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Picogram Analyses of Explosive Residues Using the Thermal Energy Analyzer (TEA®)

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ABSTRACT: The thermal energy analyzer (TEA®), interfaced to both a gas and a high performance liquid chromatograph, has been shown to be selective to nitro-based explosives at a sensitivity of 4 to 5 pg injected on-column. Analyses of "real world" explosives, post-explosion debris, handswabs, and human plasma are presented. Because of the selectivity of the technique, there was no need for sample cleanup before analysis.

KEYWORDS: criminalistics, explosives, trinitrotoluene, RDX, NG, vasodilators, PETN, EGDN, TEA® analyzer, gas chromatography, high performance liquid chromatography, post-blast residue, hand swabs, picogram detection

The analysis of explosives is of importance in several analytical areas. In forensic science there is a need to analyze pieces of explosives [1], post-explosion debris [2-4], and handswabs [1,5-7] from persons who may have recently handled explosives or fired a gun. The discovery that some military explosives are mutagenic [8] has increased the environmental need to protect factory workers and to monitor wastewater from the manufacture of explosives [9-11]. In medicine, there is an increasing need to monitor vasodilators such as pentaerythritol tetranitrate (PETN), glycerol trinitrate (NG), isosorbide dinitrate (ISDN), and their metabolites in human blood [12,13].

The ideal technique for the ultra-trace analysis of explosives would be both simple and rapid, require minimal sample cleanup, be sensitive to as little as 1 to 10 pg (picograms, 10^{-12} g) quantities of all the compounds of interest, and would work equally well on both complex samples from the real world and on high-grade laboratory standards made up in pure solvents. Three detection techniques for gas chromatography, electron capture [5-7,14], mass spectrometry [13,15-18], and the thermal energy analyzer (TEA®) [12,13,19-21], have the required sensitivity. However, the most difficult problem in determining the presence of low picogram quantities of material on real world samples is detector selectivity.

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Because selectivity implies lack of response for co-eluting compounds, in the analysis a common chemical or physical aspect must be used which distinguishes explosives from other chemicals that might be found coincidentally in the specimen examined. A distinguishing facet of most explosives of commercial, military, and biomedical interest is the nitro ($-\text{NO}_2$) functional group. It can be attached to a carbon atom as in trinitrotoluene (TNT), to a nitrogen atom as in cyclo-1,3,5-trimethylene-2, 4, 6-trinitramine (RDX), or to an oxygen atom as in PETN.

There is a similarity between the analytical problems faced in the late 1970s by chemists trying to measure environmental levels of the carcinogenic *N*-nitrosamines and today's problem of explosive residue analysis. First, as with explosives, *N*-nitrosamines have a common functional group, the NO moiety. The NO_2 that is produced by explosives under conditions of catalytic pyrolysis can readily be converted into NO. Second, electron capture and *N*-specific detectors have adequate sensitivity for *N*-nitrosamines, but, as with explosives, they lack the necessary specificity. While mass spectrometry can be both sensitive and selective, the output requires a large degree of human interpretation by a skilled analyst to be reliable. Third, the *N*-nitrosamine problems were resolved [22-25] by the development of a nitrosyl-specific detector, called the TEA analyzer.

The possible application of the TEA analyzer to the problem of the analysis of explosives has been addressed recently [12, 13, 19-21]. The performance of the TEA analyzer as a detector of explosives in high performance liquid chromatography (HPLC) [26] and gas chromatography (GC) [19] has been described. Three laboratories [12, 13, 27] have developed procedures for the routine analysis of nitrate esters such as NG, ISDN, and PETN in blood at levels as low as 100 pg/mL. A comparison study of the TEA analyzer with three other GC detectors: electrolytic conductivity, thermionic, and electron capture, has been made for the analysis of nitroaromatics such as nitrobenzene, and the dinitrotoluenes in sludge wastes [21]. A recent report demonstrated the low picogram detection of explosives by using silica capillary column GC with a TEA analyzer [28]. This paper expands the applicability of the TEA analyzer for trace explosive residue analysis, to the low picogram level, on real world samples such as pieces of explosives, post-blast residue and handswabs. It is also shown that by using parallel HPLC-TEA and GC-TEA techniques it is possible to increase greatly the selectivity of a procedure.

Experimental Procedure

Reagents

All solvents were of a grade that had been distilled in glass (Burdick and Jackson). The explosives used in this study were glycerol trinitrate (NG), pentaerythritol tetranitrate (PETN), ethylene glycol dinitrate (EGDN), 2,4-dinitrotoluene (2,4-DNT), 2,4,6-trinitrotoluene (TNT), cyclo-1,3,5-trimethylene-2,4,6-trinitramine (RDX), trinitro-2,4,6-phenylmethylnitramine (tetryl), and cyclotetramethylene tetranitramine (HMX).

