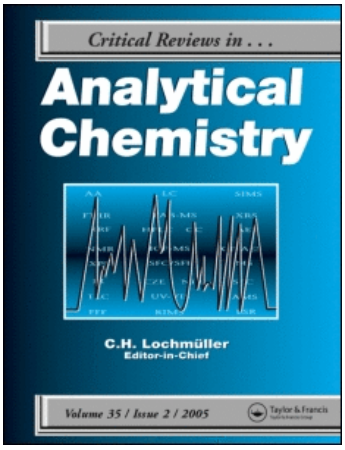

Critical Reviews in Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713400837

High-Performance Liquid Chromatographic Methods for the Analysis of Explosives

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To cite this Article Gaurav, Dhingra , Malik, Ashok Kumar and Rai, P. K.(2007) 'High-Performance Liquid Chromatographic Methods for the Analysis of Explosives', Critical Reviews in Analytical Chemistry, 37: 4, 227 — 268
To link to this Article: DOI: 10.1080/10408340701244698
URL: http://dx.doi.org/10.1080/10408340701244698

Critical Reviews in Analytical Chemistry, 37:227–268, 2007

ISSN: 1040-8347 print / 1547-6510 online
DOI: 10.1080/10408340701244698

High-Performance Liquid Chromatographic Methods for the Analysis of Explosives

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This review gives an overview of the developments in the field of analysis of explosives by HPLC for forensic applications. The review covers almost all aspects of analysis such as the analyte's category, matrix involved, technique and conditions used for preconcentration, column and mobile phase used, and subsequent detection in tabular form. This paper covers only organic explosives, e.g., nitroaromatic, nitramine, nitrate esters, peroxide-based explosives and their metabolites and their analysis in various environmental and biological samples like air, water, soil and blood serum.

Keywords HPLC, explosives, SPME, SPE, analysis, review

INTRODUCTION

Explosives are the chemical compounds (Table 1) or mixtures that will, on application of an external stimulus such as heat, shock, friction or ignition, undergo rapid chemical decomposition. The chemical reaction results in sudden release of large amount of energy due to liberation of gas and temperature. The pressure thus released is thrust out equally in all directions.

Chemical explosives are the most commonly used, although there are also mechanical and nuclear explosives. A mechanical explosive is one in which a physical reaction is produced, like that caused by overloading a container with compressed air. Nuclear explosives, which produce a sustained nuclear reaction, are by far the most powerful, but their use has been restricted to military weapons, although studies and experiments have been conducted with regard to their controlled use in certain industrial operations.

The principal chemical explosives include black powder, nitroglycerine, dynamite and trinitrotoluene (TNT). A chemical explosive can be gaseous, liquid or solid, although the latter two are generally capable of producing more powerful explosions. There are two basic types of chemical explosives: detonating or high, explosives; and deflagrating or low, explosives. Detonating explosives, such as dynamite, decompose rapidly and create high pressure, while deflagrating explosives, although they may burn quickly, produce considerably lower pressures. Detonating explosives are also divided into primary and secondary detonators. Primary explosives are detonated by ignition for example, a flame or heat producing impact whereas secondary explosives require a separate detonator.

Nitroglycerin and dynamite succeeded black powder as the chief explosives. An Italian chemist, Ascanio Sobrero, discovered nitroglycerin in 1846. The Swedish scientist, Alfred Nobel invented dynamite in 1867, the original explosive being a mixture of 75% nitroglycerin and 25% ghur (a porous, absorbent material that made the product easier to control and safer to use). Nobel developed gelatinous dynamite in 1875 by creating a jelly from the dissolution of a colloid-type nitrocotton in nitroglycerin, producing a more powerful explosive than the straight dynamites and one that proved to be safer. Later, ammonium nitrate was used in dynamite, which made it even more safer to use and less expensive to produce.

Explosives are broadly classified into two classes: low and high explosives.

i. A low explosive is combustible substance that decomposes rapidly (deflagration) but does not explode under normal condition but get detonated through combined use with high explosives. Low explosives are normally employed as propellants. They undergo deflagration at rates that vary from a few cm/sec to approximately 400 meters/second. Included in this group are pyrotechnics and smokeless powder.
ii. High explosives are normally employed in mining, demolition and military warheads. They undergo detonation at rates of 1,000 to 9,000 meters/second. High explosives are conventionally subdivided into two classes differentiated by sensitivity: (a) Primary Explosive: They are extremely sensitive to shock, friction and heat. They will burn rapidly or

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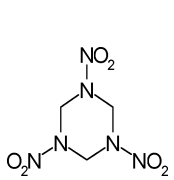
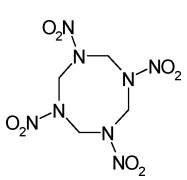
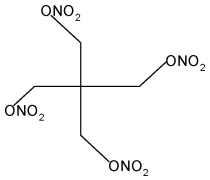
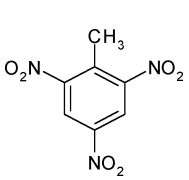
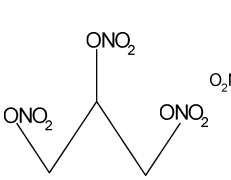
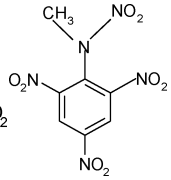
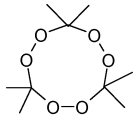
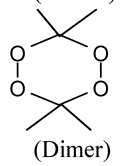
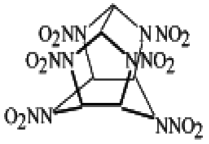
TABLE 1
Some Characteristics of Commonly Used Explosives

	RDX	HMX	PETN	TNT	Nitro Glycerine	TETRYL	Acetone Peroxide	CL-20
Common Name	Cyclonite, hexogen	Octogen	Penthrite, nitropenta	Trotyl	Glyceryl Trinitrate	Tetralite	TATP, TCAP	Hexanitroiso wurtzitane
IUPAC name	1,3,5-trinitro-perhydro-1,3,5-triazine	1,3,5,7-tetranitro perhydro-1,3,5,7-tetra-azocine	2,2-bis(nitroxy methyl)-l,3-propane diol-l,3-dinitrate	2,4,6-trinitro toluene	1,3-dinitro oxypropan-2-ylnitrate	2,4,6-tri nitrophenyl-N-methyl nitramine	3,3,6,6-tetramethyl-1,2,4,5-tetraoxane (Dimer) 3,3,6,6, 9,9-hexamethyl-1,2,4,5,7,8-hexaoxacyclononane (Trimer) (Trimer)	Octahydro-1,3,4,7,8,10-hexanitro-5,2,6-(iminomethenimino)-1H-imidazo[4,5-b] pyrazin
Chemical structure							 (Trimer)  (Dimer)	
Chemical formula	$C_3H_6N_6O_6$	$C_4H_8N_8O_8$	$C_5H_8N_4O_{12}$	$C_7H_5N_3O_6$	$C_3H_5N_3O_9$	$C_7H_5N_5O_8$	$C_6H_{12}O_4$ (Dimer) $C_9H_{18}O_6$ (Trimer)	$C_6H_6N_{12}O_{12}$
CAS No.	121-82-4	2691-41-0	78-11-5	118-96-7	55-63-0	479-45-8	17088-37-8	14913-74-7
Molecular mass (g/mol)	222.117	296.20	317.15	227.131	227.08	287.15	148.157 (Dimer) 222.24 (Trimer)	438.19
Shock sensitivity	Low	Low	Very high	Insensitive	Very high	Insensitive	High	—
Friction sensitivity	Low	Low	Very high	Insensitive	Very high	Insensitive	High	—

Density (g/cm^3)	1.82	1.91	1.773	1.654	1.13	1.73	1.18	2.04
Explosive velocity (m/s)	8,750	9100	8400	6900	7700	7570	5300	10300
RE Factor	1.60	1.70	1.66	1.00	1.50	1.25	—	—
Melting point (°C)	205.5	276 to 286	141.3	81	13.2	129.5	91	273
Ignition temp. (°C)	229	335	145	300	—	—	—	—
Decomposition temp. (°C)	170	280	190	295	50 to 60	—	—	—
Appearance	Colorless solid crystals	Colorless solid crystals	Odorless white crystalline Solid	Pale yellow Crystals	Clear yellow/ Colorless oily liquid	Odorless yellow Crystalline Solid	White crystalline	Odorless monoclinic crystals
Oxygen balance	−21.6%	−26.1%	−10.1%	−73.9%	13.5%	−47.4%	—	—
Nitrogen content	37.84%	37.83%	17.72%	18.5%	18.5%	24.39%	—	—
Volume of detonating gas (l/Kg)	900	927	823	730	780	800	—	—
Heat of combustion (Kcal/Kg	2285	2255	—	3590	—	—	—	—
Heat of explosion/ detonation (Kcal/Kg)	—	1355	1421	1011	—	—	—	—
Vapor pressure	4.6×10^{-9} Torr at 25°C	—	8.38×10^{-4} Torr at 97°C	5.8×10^{-6} Torr at 25°C	2.3×10^{-4} Torr at 20°C	—	—	—
Solubility	Soluble in acetone and insoluble in water and sparingly soluble in alcohol, ether and benzene	Insoluble in water	Soluble in acetone and methyl acetate, sparingly soluble inalcohol, ether & benzene insoluble in water	Soluble in benzene and acetone sparingly soluble in alcohol, insoluble in water	Insoluble in water but soluble in other organic solvent	Slightly soluble in water and other solvents	—	—

detonated, if ignited, and (b) Secondary Explosive: These are also called base explosives and relatively insensitive to shock, friction and heat. They burn when ignited in small unconfined quantities, but detonation can occur. Dynamite, RDX, HMX, PETN, TNT and others are secondary explosives. PETN is often considered as benchmark explosive with materials that are more sensitive than PETN are being classified as primary explosives.

