.8) [a]
4-NT*	60	47 (2.7)	0.24 (2.8)	7.4 (4.1) [a]	1.9 (4.2) [a]
3-NT*	75	64 (3.1)	0.24 (2.9)	7.3 (4.0) [a]	1.7 (4.0) [a]
EGDN**	68	51 (3.7)	0.22 (3.8)	550 (5.4) [b]	120 (4.6) [b]
NG**	97	89 (2.8)	0.58 (2.9)	500 (5.5) [b]	110 (4.8) [b]
PETN***	123	94 (3.7)	0.61 (3.8)	380 (5.1) [b]	80 (4.3) [b]

**** Completely decomposed; *** Significantly decomposed; ** Moderately decomposed; * Slightly decomposed at GC experiment conditions by SPME/GC-ECD method. For SPME method, all samples in 25% NaCl aqueous solutions and acetonitrile/water ratio was 1:199. [a]UV detection at 254 nm. [b]UV detection at 220 nm.

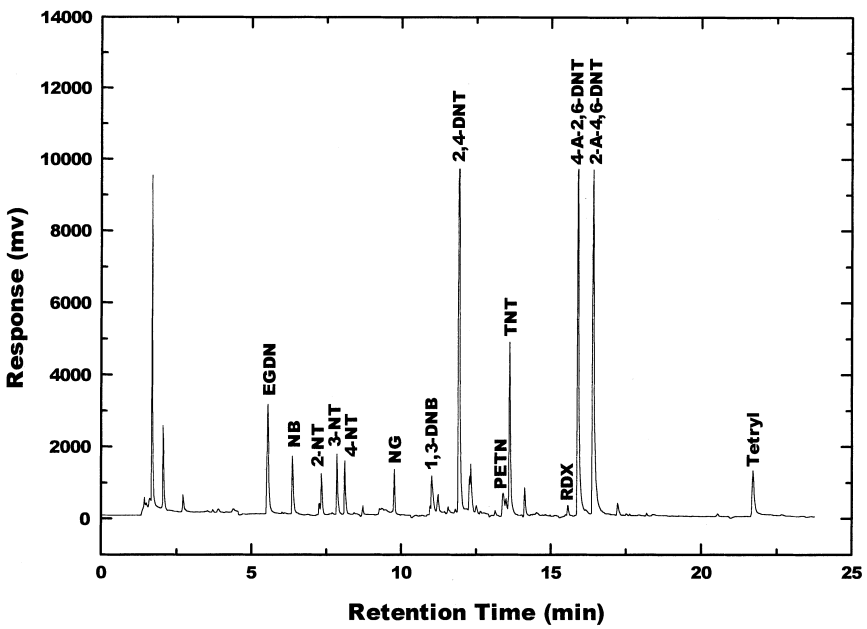

FIG. 2—*Chromatogram of SPME/GC-ECD of 14 explosive standard mixture. (500 pg/mL each in 25% NaCl aqueous solution and $CH_3CN:H_2O$ ratio of 1:199.)*

TABLE 2—*Deflagration temperature and vapor pressures (at 1 atm) of high explosives.*

Explosives	Deflagration Temperature (°C)	Vapor Pressure (Torr)* 25°C	Vapor Pressure (Torr)† 100°C	Vapor Pressure (Torr)† 200°C
TNT	>333	7.1×10^{-6}	6.9×10^{-2}	360
RDX	>229	4.6×10^{-9}	1.6×10^{-4}	3.1×10^{-1}
PETN	>209	1.4×10^{-8}	8.0×10^{-4}	26.9
NG	>200	4.4×10^{-4}	3.9×10^{-1}	78.6
EGDN	>200	2.8×10^{-2}	22.2	2582

* Recalculated from Ref 32.
† Recalculated from Ref 33.

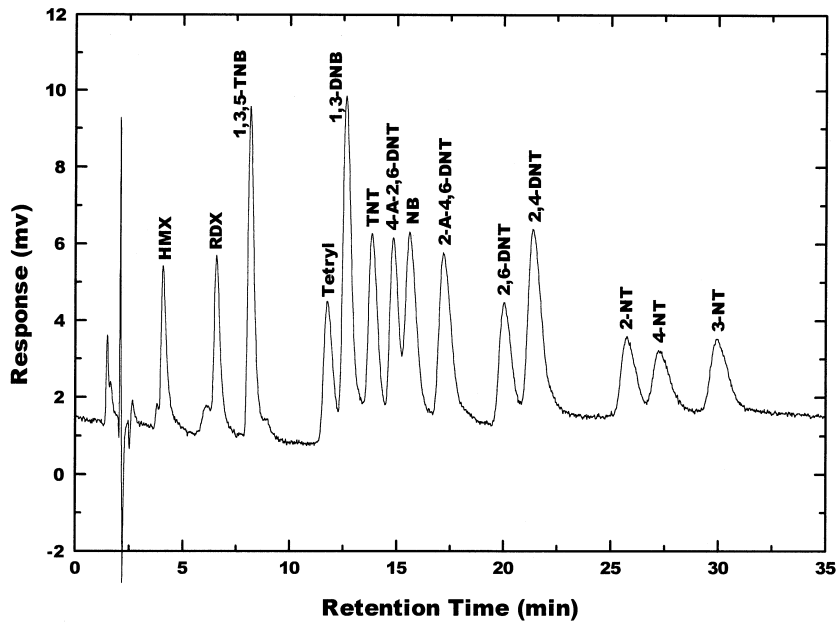

FIG. 3—*Chromatogram of SPME/HPLC-UV of EPA 8330 mixture standard. (20 ng/mL each in 25% NaCl aqueous solution and $CH_3CN:H_2O$ ratio of 1:199.)*