GC-TEA

A gas chromatograph (Hewlett Packard, Model 5840A), equipped with an on-column injector (SGE Scientific, Model OCI-3) was used. The fused silica capillary column (DB-5) was 30 m long, 0.32-mm inside diameter, and had a 0.25- μm film thickness. The carrier gas was helium at a head pressure of 124 kPa (18 psi). The injection port temperature was ambient. The oven temperature was held at 60°C for 1 min, and then increased at 15°C/min to 240°C, and then held at 240°C for 3 min. The detector was a TEA analyzer (Thermo Electron, Model 610), operating in the nitro mode. The interface temperature was 285°C and the pyrolyzer temperature was 900°C. The reaction chamber was held at 1.8 mm Hg, with an O_3

flow of 5 mL/min. The cold trap was maintained at -100°C with a slush bath of ethanol and liquid nitrogen. The amount of material injected on column was 0.2 to 1.0 μL .

HPLC-TEA

The high performance liquid chromatographic system consisted of a solvent pump (Altex, Model 110) with an injector (Waters Associates, Model U6K). The column was a 10- μm uBondapak CN, 30 cm long by 3.9-mm inside diameter (Waters Associates). For most of the work, two detectors, connected in series, were used. The column effluent flowed first through a variable wavelength ultraviolet (UV) detector, set at 254 nm (Schoeffel, Model 770) and then into a TEA analyzer (Thermo Electron, Model 510). Some of the data were collected with only the TEA analyzer. For screening 2,4-DNT, EGDN, TNT, NG, PETN, tetryl, and RDX, the solvent system was isooctane/methylene chloride/methanol in the ratio 165/35/10. For screening NG, PETN, RDX, and HMX, the ratio of solvents was 60/30/10. The solvent flow rate was maintained at 1.5 mL/min. Typically, the amount injected, on column, was 25 μL . The TEA catalytic pyrolyzer was operated at 550°C . The reaction chamber vacuum was 1.8 mm Hg, with an O_3 flow rate of 5 mL/min. The TEA carrier gas was N_2 , at a flow rate of 20 mL/min. The TEA cryogenic trap was maintained at -78°C with a slush bath of ethanol and solid carbon dioxide.

Description of the TEA Analyzer

The TEA itself, and its detailed principle of operation have been described previously [19,22,23,26]. Effluent from the chromatograph enters a catalytic pyrolyzer, where NO_2 is released from organic nitro compounds and simultaneously converted into NO by the catalytic surface. Solvent vapors and pyrolysis products are then removed by a cold trap which is maintained at about -100°C . The NO gas which survives the trap is reacted with ozone (O_3) in the reaction chamber at reduced pressure to produce the characteristic infrared chemiluminescent reaction, the intensity of which is monitored by an infrared-sensitive photomultiplier tube. While the technique is sensitive at the picogram level, it is also highly selective. The rejection ratio of the TEA to hydrocarbons and *N*-containing organics is greater than 10^6 to 1 [23]. A partial list of compounds that have been shown to give no response on the TEA is shown in Fig. 1. The selectivity stems from four factors. First, only compounds that have the NO_2 or NO functional group can give a response. Second, the reactive species must survive the -100°C cold trap. For highly contaminated samples, a -160°C trap can be used. Third, the reactive species must react with O_3 to produce a chemiluminescent light in the narrow wavelength range of 0.6 to 2.8 μm . Fourth, the reaction with O_3 must be rapid enough to occur while the effluent is in the reaction chamber, and not in the vacuum pump.

Linearity

The detector has been shown to be linear over six orders of magnitude [23].

Precision

For the HPLC-TEA, data on the precision has been demonstrated using NG, PETN, and ISDN at the 1-, 5-, 20-, and 50-ng level injected on column [12]. At the 1-ng injection level, for example, the relative standard deviations were $\pm 2.3\%$ for NG, $\pm 5.9\%$ for PETN, and $\pm 8.6\%$ for ISDN. For capillary column GC-TEA, the precision, expressed as relative standard deviations, that was attained over five injections of approximately 1 ng on column was $\pm 1.6\%$ for EGDN, $\pm 1.4\%$ for NG, $\pm 2\%$ for 2,4-DNT, $\pm 1.3\%$ for TNT, $\pm 5.7\%$ for