Sometimes definition add a third category, i.e., tertiary explosives or blasting agents, which are so insensitive to shock that they cannot be reliably detonated by practical quantities of primary explosives and instead require an intermediate explosive booster of secondary explosives. Examples included ammonium nitrate/fuel oil mixture (AFNO) and slurry of "wet bag" explosives. These are primarily used in large-scale mining and construction operations.

Secondary explosives are mostly organic compounds and can be further classified according to their functional groups, e.g., nitro aromatic, nitramine, and nitrate esters. Thus on the basis of wide applications of explosives, it seems to be necessary to summarize recent chromatographic procedures. So, we opted for the analysis of important organic high explosives and their metabolites in various complex matrices by HPLC technique. The aim of this compilation is to give an overview and evaluation on complete HPLC analysis of organic high explosives (including extraction/isolation procedure, separation and quantification by HPLC hyphenated with various types of detector), focusing to proposals published till now.

HPLC Analysis of Explosives

In the last 20 years the development of analytical methods that are capable of detecting ultra-low trace quantities of explosives has become increasingly important in the field of forensic science. Routine analyses rely on the detection of nanogram quantities to confirm the link between a suspect and the manufacture or use of explosives. Most of these reviews (1–8) do not provide up-to-date information on HPLC for the analysis of the explosives. So there is a need for a comprehensive review for their analysis. Since HPLC has been used extensively for the analysis of the explosives and there is no comprehensive review is available to provide up-to-date information. Therefore, we have considered to summarize all methods of extractions, analysis by HPLC and subsequent detection for high explosives.

HPLC-UV Methods

UV absorbance detection is one of the most popular universal detection methods used in microseparations due to its simplicity, ruggedness, ease-of-use and low cost. The majority of organic compounds can be analyzed by UV detectors and most HPLC analyses are performed using UV detectors. So the hyphenation of UV system with HPLC is of very high importance.

Nefso et al. (11) analyzed the abiotic degradation of dissolved TNT in the presence of ferrous iron (Fe^{2+}) and six

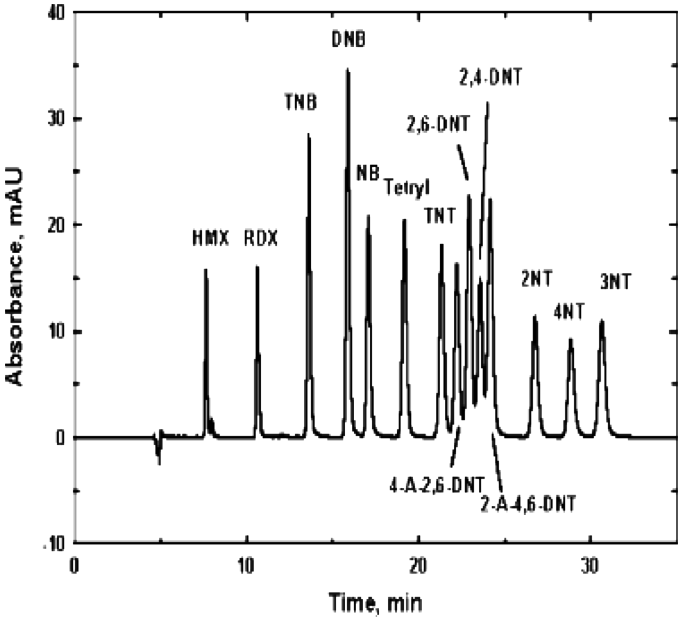


FIG. 1. Complete resolution of a mixture of 14 explosives standards by combining two reverse phase C18 columns in series with UV detection. (With permission from (15).)

different minerals. MacCrehan et al. (12) analyzed the additives in smokeless powder an integral part of improvised explosives devices (IEDs) and in the evaluation of organic gunshot residues (OGSR). The RM, a smokeless rifle powder, was chosen after evaluation of three candidate powders. The additives were determined using solvent extraction with ultrasonic agitation followed by liquid chromatography (LC). RM 8107 provides reference values obtained by LC analysis for the key additives nitroglycerin, diphenylamine (and its nitration product, *N*-nitrosodiphenylamine), and ethylcentralite. Hewitt et al. (14) examined the TNT and RDX residue from soil samples at live fire and blow-in-place detonations sites. Snow was used as a collection medium to examine RDX and TNT residues. Marple et al. (15) determined the nitroaromatic and nitramine explosives from environmental samples including groundwater and soil with UV detection. The chromatogram of various explosives studied is presented in Figure 1.

In another approach by Marple et al. (16), HPLC-PAED was used in conjunction with ultraviolet absorbance (UV) detection for determining explosives (Figure 2) in environmental samples. The system utilizes an on-line solid-phase extraction technique for sample pretreatment thus reducing the required ground water sample size from 1 litre to 2 ml and minimizing sample handling.

Monteil-Rivera et al. (23) determined RDX, HMX, TNT, DNB and DNTs from water samples at trace levels. In this work solid-phase microextraction (SPME) technique for the recovery of nine explosives from aqueous samples using HPLC with ultraviolet detection (HPLC-UV) is reported. Nipper et al. (24) studied the role of microbial activities and UV exposure

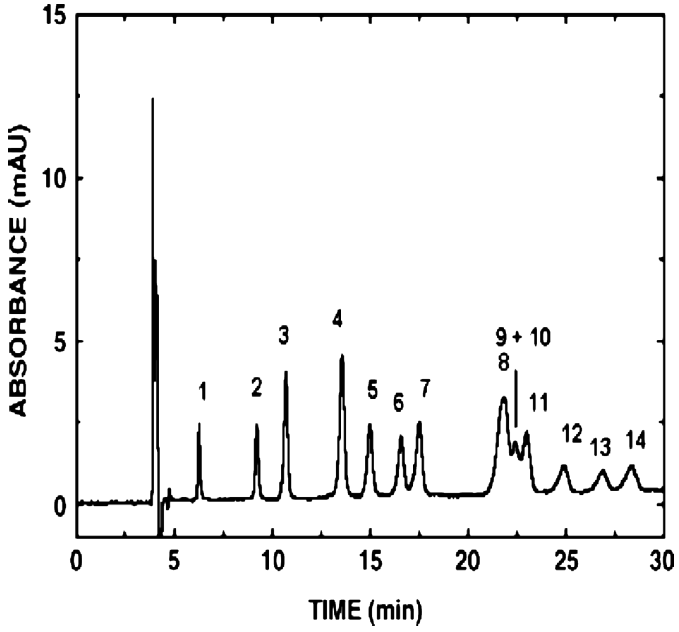


FIG. 2. Optimized separation of explosives with UV detection at 254 nm; Mobile phase: 50% methanol in 20 mM acetate buffer, pH = 4.5, flow rate: 1.0 mL/min; guard column: Phenomenex Security Guard with 4 mm × 3.0 mm C8 cartridge; Column: C18, 5 μm, 4.6 mm × 250 mm; column oven temperature: 30°C. Peak 1 = HMX; 2 = RDX; 3 = 1,3,5-TNB; 4 = 1,3-DNB; 5 = NB; 6 = Tetryl; 7 = TNT; 8 = 4-A-2, 6-DNT; 9 = 2,6-DNT; 10 = 2-A-4, 6-DNT; 11 = 2,4-DNT; 12 = 2-NT; 13 = 4-NT; 14 = 3-NT. (With permission from (16).)

in biodegradation of DNT and picric acid in marine sediments and water with HPLC and other analytical techniques. Bio- and photo-transformation of two munitions and explosives of concern, 2,6-dinitrotoluene (2,6-DNT) and 2,4,6-trinitrophenol (picric acid) were assessed in spiked marine sediments and water. Sterilized sediments were used as controls for biotic vs. abiotic transformation. Transformation products were analyzed by HPLC and other analytical techniques.

Szecsody et al. (25) analyzed the sorption and oxic degradation of the explosive CL-20 and its degradation products during transport in subsurface sediments. RDX and CL-20 is detected with UV lamp while CL-20 degradation products are detected by MS. Dutta et al. (29) used HPLC to test the ability of *S. meliloti* to degrade 2,4-DNT. The possible presence of 2,4-DNT remaining in the treated soil was tested and no 2,4-DNT had been absorbed by the soil. Ozhan et al. (30) developed a simple and sensitive HPLC method for the assay of cyclonite (RDX) in human plasma. The method was applied to evaluate RDX concentration in plasma samples obtained from soldiers exposed to RDX. Schutle-Ladbeck et al. (35) analyzed the air samples for the determination of triacetonetriperoxide (TATP). The high volatility of the peroxide leads to significant concentrations in the air surrounding even minute quantities of TATP, thus enabling the analyst to avoid direct contact with the sensitive explosive. Air sampling is performed using gas-washing bottles filled with acetonitrile and air sampling pumps. After sampling, two different analytical methods were used as first, reversed-phase HPLC with subsequent post-column UV irradiation and electrochemical detection and second, photochemical degradation of TATP with enzyme-catalyzed photometric detection. Smith et al. (36) used a simple, semi-automated, micro column solid-phase extraction (SPE) system for the extraction, pre-concentration and HPLC analysis of seven different explosives and explosive derivatives contaminating seawater, river water and well water samples.