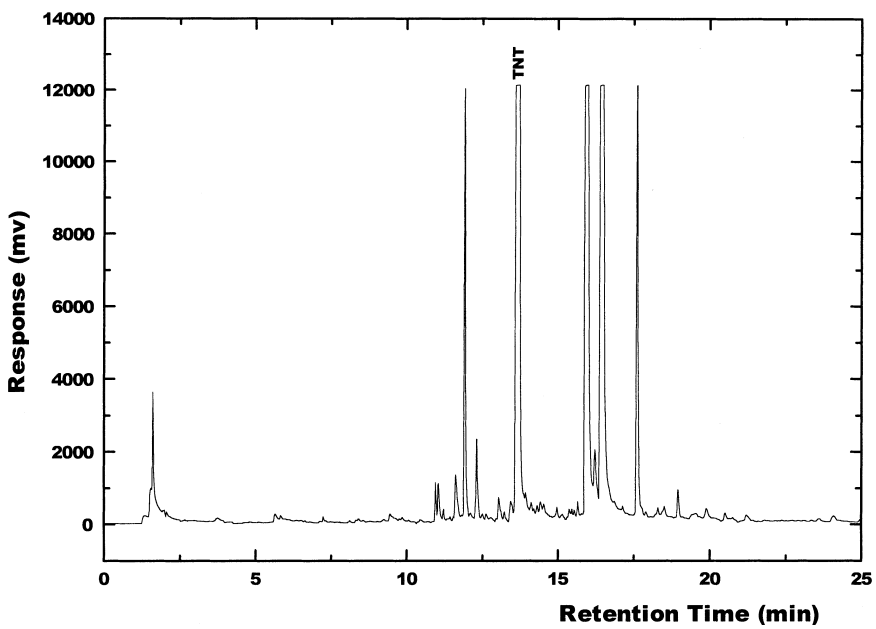

FIG. 4—*Chromatogram of SPME/GC-ECD of TNT in a post-explosion soil sample from the crater produced after 5 g of TNT were detonated.*

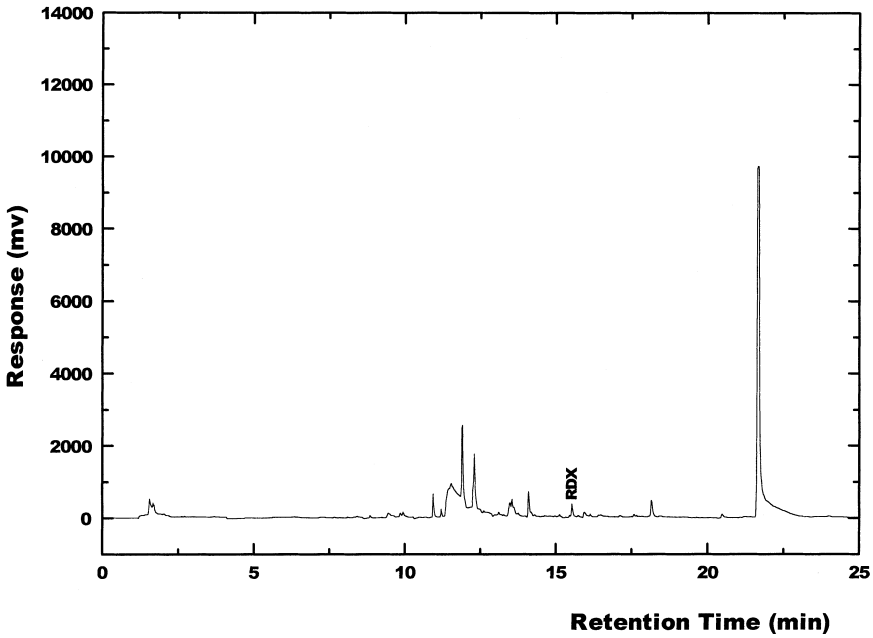

FIG. 5—*Chromatogram of SPME/GC-ECD of RDX in a post-explosion soil sample from the crater produced after 5 g of RDX were detonated.*