REPRESENTATIVE LIST OF COMPOUNDS WHICH WERE FOUND TO
GIVE NO INTERFERENCE ON THE TEA

Acetic acid	Ethyl acetate	Oxalic acid
Acetone	Ethyl carbamate	n-Pentane
Acetonitrile	Ethylene glycol	Phenyl hydrazine
Alizarin red	Fluorobenzene	d,1-Phenylalanine
Ammonia (gas)	Gasoline	p-Phenylazoaniline
Benzene	Glycerol	Phosphoric acid
Benzylsalicylate	d-Glucose	Propane (gas)
2-Butoxy ethanol	Glutamic acid	Pyridine
Carbon dioxide	n-Hexane	Quinine
Carbon disulfide	Hydrogen (gas)	Sodium acetazolamide
Carbon monoxide (gas)	Hydroquinone	Sulfadiazine
Carbon tetrachloride	8-Hydroxyquinoline	Sulfanilic acid
Chloral hydrate	Inosine	Tetrahydrofuran
Chlorobenzene	d,1-iso-leucine	Theophylline
1-Chloropropane	Methane (gas)	Toluene
2-Chloropropane	Methyl acetate	2,4,6-Trichlorophenol
Cyclohexane	N-Methyl bisacrylamide	2,2,4-Trimethylpentane
Cyclopentane	2-Methyl butane	d,1-Tryptophane
1,2-Dichloroethane	Methyl formamide	Urea
2,3-Dichloropropane	Methyl isobutyl ketone	Uric acid
Diethylether	Methyl orange	Urethane
Dimethylamine (gas)	Methyl red	Water
p-Dioxane	Naphthalene	Xylene
Diphenylamine	Nitrogen (gas)	

FIG. 1

RDX, and $\pm 2.6\%$ for tetryl. The precision at the 0.1 ng was also conducted, and the results are presented in Table 1.

Sensitivity

The sensitivity attainable on the capillary column GC-TEA is demonstrated by the three chromatograms shown in Fig. 2. Figure 2a is the chromatogram for 10 pg of NG, 8 pg of TNT, and 7 pg of RDX, introduced on column. The three peaks are clearly discernible above the background. The minimum detectable level at a signal-to-noise ratio of 3/1, is estimated to be 4 pg for TNT and RDX; 5 pg for EGDN, NG, and DNT; and 25 pg for tetryl. Although the chromatograms of Fig. 2 were obtained with standard solutions, little degradation in performance is observed when analyzing complex explosive residue samples.

TABLE 1—Precision of capillary GC-TEA on
explosives at the 1- and 0.1-ng level (n=5).

Compound	Amount Injected, ng	Percent of Relative Standard Deviation
EGDN	1.2	1.6
	0.12	4.9
NG	0.8	1.4
	0.08	5.1
2,4-DNT	1.1	2.0
	0.11	5.2
TNT	1.3	1.3
	0.13	6.5
RDX	1.0	5.7
	0.10	7.2
Tetryl	1.7	2.6
	0.17	11

Peak	Amount A.	Injected B.	Picograms C.
① NG	10	20	50
② TNT	8	16	40
③ RDX	7	14	35

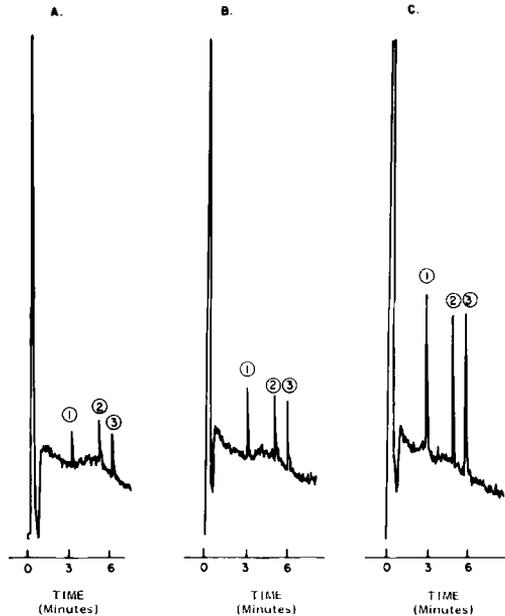


FIG. 2.—Sensitivity of capillary column GC-TEA for 0.2- μ L injection of NG (Peak 1), TNT (Peak 2), and RDX (Peak 3). The amount of material introduced on-column was (a) NG-10 pg, TNT-8 pg, and RDX-7 pg; (b) NG-20 pg, TNT-16 pg, and RDX-14 pg; and (c) NG-50 pg, TNT-40 pg, and RDX-35 pg.

Parallel GC-TEA/HPLC-TEA Confirmation

The operation of a selective detector with both GC and HPLC, offers a novel self-confirmatory capability. In GC, separation of the compounds is achieved by differences in vapor pressure and solubility in the liquid phase of the column. In HPLC, however, polarity, physical size, and shape characteristics determine the chromatographic selectivity. The result is that the elution order of the explosives is different on GC and on HPLC. Thus, an analysis that is highly specific could be interpreted as confirmatory by an examiner if three criteria are met:

- (1) the peak elutes at the proper retention time on both GC-TEA and HPLC-TEA,
- (2) both chromatograms are relatively clean, and
- (3) identical quantitation is achieved on both systems.