The first method for quantitative trace analysis of peroxide-based explosives was described by Schulte-Ladbeck et al. (38). A reversed-phase HPLC method with post-column UV irradiation and fluorescence detection for the analysis of triacetone triperoxide (TATP) and hexamethylene triperoxide diamine (HMTD) has been developed. After separation, the analytes are degraded photochemically to hydrogen peroxide, which is subsequently determined on the basis of the peroxidase-catalyzed oxidation of *p*-hydroxyphenylacetic acid to the fluorescent dimer. Adrian et al. (41) studied the anaerobic biodegradation of high explosives by the addition of hydrogen and electron donor that produces hydrogen. Jenkins et al. (43) used snow-covered ranges to estimate the amount of explosives residues that resulted from detonation of individual mortar rounds and a small antipersonnel land mine. Mortars were fired and land mines were detonated by EOD (explosives ordnance disposal) personnel after attaching C4 (RDX) and/or a blasting cap. The locations where residues were deposited were identified by the presence of soot from the detonation of TNT on the surface of the otherwise clean snow. Large surface snow samples were collected with a snow shovel and the melted snow was extracted and analyzed by reversed-phase HPLC.

Halasz et al. (48) analyzed the polynitro organic explosives and their degradation products in soil environment. Batlle et al. (49) developed an analytical method for determining nitroaromatic explosives in vapor phases. Samples were collected by pumping air through glass fiber filters and polyurethane foam adsorbents, and an on-line extraction system combining SFE and HPLC was developed. This allows analytes to be transferred from the adsorbent to the HPLC system via a porous graphitic carbon trap. Most of the nitroaromatic isomers were separated by gradient elution with a suitable mobile phase. The proposed method is fully automated and it is compatible with most of the organic solvents commonly used as SFE modifiers or additives. The method was applied for assessing whether areas are mined in landmine-clearing operations. Reifenrath et al. (52) analyzed the radiolabel extract from the dermis and receptor fluid. The percutaneous absorption potentials of ^{14}C-labeled TNT, trinitrobenzene, 2,4-DNT, 2,6-DNT, 2-amino-4,6-dinitrotoluene, 4-amino-2,6-dinitrotoluene, 2,4-diamino-6-nitrotoluene, 2,6-diamino-4-nitrotoluene, *N*-methyl-*N*-2,4,6-tetranitrobenzamine, RDX, HMX and 2,2-thiobis(ethanol) were

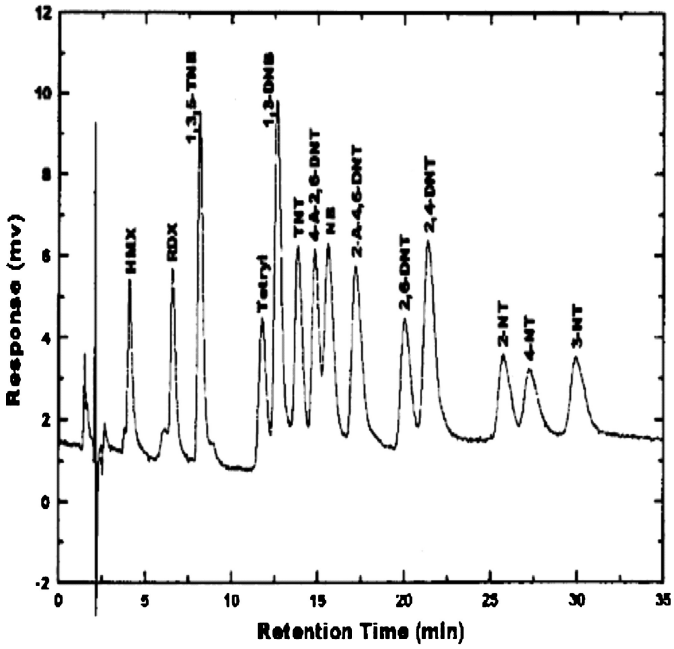


FIG. 3. Chromatogram of SPME/HPLC-UV of EPA 8330 mixture standard (20 ng/mL in each 25% NaCl aqueous solution and acetonitrile: water ratio of 1:199). (With permission from (67).)

determined from two soil types. Walsh et al. (54) studied the effect of particle size reduction by grinding on sub-sampling variance for explosives residues in soil. Goodpaster et al. (56) separated nitramine and nitroaromatic explosives by capillary liquid chromatography and subsequent UV detection. This method was then applied to the determination of RDX, HMX and 2,4,6-TNT in commercial-grade and military-grade explosive samples.

Onuska et al. (58) optimized the accelerated solvent extraction for the analysis of munitions residue in sediment samples followed by UV detection. Rodgers et al. (59) analyzed the changes in concentration of 2,4,6-trinitrotoluene and 2,4- and 2,6-dinitrotoluenes and some of their electrolysis products during electrochemical reduction in aqueous solution. According to Chrompack application Note (60) water containing explosive residues were preconditioned on SPE and were analyzed by HPLC on C18 column.Varian application note (61) includes analysis of explosives from water using a styrene-divinyl benzene cartridge**.** Chromopack application note (62) includes detection of explosives from surface water using a Bond Elut SDB cartridge. The Macherey-Nagel application Note (65) includes detection of nitro explosive compounds separated on Nucleosil C18 column. Furton et al. (67) incorporated SPME for extraction of explosives from aqueous samples and real post-explosion soil debris samples followed by separation with HPLC and subsequent detection with UV. A modified SPME-HPLC interface using dual six-port valves allowed for independent optimization of SPME desorption and injection variables provides improved resolution and sensitivity (Figure 3).

Harkins et al. (70) analyzed soil and groundwater contaminated with differing combinations of high explosives including RDX, HMX, and TNT. Ellwanger et al. (72) analyzed nitroaromatic explosives and their decomposition products on fluorenyl stationary phases. Good selectivity was obtained on a stationary phase with a hexyl spacer and a high ligand density, with only the two heterocyclic compounds co-eluting. Chrompack application Note (73) includes the analysis of nitro explosives from soil extracts on a Zorbax C18 column. Lang et al. (74) studied the complete separation of nitroaromatics and nitramines by HPLC using the two phase approach for the improvement of EPA method 8330.

Xu et al. (76) studied the percentage purity of hexanitrohexaazaisowurtzitane by reversed-phase high-performance liquid chromatography. Hilmi et al. (77) analyzed explosives including TNT, HMX and RDX in soil and ground water by liquid chromatography-UV and amperometric detection. Alnaizy et al. (78) studied the total organic carbon to monitor the oxidation treatment of waste water contaminated with explosives. Larson et al. (79) analyze the explosives using HPLC and GPC in plant tissues for RDX, trinitrotoluene and their metabolites using the U.S. EPA Method 8330 modified for analysis of plant tissues. Wu et al. (80) analyzed the EPA method for explosives with SPME-HPLC technique. The improved SPME/HPLC interface was used in the technique. The new interface gave an increase in peak areas and smaller RSD than the conventional interface. Reproducibility was also excellent. Linearity studies were performed by extracting spiked explosives samples. Jenkins et al. (82) analyzed the TNT from soil extracts using U.S. EPA method for nitroaromatics and nitramine explosives. Drzyzga et al. (84) studied the anaerobic incorporation of explosive TNT and metabolite into the organic soil mixture of contaminated soil after different treatment procedures through HPLC analysis. Dutta et al. (85) reported the peroxide independent degradation of TNT by non-ligninolytic P. chrysosporium. Significant disappearance of TNT from highly contaminated soil using P. chrysosporium has been observed through analysis by HPLC.

Walsh et al. (89) employed an analytical method for nitroaromatic, nitramine, and nitrate ester explosives and co-contaminants in water through SPE. Concentration estimates for well water extracts from military sites were analyzed by HPLC. Koehne et al. (90) incorporate the two-dimensional HPLC for the separation of complex mixtures of nitrobenzene and nitrotoluene explosives and their by-products. Godejohann et al. (91) analyzed organic explosives compounds in mixtures by HPLC with UV detection. IST Application Note (92) includes the preconcentration and subsequent analysis of seven nitroaromatic explosives. Spiegel et al. (94) monitor the degradation process of explosives by HPLC and subsequent UV, amperometric detection. Brindle et al. (95) analyzed the separation of 16 nitro explosives with the help of *N*-Fluoren-2-yl-glutaric acid monoamide bonded to 3-aminopropylsilanized silica as a new stationary phase. Renner et al. (96) analyzed organic pollutants including

explosives in water at trace levels using fully automated solid-phase extraction coupled to HPLC from water based on SPE and HPLC. Jenkins et al. (97) analyzed the extracted samples from soil before colorimetric detection. Harvey et al. (98) analyzed the traces of high explosives, e.g., TNT and RDX from field crop. Preiss et al. (102) analyzed the high explosives, e.g., RDX, TNT by HPLC during comparison of high-field proton nuclear magnetic resonance spectroscopy for the analysis of explosives and related compounds in groundwater samples with HPLC method. Haag et al. (104) elaborated the application of coupling of SPME and HPLC to the analysis of explosives. Renner et al. (106) analyzed the explosive residue and decomposition products in aqueous media. Hawari et al. (107) analyzed the recovery of RDX from soil. Lewin et al. (108) employed high-performance liquid-chromatographic with UV-electrochemical detection for residues of explosives in water samples around a former ammunition plant. Baram et al. (109) analyzed the polynitro explosives in field samples. Caton et al. (110) determine the explosives and some metabolites of TNT in biological and environmental samples by liquid chromatography on a mixed-mode C_{18}-anion column. Shirey et al. (111) described the principles of SPME and the development of the cited interface. A chromatogram was presented that shows the separation of 14 explosives in water on Supelcosil LC-8 after sampling by use of this system.