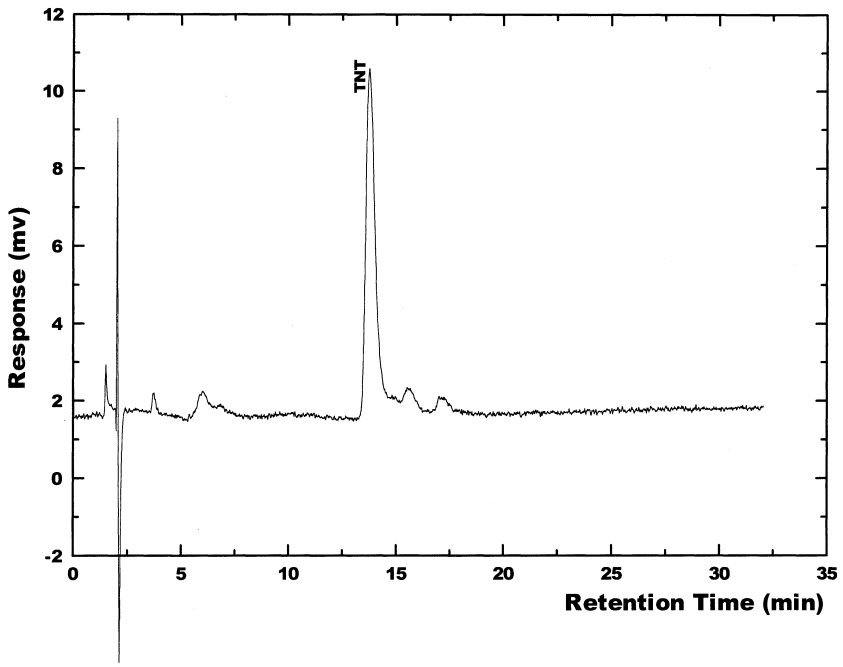

FIG. 6—*Chromatogram of SPME/HPLC-UV of TNT in a post-explosion soil sample from the crater produced after 5 g of TNT were detonated.*

same response axis. The RDX peak is nearly undetectable under the GC conditions employed, yet identical to TNT using HPLC analysis indicating the difference is due to the chromatographic process (thermal decomposition at the elevated temperatures required for gas chromatography) and not differences during explosion or sample manipulation. It is also notable that the chromatograms in Figs. 6 and 7 are relatively simple with the major peak identified as TNT for the dynamite sample and RDX for the C-4 samples, respectively (the minor peaks were not identified). Using the same method, no explosives were detected from the blank soil samples taken prior to detonation of the explosives.

Conclusions

This study demonstrates that SPME is a rapid, precise, reproducible, and sensitive means for the extraction of explosives from water and soil. The optimum conditions for extracting explosives to obtain a high recovery are at low CH_3CN:H_2O ratios and high NaCl salting concentrations. A modified SPME/HPLC interface has been developed to improve SPME/HPLC desorption efficiency and chromatographic resolution. Limits of detection at the ppb level for the determination of explosives in water using SPME/GC-ECD and SPME/HPLC-UV were achieved.

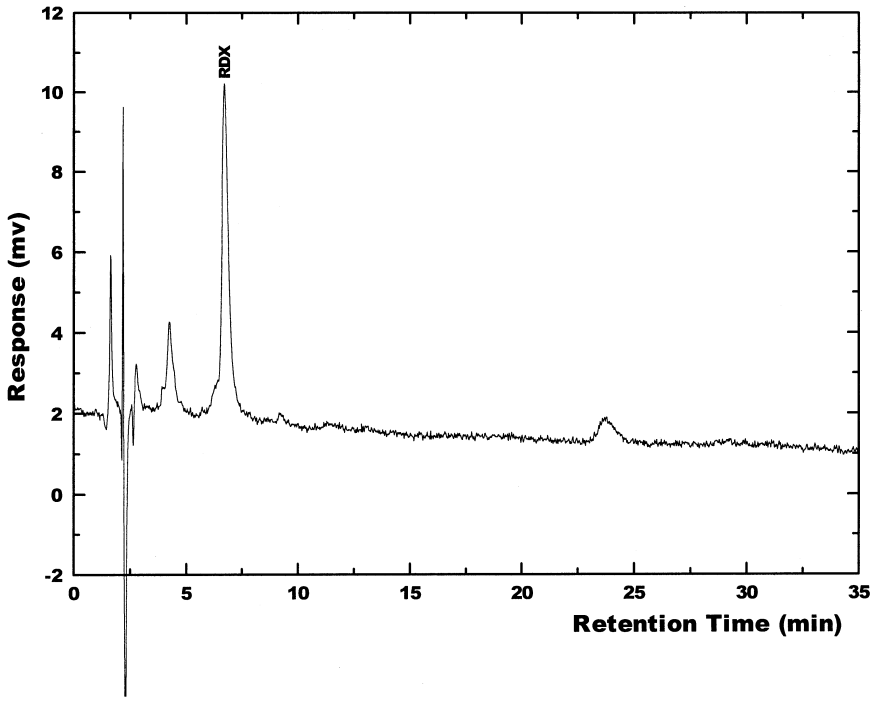

FIG. 7—*Chromatogram of SPME/HPLC-UV of RDX in a post-explosion soil sample from the crater after 5 g of RDX were detonated.*

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