If some doubt exists because of multiple peaks, the compound can be isolated off HPLC-TEA by collecting the effluent at the retention time indicated by a previous HPLC-TEA run before the compound enters the pyrolyzer, concentrated, and reinjected on both HPLC-TEA and GC-TEA. A single peak of the proper quantitation eluting at the proper retention time, can be taken as confirmatory. Data, based on this principle, are presented later in this paper. Parallel GC-TEA/HPLC-TEA determinations have been used successfully in the *N*-nitrosamine field, when the amount of sample was too small to be handled by other methods [24,25], and have been interpreted by workers in that field as confirmatory.

Results and Discussion

Explosives

Small pieces of military and commercial explosives were dissolved in acetone to a concentration of 1%. The samples were then diluted in methanol to obtain a 10-ppm (weight/volume) solution. No cleanup was used.

Parallel GC-TEA and HPLC-TEA chromatograms are shown in Fig. 3*a* and *b* for a sample of gelatin dynamite, and in Fig. 3*c* and *d* for double-based smokeless rifle powder. The dynamite sample gave only two peaks, one of NG and the other of EGDN. The double-based smokeless powder sample gave only a single peak of NG. Nitrocellulose, another basic ingredient in smokeless powder, was not eluted under the chromatographic conditions used. Constituents other than the explosive components did not interfere with either the GC-TEA or HPLC-TEA analyses.

Figure 4*a*, *b*, and *c* are the HPLC-UV-TEA chromatograms for C-4, Flex-X, and a sample cut from a defused letter bomb of Middle East origin. For C-4, RDX is the major peak, with a small amount of HMX also being present. Flex-X was shown to contain only PETN. PETN was also the only explosive found to be present in the letter bomb.

Although all five samples undoubtedly contained a multitude of materials other than the explosive constituents (plasticizers, and so forth), no extraneous peaks were observed, even though no cleanup was used before analysis.

Postblast Debris

Postblast debris was collected from three test bombs (TNT, C-4, and detonating cord), that were detonated by the FBI at the U.S. Marine demolition range at Quantico during January 1983. The bombs were made by placing the explosive inside a 151-L (40-gal) metal trash can, with a stone weight on the lid. Military blasting caps, equivalent to the No. 8 civilian blasting caps and equipped with a safety fuse, were used to detonate the devices. For TNT, a 0.5-kg (1-lb) demolition block was used; for C-4, a 0.6-kg (1 1/4-lb) charge; and for the detonating cord alone, about 4.5 m (15 ft) was wrapped around the can. After detonation, about 500 g of debris including metal fragments, soil, and fabric were collected and sent to Thermo Electron Corp. for analysis.

About 50 g of assorted debris was placed in a beaker (the metal fragments had to be cut so that they would fit into the beaker). After sonication in methylene chloride for 10 min, the methylene chloride was concentrated to 15 mL on a rotary evaporator, filtered through a Millex-SR filter, and then concentrated under a stream of N₂ gas to 2 mL. Aliquots were then analyzed by both GC-TEA and HPLC-TEA.

Figure 5*a* and *b* showed the GC-TEA and HPLC-TEA analyses of the debris of the TNT bomb. Although TNT was expected to be the major component from the post-blast debris, it was determined to be only 0.3%. On GC-TEA, the amount present in the 1- μ L injection of the debris extract was 0.1-ng EGDN, 4.4-ng NG, 0.15-ng 2,4-DNT, 0.03-ng TNT, and 0.5-ng RDX. Since 2,4-DNT is usually present as an impurity in TNT and is also a degradation product of TNT, its presence in the debris is to be expected. The detection of RDX in the sample could possibly be attributed to the military blasting cap that was used, where RDX was incorporated as a primary booster. For the detonating cord debris (Fig. 5*c* and *d*), the GC-TEA chromatogram showed a trace of EGDN and a considerable amount of NG. The HPLC-TEA showed NG, as well as a trace of PETN (PETN partially decomposed in the GC). As for debris from the C-4 bomb, shown in Fig. 6, 9 ng of RDX, 7.8 ng of PETN, and 5 ng of NG were found in a 20- μ L injection on HPLC-TEA.

The presence of NG in the debris is intriguing, but initial GC-TEA results were re-examined by HPLC-TEA analysis, showing NG to be present in equivalent quantities by the two approaches. The finding may be taken as confirmatory as the criteria set forth earlier are