Harvey et al. (112) analyzed the TNT and RDX from water samples using on-line trace enrichment using a divinylbenzene-vinylpyrrolidone co-polymer precolumn with a reversed-phase C_{18} HPLC analytical column. Bouvier et al. (113) analyzed and identfied the nitroaromatic and nitramine explosives in water using HPLC and UV/photodiode-array detection. Henderson et al. (114) analyzed the samples containing nitroaromatic and nitramine compounds in explosive mixture using US EPA method 8330. Lewin et al. (117) analyzed the contaminants from armament wastes. Zhou et al. (120) analyzed k′ value of nine explosives. The relationship between the composition of the mobile phase and the capacity factor (k′) in LC was studied based on the Grey Model. The experimental results were in good agreement with those predicted by the model calculations. Harvey et al. (122) analyze the explosive tetryl in bush bean plants. Baj et al. (123) employed high-performance liquid-chromatographic method for the determination of dynamites. Levsen et al. (125) analyzed nitroaromatics and nitramines in ammunition waste water and in aqueous samples from former ammunition plants and other military sites. Harvey et al. (126) analyzed tetryl and its transformation products in soil. Major et al. (127) analyzed the soil of open burning/open detonation (OB/OD) sites. Bauer et al. (128) analyzed the nitroaromatic explosives in soil. Jenkins et al. (129) analyzed the nitramine explosives in soil. Jenkins et al. (132) compare the four extraction techniques for munitions residues in soil samples. Turley et al. (133) analyzed the RDX in biological fluids using solid-phase extraction.

Yinon et al. (135) analyzed the metabolites of TNT in human and rat urine incorporating UV and mass spectrometry detection. Dahl et al. (136) determine black and smokeless powder residues in firearms and improvised explosive devices. Murphy et al. (137) used HPLC for analysis of nitroaromatic compounds on an N-propylaniline-bonded stationary phase. Den et al. (139) employed donor–acceptor complex chromatographic separation of explosives on 3-(10-methyl-9-anthryl)propylsilane stationary phase by HPLC. Yinon et al. (140) analyzed TNT and its metabolites in urine of munition workers by micro liquid chromatography-mass spectrometry incorporating sequential UV chemical ionization detection. Yinon et al. (142) analyzed TNT and its metabolites in blood of rabbits by HPLC and UV detection. Burrows et al. (145) analyzed RDX, HMX and their acetyl derivatives from water incorporating HPLC-UV detection. Yinon et al. (147) analyzed TNT and its metabolite in biological fluid with HPLC and subsequent UV detection. Bongiovanni et al. (149) analyzed trace amounts of six selected poly-nitro explosive compounds in soils. Prime et al. (151) analyzed the ethanediol mononitrate and monomethylamine nitrate from commercial blasting agents in post-blast samples. Yinon et al. (152) incorporated high-performance liquid chromatography-mass spectrometry for the analysis of explosives. Lyter et al. (153) investigated NG, TNT, HMX, RDX, PETN and Tetryl through hplc and subsequent UV detection. Brueggemann et al. (154) analyzed the nitramine including RDX, HMX, etc., explosives in waste water. Kayser et al. (162) analyzed the explosive materials for e.g., polynitro-compounds in explosives containing samples.

HPLC-PDAD Methods

Shin et al. (18) analyzed the anaerobic biotransformation of dinitrotoluene isomers by Lactococcus lactis subsp. lactis strain 27 isolated from earthworm intestine. Bausinger et al. (19) determined the mono-, di- and trinitronaphthalenes in soil samples contaminated by explosives. Paull et al. (22) presented the rapid screening of various high-grade explosives by HPLC with monolithic stationary phases. Two gradient methods were developed, the first for quantitative analysis of 11 explosives in under 14 min. The second method separated seven explosives in under 2 minutes and is suitable for rapid screening to determine the presence of specific and/or class of explosive. The rapid screening methods were successfully applied to soils spiked with known amounts of target explosives.

Monteil-Rivera et al. (26) measured the photophysical properties of polycyclic energetic nitramine explosive CL-20 and subsequently compare with RDX and HMX (Figure 4). Environmental fate, analytical and physicochemical data were made available. CL-20 found to be less persistent in the environment than RDX and HMX. Borch et al. (27) by incorporating reversed-phase HPLC - diode array detection analyzed the complete separation of 2,4,6-trinitrotoluene metabolites and EPA method 8330 explosives. Temperature was identified as the key parameter for optimal baseline separation. Increased temperature resulted in shorter retention times and better peak resolution especially for the aminoaromatics (Figure 5).

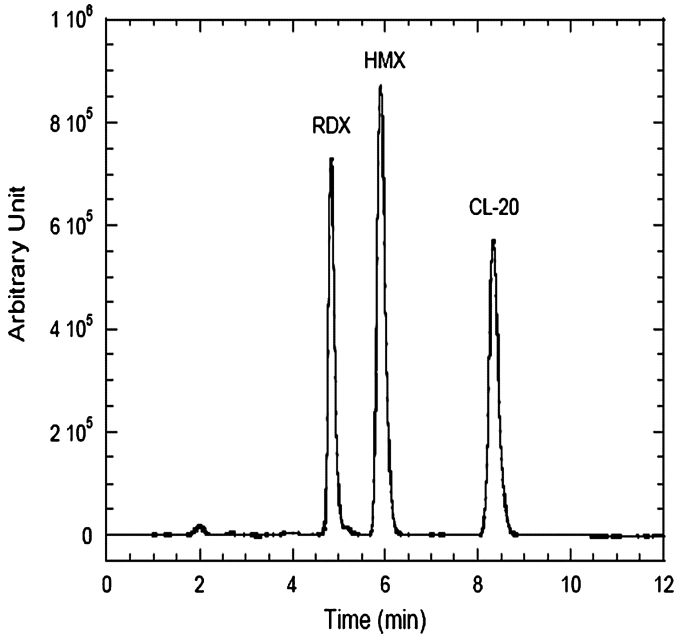


FIG. 4. A typical HPLC chromatogram showing separation of CL-20 from RDX and HMX; column: LC-CN; mobile Phase water–methanol (30:70); flow rate: 1.0 ml/min; detection DAD ($\lambda = 230$ nm). (With permission from (26).)

Robidoux et al. (28) analyzed RDX, HMX, TNT and its metabolites for the toxicity assessment of contaminated soil from an anti tank firing range. Smedts et al. (31) analyzed the separation of arsines and trinitrotoluene from explosives and arsine compound mixture with reversed phase HPLC. Campbell et al. (33) analyzed the nitroaromatic explosives with LC-MS from soil samples from OB/OD sites. The results obtained with this procedure agreed well with those obtained by an independent laboratory following the standard U.S. Environmental Protection Agency (EPA) method SW-846 8330. Compared with the EPA method, this method provides MS confirmation of the analytes. Groom et al. (34) analyzed the cyclic nitramine explosives viz. RDX, HMX and CL-20 from environmental samples with sulfobutyl ether- -cyclodextrin-assisted electrokinetic, chromatographic method. Didaoui et al. (39) utilized the computer-assisted optimization in the development of high performance liquid chromatographic methods for the analysis of some explosive and related compounds. Fuller et al. (42) analyzed the tetryl from soil sample through bioslurry treatment procedure. Radtke et al. (51) analyzed the particulate explosives at historical explosives testing area. A solvent-based sampling method was reported for determining TNT and its degradation products in contaminated soil. The reported method avoids the large variations in results which were observed with other sampling methods and attributed to the presence of particulate explosives in the soil.

The cyclic nitramine explosives RDX and HMX were examined in field and microcosm soil samples to determine their patterns of degradation and environmental fates by Groom et al. (57). A number of other analytical techniques, including SPME with on-fiber derivatization, GC-MS, GC-ECD, LC-MS and MEKC, were required for the analyses. Two different classes of intermediates were detected, both of which lead ultimately to the formation of N_2O and CO_2. The first class was identified as the nitroso derivatives formed by the sequential reduction of nitro functional groups. The second class of intermediates consisted of ring cleavage products including bis-(hydroxymethyl)nitramine and methylenedinitramine. Chrompack Application Note (60) includes the extraction, analysis and subsequently detection of various nitro and nitramine explosives from water samples. Robidoux et al. (63) analyzed the HMX from soil samples while determining the chronic toxicity of HMX in soil using the earthworm (Eisenia andrei) reproduction test. Rajagopal et al. (64) analyzed TNT, DNT and NB from aqueous solution while developing the adsorptive removal process for treatment of explosives contaminated wastewater using activated carbon. The adsorption characteristics of nitro-organics such as trinitrotoluene (TNT), dinitrotoluene (DNT) and nitrobenzene (NB) on granular activated carbon (GAC) were studied to understand their dynamic adsorption behavior for dilute aqueous solutions and a model was developed to predict the dynamics of the adsorption process and the effect of various design and operating parameters on adsorption characteristics. Thompson et al. (75) recovered and analyzed nitroexplosives from cotton swabs. Nitro-organic explosives were extracted from cotton swabs into H_2O, isolated and screened by LC with UV detection. The H_2O extraction/SPE process was effective in recovering organic explosives from cotton swabs giving greater selectivity with most samples. Bruns-nagel et al. (86) analyzed a compost mixture consisting of 2,4,6-trinitrotoluene (TNT)-contaminated soil, chopped sugar beet and straw anaerobically percolated with tap water. The anoxic conditions led to the transformation of 90% of the TNT to monoaminodinitrotoluenes and diaminomononitrotoluenes. An efficient TNT remediation system was presented that led to the identification of three conjugated TNT metabolites.

Godejohann et al. (93) analyzed the ground water near former ammunition plants for the detection of nitroaromatic high explosives. This method reported was able to identify more compounds derived from explosives. Godejohann et al. (101) analyze nitrophenols, nitrobenzoic acids and polar explosives by HPLC-diode-array detection in groundwater samples of former ammunition plants. Sample could be injected directly on to the column without any sample preparation for a fast determination. Gates et al. (105) analyze nitroaromatic explosives and their degradation products in unsaturated-zone water samples by high-performance liquid chromatography with photodiode-array detection. Bouvier et al. (113) analyzed and identified nitroaromatic and nitramine explosives in water using photodiode-array detection. Feltes et al. analyzed explosive (115) in water and soil incorporating DAD and nitro-aromatics

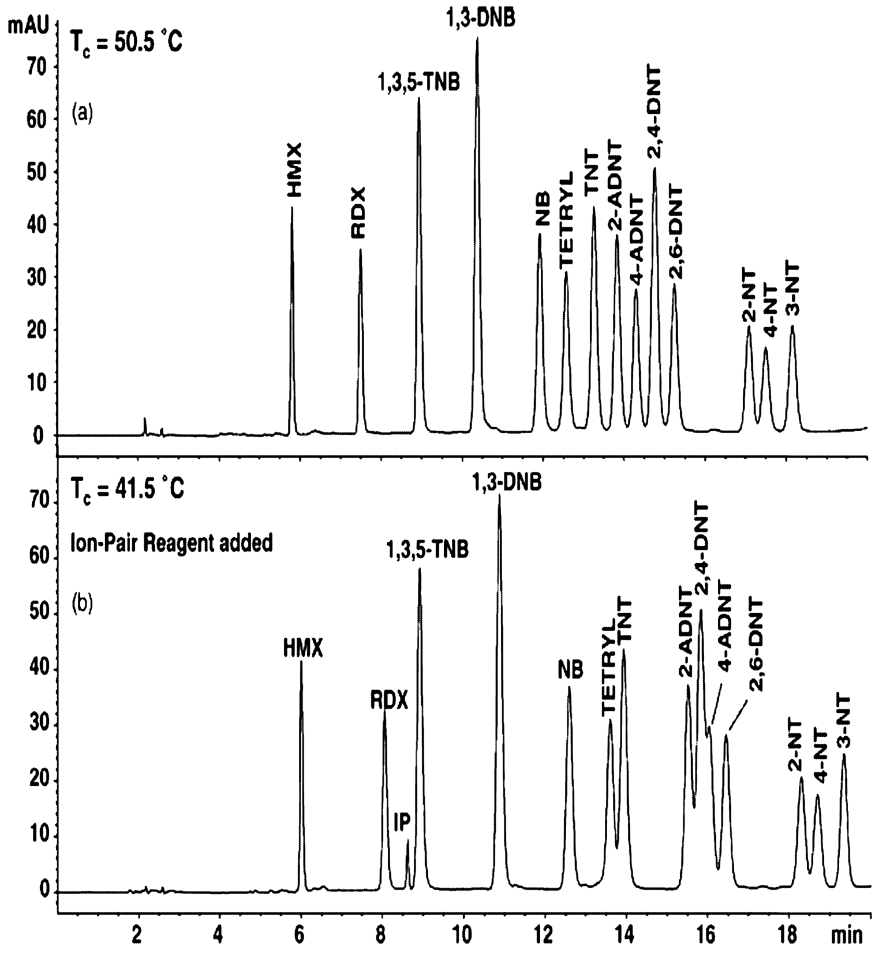


FIG. 5. (a) Baseline separation of EPA Method 8330 compounds at the optimized temperature of 50.5°C (no ion-pair reagent added). (b) Separation of the 14 EPA Method 8330 chemicals in the presence of an ion-pair reagent at the optimized temperature of 41.5°C. Note how the ion-pair reagent prevents baseline separation of several compounds. (With permission from (27).)

(130) from former ammunition plants in surface waters with reversed-phase high-performance liquid-chromatographic determination and photodiode-array detection. Bi et al. (131) qualitatively and quantitatively analyzed the nitroglycerine and centralite [NN′-diethyl-NN′-diphenylurea] in double-base powder with same method.

HPLC-MS Methods

HPLC mass spectrometry is an extremely versatile instrumental technique. As the name suggest the instrumentation comprises a high-performance liquid chromatograph (HPLC) attached, via a suitable interface, to a mass spectrometer (MS). The primary advantage HPLC/MS has over GC/MS is that it is capable of analyzing a much wider range of components. Compounds that are thermally labile, exhibit high polarity or have a high molecular mass may all be analyzed using HPLC/MS. Solutions derived from samples of interest are injected onto an HPLC column that comprises a narrow stainless steel tube (usually 150 mm length and 2 mm internal diameter, or smaller) packed with fine, chemically modified silica particles. Compounds are separated on the basis of their relative interaction with the chemical coating of these particles (stationary phase) and the solvent eluting through the column (mobile phase). Components eluting from the chromatographic column are then introduced to the mass spectrometer via a specialized interface. The two most common interfaces used for HPLC/MS are the electrospray ionization (ESI) and the atmospheric pressure chemical ionization interfaces (APCI). Therefore MS provides a powerful detection tool in combination with HPLC.

Pan et al. (9, 10) analyzed the RDX and its N-nitroso derivatives in soil and HMX in environmental samples like lizard egg extracts using electronspray ionization-mass spectrometric method. Holmgren et al. (13) developed a new LC–MS method for the determination and characterization of three groups of commonly used organic explosives i.e., nitroaromatic compounds, cyclic nitroamines and nitrate esters using a porous graphitic carbon (PGC) (Hypercarb) column. Twenty-one different explosive-related compounds including

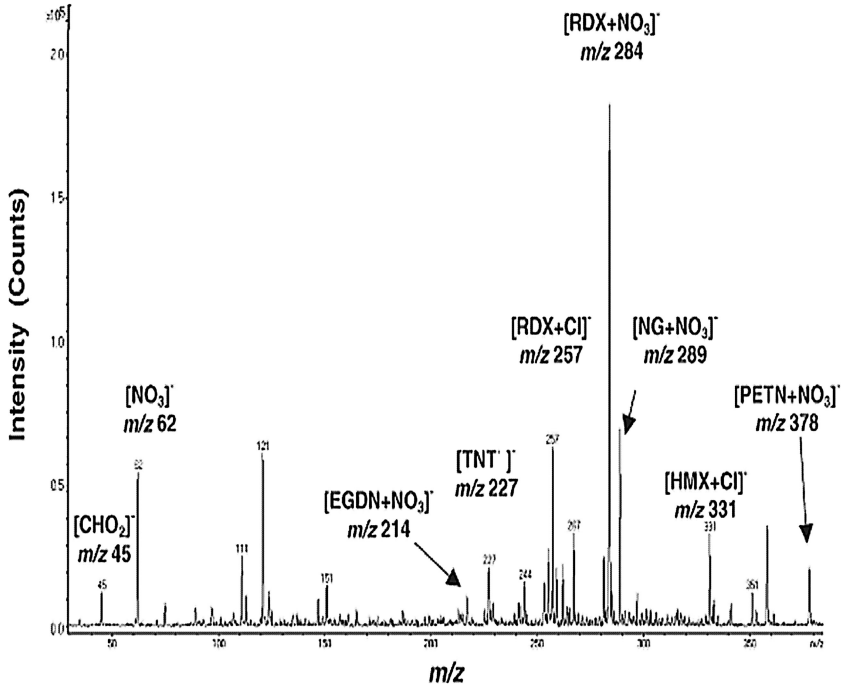


FIG. 6. Mass spectrum of a mixture of high explosives containing EGDN, NG, TNT, PETN, RDX and HMX in 50% MeOH/50% aqueous mixture with 0.3 mM ammonium chloride, ammonium formate and ammonium nitrate. (With permission from (17).)

2,4,6-trinitrotoluene, its by-products and its degradation products were chromatographically separated in a single analysis. LC–MS equipped with an atmospheric pressure chemical ionization (APCI) interface was used. Mathis et al. (17) analyzed the high explosives (Figure 6) by liquid chromatography/electrospray ionization mass spectrometry. The negative ion electrospray ionization mass spectrometric (ESI-MS) detection of adducts of high explosives with chloride, formate, acetate and nitrate was used to demonstrate the gas-phase interaction of neutral explosives with these anions. The relative intensities of the adduct species were determined to compare the competitive formation of the selected high explosives and anions. This method provides a qualitative and quantitative approach for the analysis and identification of high explosives. Shin et al. (18) analyzed the anaerobic biotransformation of dinitrotoluene isomers. *Lactococcus lactis* subspecies lactis strain 27 was isolated from earthworm intestine. In this study, the anaerobic biodegradation of four dinitrotoluene isomers, i.e., 2,3-, 2,4-, 2,6- and 3,4-DNT, were investigated using *Lactococcus lactis* subsp. lactis strain 27.

Xu et al. (20) employed HPLC-API-MS for the analysis of nitroaromatic, nitramine and nitrate ester explosives in his investigation. Various experimental conditions have been applied to allow for the detection of all explosive compounds. The API method was shown to have distinct advantages, including simplicity and stability, etc. Xu et al. (21) analyzed peroxide explosives, e.g., hexamethylenetriperoxidediamine (HMTD) and triacetonetriperoxide (TATP) by HPLC-APCI-MS/MS in forensic applications. With this method, HMTD and TATP were analyzed in the same run. The method was applied successfully for the identification of peroxides in the bulk solid state (powder sample), as well as in post-blast extracts originating from a forensic case. For the post-blast extracts, the use of tandem MS was has great importance for the identification and detection of the peroxide explosives (Figure 7). Szecsody et al. (25) analyzed the abiotic sorption and oxic degradation of the explosive CL-20 during transport in subsurface sediments. RDX and CL-20 were detected with UV lamp while CL-20 degradation products were detected by MS during analysis.