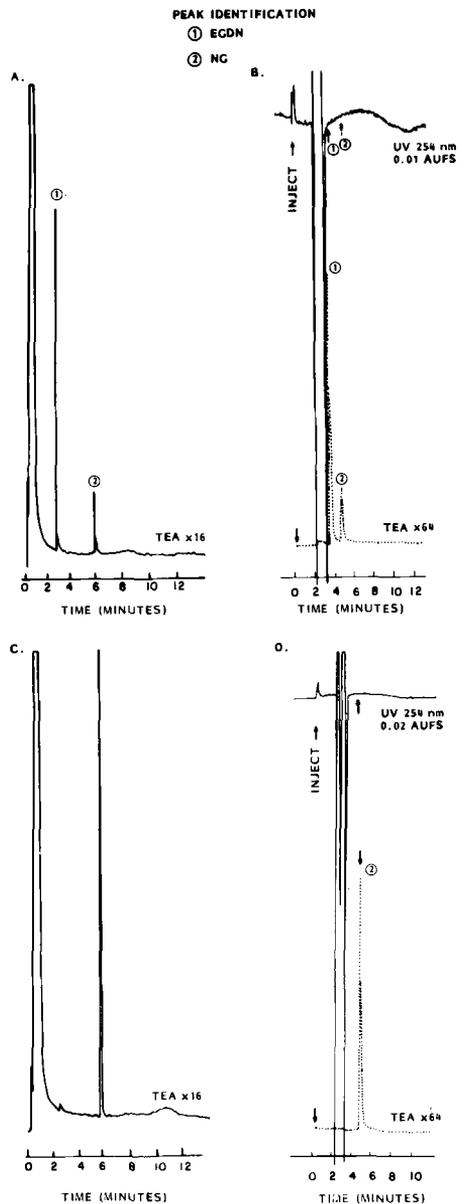


FIG. 3—Chromatograms on GC-TEA (a), and HPLC-UV-TEA (b) of a 10-ppm extract from gelatin dynamite. GC-TEA was obtained with 1- μ L injection, with Peak 1 being 0.65 ng of EGDN and Peak 2 of 0.25 ng of NG. For HPLC, 20 μ L was injected on-column. The EGDN peak was 13 ng, and the NG peak was 4.9 ng. Chromatograms of GC-TEA (c) and HPLC-UV-TEA (d) of 10-ppm extract of double-based smokeless rifle powder. For GC-TEA, a 0.5- μ L injection was used, with the NG peak of 1.1 ng of NG. For HPLC-TEA, 7 μ L was injected, with the NG peak being 15 ng.

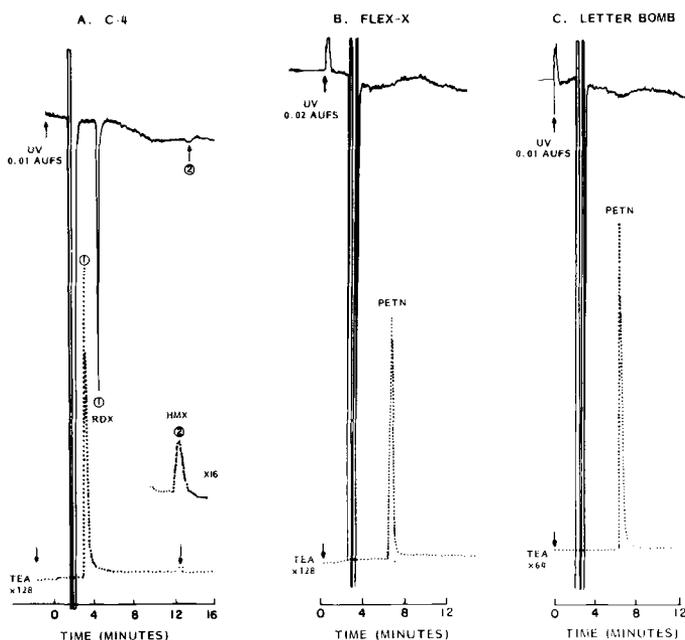


FIG. 4—(a) Chromatogram on HPLC-UV-TEA for 10- μ L injection of a 10-ppm extract of C-4. Peak 1, at the retention time of RDX, was 117 ng. Peak 2, of HMX, corresponds to 4.2 ng. (b) Chromatogram on HPLC-UV-TEA for 3- μ L injection of a 10-ppm extract of Flex-X. The single peak was 38 ng of PETN. (c) Chromatogram on HPLC-UV-TEA for 5- μ L injection of a 10-ppm extract of a letter bomb. The single peak was 33 ng of PETN.

met [24,25]. The most likely explanation of the apparent NG in the debris is a result of cross-contamination from the demolition range, which has been in continuous use for over 40 years. Unfortunately, a control sample from the range was not made available at the time the experiment was conducted.

The data in Figs. 5 and 6 demonstrate the capability of identifying postblast explosive debris at the picogram level, even when the only sample preparation was extraction of the organic residue into a suitable solvent. Again, cleanup was unnecessary since there were few interfering peaks.

Postblast Air Sample

A Thermo sorb/*N* (Thermo Electron) *N*-nitrosamine air sampling cartridge [29] was evaluated for its capability of trapping postblast air samples. Following the detonation of a commercial dynamite bomb at the U.S. Marine Corps demolition range in April 1982, 10 L of air were drawn through a cartridge with a bicycle pump. The cartridge was capped and sent to Thermo Electron for analysis.