Subsurface environment were investigated to determine the groundwater contamination. Sorption of aqueous CL-20 was relatively small which results in only slight retardation relative to water movement. Thus, CL-20 could move quickly through unsaturated and saturated sediments of comparable composition to groundwater. Sanchez et al. (32) analyzed nitroaromatic explosive compounds in air samples at femtogram level using C_{18} membrane sampling and on-line extraction with LC-MS. In the procedure analytes were desorbed after sampling then separated onto a porous graphitic carbon (PGC) HPLC column and finally were analyzed by an LC-MS/MS detector equipped with an atmospheric pressure chemical ionization (APCI) interface. Campbell et al. (33) analyzed the nitroaromatic explosives with LC-MS from soil samples from OB/OD sites. Mathis et al. (37) analyzed compositional variation in the organic additives of smokeless powder incorporating gradient reversed-phase liquid chromatographic-electrospray ionization mass spectrometric (LC-ESIMS).

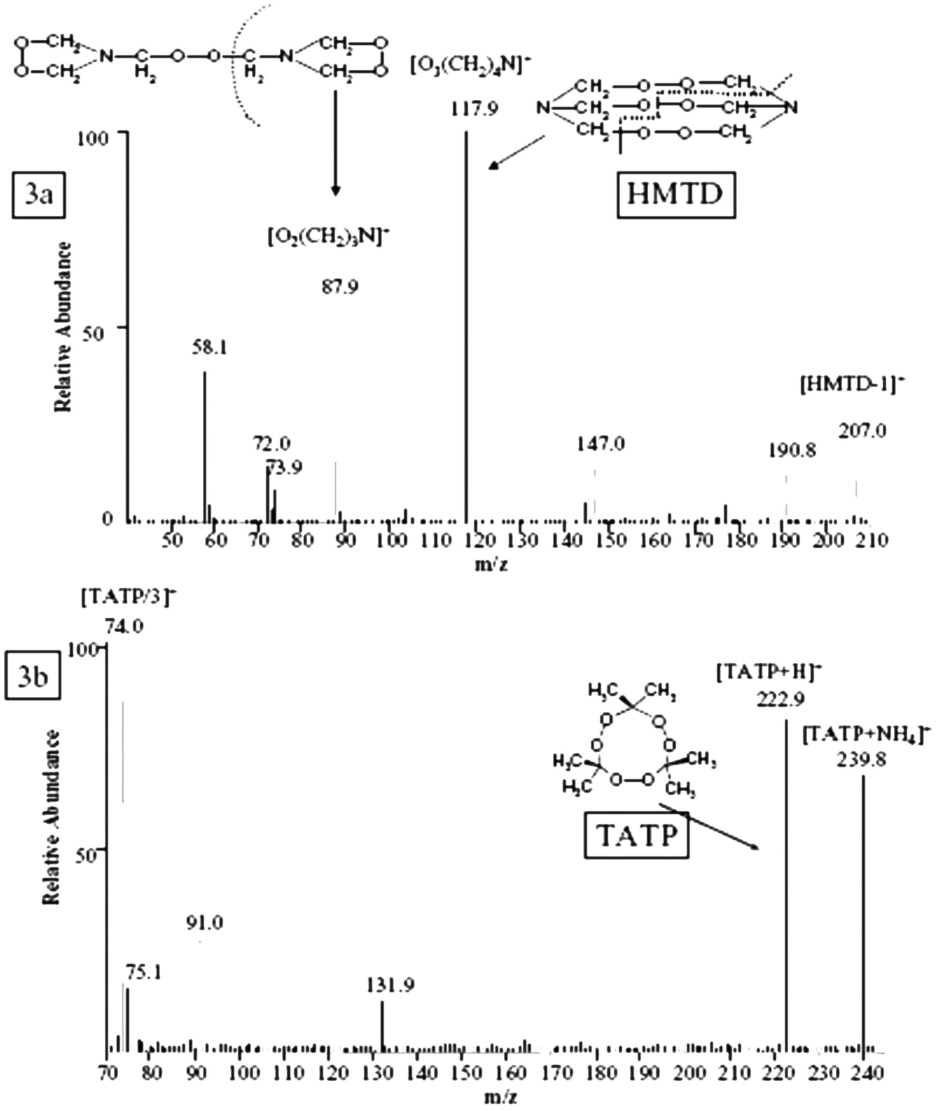


FIG. 7. HPLC-MS/MS spectra of the reference peroxide HMTD (a) and TATP (b) obtained in the Full Scan mode. The precursor ions selected were for HMTD m/z 207 and for TATP m/z 240. (With permission from (21).)

Beller et al. (44) analyzed the bacteria enriched from RDX-contaminated aquifer sediments consumed RDX in a defined, bicarbonate-buffered, anaerobic medium containing hydrogen as the sole electron donor and RDX as a potential electron acceptor and sole nitrogen source. RDX was not consumed in live controls that did not contain hydrogen. Transient formation of mononitroso- and dinitroso-RDX metabolites (hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine and hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine, respectively) was documented by liquid chromatography- mass spectrometry. Widmer et al. (46) analyzed triacetone triperoxide (TATP) using LC/MS. Due to the lower temperatures used in LC, the problem of stationary phase activation was not encountered. Zhao et al. (47) analyzed nitrate ester explosives by liquid chromatography-electrospray ionization and atmospheric pressure chemical ionization mass spectrometry in the negative-ion mode. Three widely used nitrate ester explosives analyzed were namely pentaerythritol tetranitrate, nitroglycerin and ethylene glycol dinitrate, as well as six additional nitrate esters. Beller et al. (50) used the liquid chromatography/tandem mass spectrometry to detect distinctive indicators of in situ RDX transformation in contaminated groundwater.

Zhao et al. (53) analyzed TNT and its byproduct isomers including trinitrotoluene, dinitrotoluene, trinitrobenzene and dinitrobenzene by LC-APCI-MS. LC–MS with APCI, in the negative-ion mode, was found to be the most suitable method. Crowson et al. (55) detected and quantified the trace amounts of hexamethylenetriperoxidediamine (HMTD) a primary organic peroxide by LC-MS. LC-MS is well suited to the analysis of explosive compounds, such as HMTD, that are thermally labile. This property of HMTD has prevented other chromatography

separation techniques, such as GC-MS, from being successfully employed for the analysis of HMTD.

The cyclic nitramine explosives RDX and HMX were examined in field and microcosm soil samples to determine their patterns of degradation and environmental fates by Groom et al. (57). A number of analytical techniques, including SPME with on-fiber derivatization, GC-MS, GC-ECD, LC-MS and MEKC, were required for the analyses. Two different classes of intermediates were detected, both of which lead ultimately to the formation of N_2O and CO_2. The first class was identified as the nitroso derivatives formed by the sequential reduction of nitro functional groups. The second class of intermediates consisted of ring cleavage products including bis-(hydroxymethyl)nitramine and methylenedinitramine. Rye grass present in field samples was found to extract and accumulate HMX from soil without further degradation.

Rodgers et al. (59) analyzed the changes in concentration of 2,4,6-trinitrotoluene and 2,4- and 2,6-dinitrotoluenes and some of their electrolysis products during electrochemical reduction in aqueous solution. Phillips et al. (68) analyzed aqueous swab samples taken from cars and road signs before and after controlled firings. Schreiber et al. (69) elaborated the application of spectral libraries for high-performance liquid chromatography-atmospheric pressure ionization mass spectrometry to the analysis of explosive residues in environmental samples. Libraries of mass spectra made the identification of unknown substances in complex environmental samples easier and more user-friendly. Thompson et al. (75) recovered and analyzed nitroexplosives from cotton swabs. Nitro-organic explosives were extracted from cotton swabs, isolated and screened by LC with UV detection. The H_2O extraction/SPE process gave greater selectivity with most samples. Schreiber et al. (81) investigated the applicability of spectral libraries in HPLC-MS. 45 explosives-related compounds were compiled from data obtained by HPLC-MS with use of a PE-Sciex API 100 system. The best results were obtained when conditions used to obtain the library and the test spectra were the same. The technique was used to identify explosives and their breakdown products in run-off water from an explosives dump. Cassada et al. (83) determined the RDX and nitroso-RDX metabolites and other munitions from water samples with increased sensitivity and selectivity.

Duff et al. (88) described the advantages of Allure C18 HPLC column, a high-carbon (27%), densely bonded C18 phase. Its advantages over most commercial C18 phases were higher LC-MS sensitivity, maximum retention of neutral to slightly polar solutes, symmetrical peak shape for basic compounds, high reproducibility, separation of all EPA method 8330 explosive compounds on one column and high stability towards hydrolysis. Astratov et al. (99) identify and analyzed the nitroexplosives and pollutants from groundwater samples of an ammunition hazardous waste site. Cappiello et al. (100) analyzed four widely used explosives based on reversed-phase liquid chromatography coupled to a quadrupole mass spectrometer. A microflow rate particle beam interface was employed that offers simplified operation procedures and improved interfacing performance. A positive role played by the reduced size of the aerosol droplets generated by the microflow rate interface was outlined in this work. Greater vaporization efficiency and negligible thermal decomposition were observed for the selected compounds in the ion source of the mass spectrometer. Electron capture ionization allowed specific and sensitive determination of the analytes. Gates et al. (105) analyzed nitoaromatic explosives and their degradation products from water samples. Methods used were MS and tandem MS, coupled with HPLC and photodiode-array detection.

Casetta et al. (116) characterized and subsequently detect the explosives with mass spectrometry. Verweij et al. (121) analyzed the explosives from post blast residues employing liquid-chromatographic, thermospray-negative-ion, tandem mass-spectrometric (LC-TSP-MS-MS) method with greater selectivity as compared to conventional electrochemical methods. Yinon et al. (135) analyzed the metabolites of TNT from the urine of rats, in the blood of rabbits and in the urine of munition workers. Yinon et al. (140) also analyzed the 2,4,6-trinitrotoluene and its metabolites in urine of munition workers by micro liquid chromatography-mass spectrometry. Voyksner et al. (141) analyzed the explosives from hand swabs with mass spectrometry. This method was useful for identification and showed excellent sensitivity and specificity of components of commercial explosives, without interference from plasticizers and for detecting explosives in hand swabs. Yinon et al. (142) analyzed 2,4,6-trinitrotoluene and its metabolites in blood of rabbits by high-performance liquid chromatography-mass spectrometry. Yinon et al. (152) analyzed the explosives in which a direct liquid-insertion probe h.p.l.c. -m.s. interface was used with a home-built mass spectrometer to obtain mass spectra. Parker et al. (159) analyzed explosives by liquid chromatography-negative-ion chemical-ionization mass spectrometry.

HPLC-Electrochemical Detection

This detector is based on the measurements of the current resulting from oxidation/reduction reaction of the analyte at a suitable electrode. So the level of the current is directly proportional to the analyte concentration, therefore this detector could be used for quantification. Thus the combination of UV and electrochemical detection for the analysis of complex samples is more advantageous.

Marple et al. (15, 16) determined the nitroaromatic and nitramine explosives from environmental samples including groundwater and soil with PAED detection. Schulte-Ladbeck et al. (35) analyzed the air samples for the determination of triacetonetriperoxide (TATP). The high volatility of the peroxide leads to significant concentrations in the air surrounding even minute quantities of TATP. Schulte-Ladbeck et al. (40) analyzed the peroxide-based explosives using reversed-phase HPLC with post-column UV irradiation and electrochemical detection for analysis of triacetonetriperoxide (TATP) and hexamethylenetriperoxidediamine (HMTD). The analytes were

TABLE 2
Characteristics of the HPLC methods for the analysis of explosives

Sr. No.	Analyte	Matrix	Preconc. Tech.	Fiber/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
1	RDX and its N-nitroso derivatives	Soil	PLE	Acetonitrile	MS	Supelco RP C18 column (250 mm × 4.6 mm, 5 μm) at room temperature	Isocratic flow of methanol/water mixture (60:40) containing 1.0 mM aqueous acetic acid	0.5 mL/min	LOD for TNX, DNX, MNX, and RDX, were 1.93, 1.69, 1.46, and 1.46 ng/g, respectively	9
2	HMX	Lizard egg extracts	PLE	Acetonitrile	MS	Supelco RP C18 column (250 mm × 4.6 mm; 5 μm)	Isocratic flow of methanol/0.5 mM aqueous acetic acid mixture; v/v (60:40)	0.5 mL/min	0.78 g	10
3	TNT and its transformation products	Aqueous sample	NA	NA	UV	Nova–Pak C8 reversed phase column (3.9 mm × 150 mm; 4 μm)	Isopropanol/water (18:82)	1.0 mL/min	254 nm, 3 μg/L	11
4	Nitroaromatic, nitroamines and nitrate ester explosives	Explosive mixture and soil containg nitroaromatics only	MAE & SPE for soil	For MAE: 0.1 M sodium phosphate pH 8 buffer For SPE: Abselut NEXUS adsorbent from varian	UV/MS	Thermo Quest hypercarb PGC column (250 mm × 4.6 mm i.d.; 5 μm particles) at 30°C	Gradients of A,B and C (A) 49.5% water, 9.9% methanol, 39.6% acetonitrile and 1.0% dichloromethane; (B) 73% methanol, 25% acetonitrile and 2% toluene; (C) 25% acetonitrile and 75% toluene.	0.9-1.4 mL/min	290 nm, 0.5 to 41.2 ng	13
5	RDX, TNT	Snow	SPE	Porapak RDX SPE cartridge (Sep-pak, 6 cm^3, 500 mg)	UV	Reverse Phase; (15 cm × 3.9 mm) Novapak C_8; 28°C	(15:85) isopropanol/ water (v/v)	1.4 mL/min	254 nm	14

(Continued on next page)

TABLE 2
Characteristics of the HPLC methods for the analysis of explosives *(Continued)*

Sr. No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
6	Nitro and nitramine explosives	Water and soil	SPE for water And PFE for soil	For SPE: C18 column (4.6 × 75 mm; 5 μm) solvent is 7.5% methanol in a solution of 20 mM acetate and 0.5M NaCl, flow rate 1.0 mL/min ; 100% methanol for PFE	UV/ PAED	Rev. phase C-18 (4.6 × 250 mm; 5 μm) at 30°C	Methanol/20 mM acetate buffer at pH 4.5 (50/50;v/v)	1.0 mL /min	254 nm, 366 nm; 0.007- 3 μg/L (PAED) 0.9–5 μg/L (UV)	15
7	Nitro Explosives	Environmental sample/water	SPE	Rev Phase C18 (4.6 mm × 75 mm; 5 μm). Loading solvent 7.5% methanol in a solution of 0.5 mM NaCl & 20 mM sodium acetate trihydrate at pH 4.5; flow rate 1.0 mL/min	UV/PAED	Rev Phase C18 (4.6 mm × 250 mm; 5 μm) with guard column C8 (4.6 mm × 3.0 mm; 5 μm) at 30°C	50% methanol in 20 mM acetate buffer at pH 4.5	1.0 mL/min	254 nm, 0.0007-0.4 μg/L (For PAED) 0.04-0.4 μg/L(for UV)	16
8	EGDN, NG, TNT, PETN, RDX, HMX	Explosives	NA	NA	MS	Agilent C_{18} column (2.1 × 100 mm) hypersil ODS	50% MeOH / 50% aq. mixture	0.15 mL/min	m/Z 30-400	17
9	DNT and its metabolite	Earth- worm intestine	Solvent Extrac-tion	Ethyl acetate	PDAD/ MS	For PDAD : Waters ODS C18 (4.6 mm × 25 cm; 5 μm) For MS: C18 rev phase column (4.6 mm × 25 cm; 5 μm)	Water/acetonitrile mixture water/acetonitrile containing 1% formic acid	1.0 mL/min 1.0 mL/min	270 nm	18

10	Mono, di and trinitro naphthalenes	Soil	Ultrasonic & Soxhlet Extrac-tion	Ultrasonic: Acetonitrile/methanol (50:50, v/v) soxhlet: acetone	DAD	Nucleosil silica column 120-3 C18 (250 × 4 mm) with a precolumn packed with same material (3 μm, 120A°, octadecyl phase; endcapped); 10°C	Gedients of methanol/ water	0.45 mL/min	230 nm	19
11	Nitramine, nitroaromatic and nitrate ester explosives	Post blast debris	Sonification	Methanol	MS	Nova pack 4 μm C18 cartridge column (3.9 × 150 mm)	Methanol/water (v/v, 1:1) and/or ammonium acetate, glycine	0.4 mL/min	0.012–1.2 ng (full scan mode)	20
12	HMTD, TATP	Powder and post blast debris	Sonification	Acetone or methanol	MS	Nova pack 4 μm C18 cartridge column (3.9 × 150 mm	Methanol/ water (v/v, 75:25) with 2.5 mM ammonium acetate; isocratic	0.4 mL/min	HMTD: 0.26 ng TATP: 3.3 ng (full scan mode)	21
13	HMX, RDX, Tetryl, TNT, DNT isomers, 3-NT, PETN	Soil	Sonication	Acetonitrile	PDAD	Rev Phase 50 × 4.6 mm Chromolith SpeedROD RP-18e (Merck) and a 100 × 4.6 mm Chromolith Performance RP-18e (Merck).	Gradients of methanol/ water	0.2–10 mL/min	254 and 210 nm	22
14	HMX, RDX, 1,3-DNB, 3,4-DNT, TNT, 4A-2,6-DNT, 2,4-DNT	Water	SPME	50 μm film of CW/ TPR, 60 μm film of PDMS/DVB, 85 μm film of PA, complete immersion; 30 min, room temp, static soaking in 50 μL of 1:1 (v/v) water/ acetonitrile solution for desorption, rate of stirring: 500 rpm	UV	C18 column (25 cm × 4.6 mm; 5 μm) at 35°C	Isocratic 50% methanol/ water	0.75 mL/min	254 nm 1–10 μg/L	23

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TABLE 2
Characteristics of the HPLC methods for the analysis of explosives *(Continued)*

Sr. No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
15	2,6-DNT & Picric acid and their transformation products	Marine sediments & water	Ultrasonic Extraction	Acetonitrile	UV	ODS C18 column (25 cm × 4.6 mm)	Isocratic mixture of 35% methanol and 65% 0.1 M sodium acetate at pH 4.8 for picric acid and its metabolite	NR	250 nm	24
16	CL-20, RDX	Sediments	Filtration	0.45 μm teflon	UV/MS for degradation products	Keystone NA C18 column (250 mm × 4.6 mm)	Methanol/water (55:45) ; isocratic flow	0.8 mL/min	230 nm	25
17	CL-20	Soil	Extraction with sonication at 20°C	Acetonitrile	DAD	Supelcosil LC-CN column (25 cm × 4.6 mm; 5 μm) at 35°C	70% aq. methanol (isocratic)	1.0 mL/min	200–350 nm $\lambda = 230$ nm	26
18	2,4,6-TNT, EPA method explosives	Environmental sample	Evaporation for TNT metabolites	Acetonitrile	DAD	Rev Phase Supelcosil octyl C8 column (150 mm × 4.6 mm; 5 μm) and guard column LC-8 (20 mm × 4.6 mm; 5 μm) at 35°C–55°C	Gradients of aq. methanol	1.0 mL/min	200–600 nm absorbance at 220, 230, 254, 360 & 370 nm	27
19	TNT & its metabolite, HMX, RDX	Soil	Sonication	Acetonitrile	DAD	For TNT & its metabolites: Supelcosil C8 column (25 cm × 4.6 mm i.d.; 5 μm) For RDX, HMX: LC-CN column (25 cm × 4.6 mm i.d.; 5 μm) at 35°C	For TNT & its metabolite: 82% v/v of water and 18% v/v of 2-propanol For RDX & HMX: Gradients of methanol/ water	1.0 mL/min	For TNT and its metabolites: 25 and 50 ppb respectively, 254 nm For RDX, HMX : 50 and 100 ppb resp (200–350 nm)	28