The Thermo sorb/*N* cartridge was analyzed in the conventional manner by eluting with 1.8 mL of methylene chloride/methanol (75/25) into a sample vial. The solution was then analyzed by HPLC-UV-TEA on a uBondapak CN column, using a solvent system of isooctane/methylene chloride/methanol (75/20/5) at a flow rate of 1.5 mL/min. The TEA chromatogram, shown in Fig. 7, shows the presence of EGDN, which is characteristic of a dynamite blast. NG, another component of dynamite that has lower vapor pressure than that of EGDN, was not detected under these conditions. The minimum detectable level of EGDN is

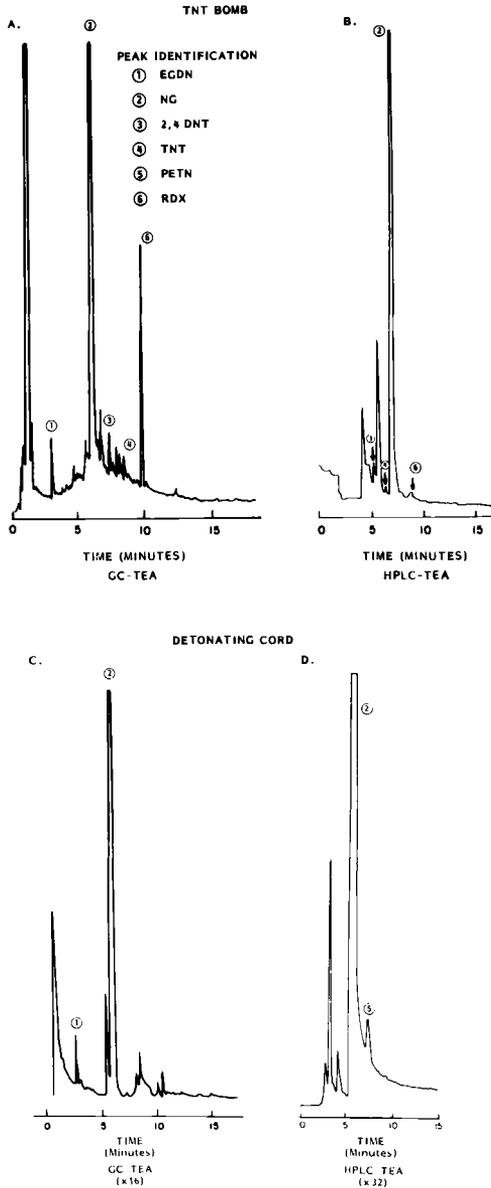


FIG. 5—(a and b) Chromatograms on GC-TEA and HPLC-TEA of extracts from postblast debris following an explosion of a TNT bomb. On GC-TEA ($\times 16$), a $0.5\text{-}\mu\text{L}$ injection of debris extract gave 4.4 ng of NG, 0.15 ng of 2,4-DNT, 0.03 ng of TNT, and 0.5 ng of RDX. For HPLC-TEA ($\times 32$) a $20\text{-}\mu\text{L}$ injection was used. (c and d) Chromatograms on GC-TEA and HPLC-TEA of an extract from post-explosion debris of a detonating cord bomb. On GC-TEA a $0.2\text{-}\mu\text{L}$ injection of debris extract showed NG, and a trace of EGDN. On HPLC-TEA, a $5\text{-}\mu\text{L}$ injection indicated NG, and a trace of PETN.

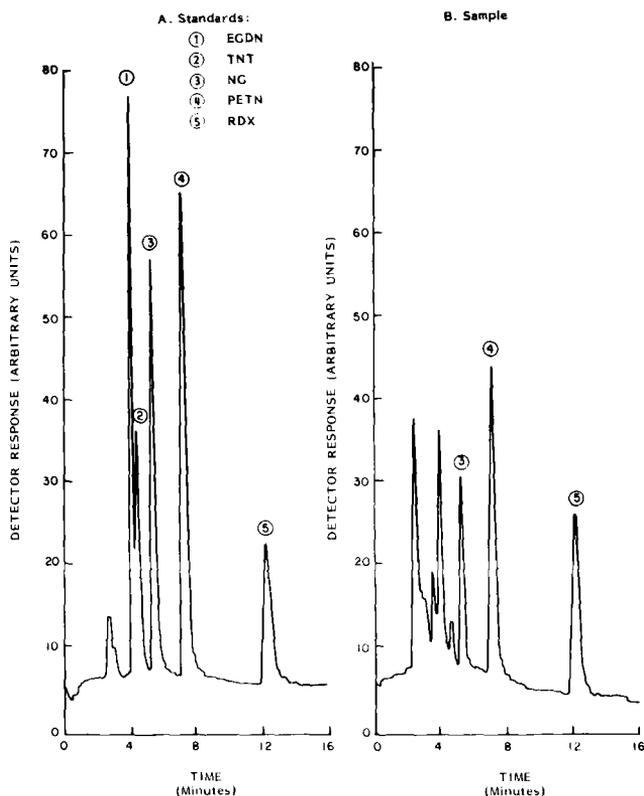


FIG. 6—Chromatograms on HPLC-TEA ($\times 16$) of five explosive standards (a) and a 20- μL injection of the extract from post-explosion debris of a C-4 bomb (b). The peak identification is 1-EGDN, 2-TNT, 3-NG, 4-PETN, and 5-RDX. The 20- μL injection of debris extract contained 5 ng of NG, 6.8 ng of PETN, and 9 ng of RDX.

less than the 9 ng shown in Fig. 7. The minimum detectable level could be further enhanced by taking a larger air sample.

Similar experiments with an RDX and a TNT bomb were unsuccessful. Further work is needed to determine whether the cartridge would have trapped and released these compounds if they had been present in postblast air.

Handswab Experiments

The feasibility of detecting trace levels of explosive residue on the hands was evaluated. The following explosives were used for the test: C-4, gel dynamite, a plastic explosive, and a piece of a letter bomb. Four volunteers held a small piece of explosive (approximately 1 by 3 cm) in the hand for 1 min. After 15 min, the palm area was washed twice with a cotton swab soaked in acetone [5-7]. The swabs were squeezed to dryness, and the acetone washings were concentrated to 2 mL under a stream of N_2 gas. Aliquots were then analyzed by GC-TEA and HPLC-TEA.

Figure 8a and b shows the chromatograms for the gel dynamite handswab. Both EGDN and NG are seen to be present in relatively large quantities (the TEA analyzer was on attenuation $\times 128$ for GC and $\times 256$ for HPLC). For the handswab from C-4, only a single chromatographic peak of RDX was observed (see Fig. 8c and d). For plastic explosive, see Fig. 8e

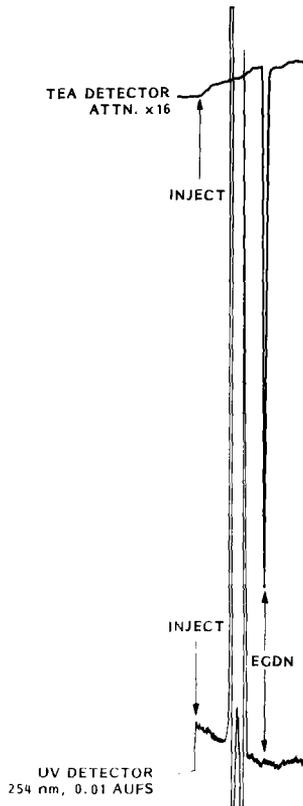


FIG. 7—Chromatograms on HPLC-UV-TEA of an air sample which had been collected on a Thermo-sorb/N cartridge, following a blast from a dynamite bomb. The 20- μ L injection indicated the presence of 9 ng of EGDN.

and *f*, the handswab extract contained mainly PETN, with only a trace of NG. The GC-TEA chromatogram shows the typical peak shape of PETN, which undergoes partial decomposition in the GC. Similarly, for the letter bomb, PETN was the only explosive that was detected on the hands.

Controlled experiments with handswabs that had been spiked with known amounts of explosives, indicated a lower detection limit of about 10 pg injected on column. Again, because of the selectivity of the detector, cleanup was not required.

Human Blood

Procedures for the analysis of the nitro-based vasodilators, at the level of 100 to 200 pg per mL of blood, are now routine [12, 13, 27]. In the method of Maddock et al [13], for example, this sensitivity was achieved on HPLC-TEA from 3.0 mL of plasma, following extraction with a 1:1 mixture of methylene chloride and ethyl acetate. The procedure was used for pharmacokinetic studies with adults using sublingual tablets, sustained-release tablets, capsule formulations, and creams of ISDN.

Because of possible occupational hazards [30], there is a need to detect military explosives and their metabolites in human blood. This capability was explored with RDX. Five millilitres of plasma was fortified with RDX at the 5-ppb level, extracted with 16 mL of methylene chloride and pentane (1:1), clarified by filtration through a Sep-Pak C-18 cartridge (Waters

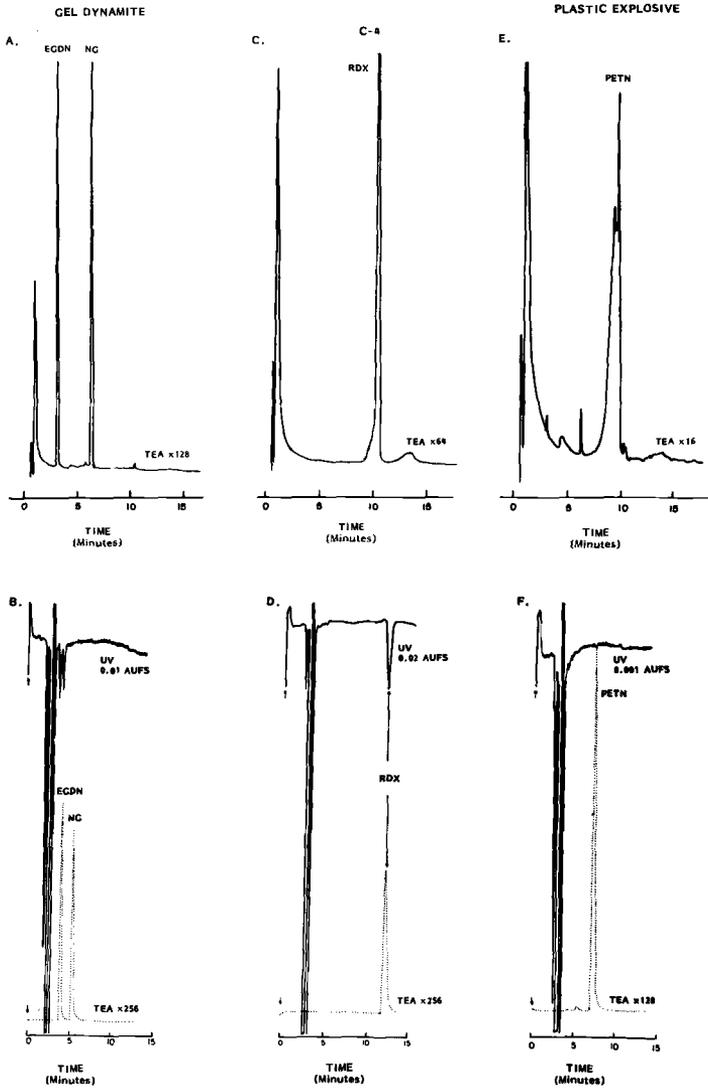


FIG. 8—Chromatograms on GC-TEA (a) and HPLC-UV-TEA (b) of a gel dynamite handswab extract. The injection volume was 0.2 μL on GC-TEA ($\times 128$) and 1 μL on HPLC-TEA ($\times 256$). Peak 1 coeluted with EGDN and Peak 2 with NG. Chromatograms on GC-TEA (c) and HPLC-UV-TEA (d) are of a C-4 handswab extract. The injection volume was 0.3 μL on GC-TEA ($\times 64$) and 20 μL on HPLC-TEA ($\times 265$). Only a single major peak, of RDX, was observed on both GC and HPLC. Chromatograms on GC-TEA (e) and HPLC-UV-TEA (f) are of a handswab from a person who had handled a plastic explosive. On GC-TEA 0.4 μL of the 2-mL extract was injected. A trace of NG (0.21 ng) and PETN was shown to be present (note the typical peak shape of PETN which decomposes on the column). For HPLC-TEA ($\times 128$), 3 μL was injected, giving a PETN peak of 84 ng.

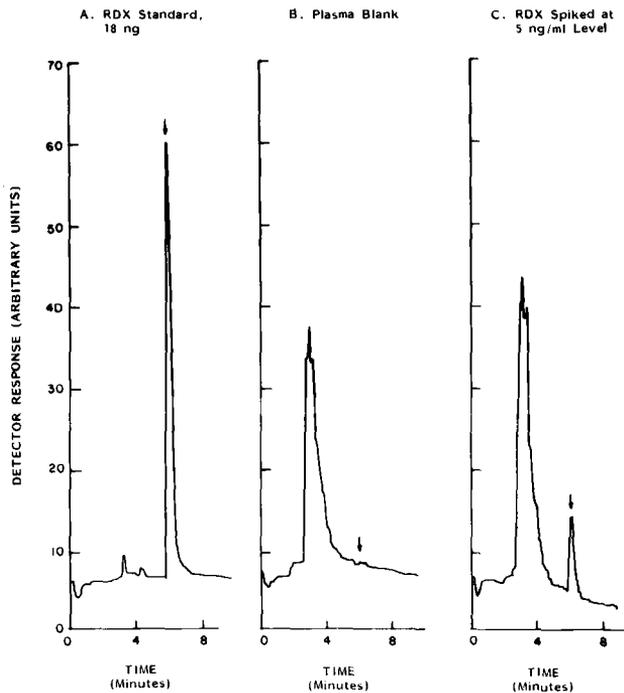


FIG. 9.—HPLC-TEA chromatograms of an RDX standard (18 ng), a human plasma blank extract, and a plasma sample fortified with RDX at the 5-ppb level. Injection volumes were 20 μ L.

Associates) and a Millex-SR filter (Millipore), concentrated under N_2 to 0.2 mL, and analyzed by injecting 25 μ L onto HPLC-TEA (see Fig. 9). The peak of RDX is clearly visible. The minimum detectable amount is estimated to be about 100 pg/mL of plasma.

Conclusion

The capability for routine detection of explosives at the low picogram level from "real world" samples of military explosives, post-explosion debris, handswabs, and human blood has been demonstrated. Because of the selectivity of the TEA analyzer, cleanup is not needed. The analytical methods are, therefore, simple and rapid. The minimum detectable amount for most explosives is 4 to 5 pg injected on column.

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