20	2,4-DNT	Soil	Solvent extraction	Methylene chloride	UV	Supelcosil LC-18 column (150 mm × 4.6 mm)	Water/methanol (54:46)	1.0 mL/min	254 nm	29
21	RDX	Human plasma	SPE	Tox-clean RC (C18) SPE cartridges (225 mg/ml)	DAD	C18, 5 mm Luna column (150 mm ×/4.6 mm i.d., Phenomenex, CA) fitted with guard column (4 mm L ×/3 mm i.d., Phenomenex, CA) packed with same material.	Isocratic flow, acetonitrile//water; 35/65 (v/v).	1.0 mL/min	240 nm	30
22	Arsines and TNT	Explosive & arsine compound mixture	NA	NA	UV-DAD	Rev Phase, Zorbax Rx C18 (4.6 × 150 mm; 3.5 μm) at 30°C	Isocratic flow of Methanol/water (40:60) followed by gradient elution	NR	210 nm 0.39 ng 1.87, 1.94, 0.12 ng for phenyldichloroarsine, diphenyl chloroarsine and triphenylarsine respectively	31
23	TNT, DNT-isomers, DNB-isomers, TNB	Air	SPE	Sampling with 47 mm empore octadecyl SPE membrane and extracted with polyether ether ketone (PEEK)	MS	Hypercarb analytical column (100 × 4.6 mm; 5 μm)	Solvent A: Water/acetonitrile/ methanol (50:40:10 ; v/v) solvent B: methanol/acetonitrile/ toluene(73:25:2; v/v)	For B: 1.0 mL/min and 2.0 mL/min	Femtogra m/L range	32
24	2A-4,6-DNT, 1,3-DNB, 2,4-DNT, NB, RDX,Octogen, 1,3,5-TNB, TNT	Soil	PFE	Methanol/ Acetonitrile (1:1, v/v)	DAD/ MS	HP zorbax SB C_{18} (narrow bore 2.1 cm × 155 mm; 5 μm) at 44°C	Gradient flow of water/methanol	0.7 mL/min	254 nm, 0.05 μg/g for 2-A-DNT,0.3 μg/g for TNT and HMX	33
25	CL-20, HMX, RDX and their degradation intermediates	Environmental sample	NR	NR	DAD	For HMX, RDX, CL-20: Supelcosil LC-CN (25 cm × 4.6 mm; 5 μm) at 35°C For nitroso derivative and ring cleavage product: LC-CN Column (25 cm × 4.6 mm; 5 μm) at 35°C	70% aq. methanol isocratic run Gradient flow of methanol/water	1.0 mL/min 1.0 mL/min	200–350 nm, λ = 230 nm 0.02 mg/L MS 0.004 mg/L	34
26	TATP	Air	Air sampling	Acetonitrile	UV/ECD	Merck LiChrospher RP-18 (5 μm) poresize 300 A° (250 mm × 3 mm)	65% acetonitrile and 35% of 4 mM/L phosphate buffer of pH 8	0.5 mL/min	550 ng/L 254 nm	35

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TABLE 2
Characteristics of the HPLC methods for the analysis of explosives *(Continued)*

Sr. No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
27	RDX, TNT, 1,3,5-TNB, 2,4-DNT, NT-Isomers	Water	SPE	SPE material (Lichrolut EN or Porapak R) filled 1/16 in.O.D.x 750 μm i.d. PTFE tubing; 3.0 mL/min, acetonitrile	UV	Supelco LC-18 (25 cm $\times$ 4 6 mm; 5 μm)	Methanol/ water (50:50; v/v)	NR	243 nm	36
28	Nitro & nitroso derivative of diphenylamine, centralite I & II, di alkylphthalate acid esters	Smokeless Powder	Solvent extraction	Methylene chloride	MS	Restek pinnacle octyl column C8 (2.1 $\times$ 100 mm; 3 μm) 120A$^\circ$ pore size at room temp	Gradients of methanol/1 mM aq. ammonium acetate	0.25 mL/min	230 nm m/z from 50–500 a.m.u.	37
29	TATP, HMTD	Sample from explosion site	Elution	acetonitrile	UV/ fluoresence	LiChrospher RP18 (250 $\times$ 3 mm; 5 μm)	Acetonitrile/ Water (60:40) isocratic flow	0.6 mL/min	254 nm, 2 μM for each; excit-ation λ 324 nm, emission λ; 405 nm; 2 $\times$ 10^{-6}mol/L	38
30	HMX, RDX, derivatives of phenol and toluene, DPA, TNB, DNB and tetryl	Explosive mixture	NA	NA	DAD	Octadecyl-modified silica C18 column (250 $\times$ 4 mm i.d.; 5 μm)	Methanol-water or methanol- phosphate buffer mixture	0.8 or 1.0 mL/min	254 nm	39
31	TATP, HMTD	Explosive mixture	NA	NA	ECD	Merck LiChroSpher RP18,pore size 300 A$^\circ$; (250 $\times$ 3 mm; 5 μm)	Solvent mixture of 65% acetonitrile and 35% aqueous 4 mM phosphate buffer at pH 8	0.5 mL/min	3 μM for each	40

32	RDX, HMX, TNT and biodegradation products	Water and soil	Elution and filtration	Acetonitrile	UV	C8 rev phase column (150 mm × 3.9 mm) with a guard column of same matrix	Water/acetonitrile (65:35)	0.8 mL/min	220 nm	41
33	Tetryl	Soil	Ultrasonic extraction	Acetonitrile	DAD	Pinnacle octyl C18 RP column	Methanol/ water (1:1; v/v)	0.85 mL/min	230 nm	42
34	HMX, RDX, NG and TNT	Snow	Salting out solvent extraction & soxhlet extraction	Acetonitrile	UV/ PDAD	For UV: Nova Pak C8 (Waters Millipore) (15 cm × 3.9 mm; 4 μm) For PDAD: Supelco LC-18 column (confirmatotry)	For UV: 15:85, isopropanol/ water For PDAD: 60:40 methanol/water	1.4 mL/min 1.2 mL/min	254 nm	43
35	RDX, MNX, DNX, TNX	Bacterial culture	NA	NA	MS	Nova-Pak C18 column (4- μm particle size, 60 A°, 2.1-mm i.d.x 150-mm; and a 20-mm guard column containing the same stationary phase.	Methanol/water (1:1)	200 μL/min	—	44
36	TATP	Explosive Solution	NA	NA	MS	FEL Study: Pro C18 analytical column (150 mm × 2.0 mm; 3 μm) with Pro C18 gaurd column (10 mm × 2 mm) at 20°C SRS Study: spherisorb ODS C18 Column (2.1 mm × 150 mm) at 30°C	70:30 (methanol/water) with 5 mM ammonium acetate 85:15 (methanol/ water) with a 5 mM ammonium formate buffer	0.1–0.2 mL/min 0.1 mL/min	100 pg / μL; m/z :50–300 a.m.u. 10 ng/100 μL m/z: 100–350 a.m.u.	46
37	PETN, NG, EGDN, BTTN, BTDN, 1,2-DNG, 1,3-DNG, 1-MNG, 2-MNG	Forensic sample	NA	NA	MS	In ESI Mode: Restek allure C18 column (100 × 2.1 mm; 5 μm) In APCI mode: Restek allure C18 column (150 × 3.2 mm;5 μm)	Methanol/water (70:30) Methanol/Water (70:30)	150 μL/min 400 mL/min	5.5 pg/ μL for PETN & NG respet. 2ng/ μL for EGDN in (ESI Mode)	47

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TABLE 2
Characteristics of the HPLC methods for the analysis of explosives (Continued)

Sr.No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
38	RDX, HMX, TNT	Water, soil, plant tissue	SFE, SPME	In SFE: Carbon dioxide, acetonitrile In SPME: Sep-pec Porapac RDX cartridge	UV	Supelcosil C8 column (25 cm × 4.6 mm; 5 μm) at 35°C	Water/2-propanol (82:18)	1.0 mL/min	254 nm	48
39	TNT, DNB & DNT isomers	Air	SFE	Carbon dioxide C-50 quality under helium headspace	UV	Hypercarb analytical column (100 mm × 4.6 mm; 5 μm) at 60°C	Gradient elution of A and B A: milli-Q water B: acetonitrile/ 2-propanol (90:10 v/v)	0.8 mL/min	254 nm	49
40	RDX, MNX, DNX, TNX, MEDINA	Water	Centrifugation	NA	MS	Novapak C18 column (150 mm × 2.1 mm i.d.; 4 μm), 60A°	Methanol/ water (50:50; v/v)	200 μL/min	0.1 μg/L	50
41	TNT and its degradation products	Soil	Slurry formation and Extraction	Slurry in acetone	PDAD	Alltech C18 anion column (25 cm × 4.6 mm i.d.) and C18 guard column; 2 cm)	Methanol/water (1:1)	0.64 mL/min	254nm 39100 ppm	51
42	TNT, TNB 2,4-DNT, 2A-4,6-DNT, 4A-2,6-DNT, 2,4-DA-6-NT, 2,6-DA-4-NT, Tetryl, RDX, HMX, TNG	Dermis and receptor fluid	Extraction by sonicatio	Methanol	UV	Supelcosil LC-18-S column (250 × 4.6 mm; 5 μm) and guard column (supelguard LC-18-S;2 cm)	Methanol/water (1:1), isocratic flow	1.0 mL/min	254 nm	52
43	TNT and its by product isomer	Explosives	NA	NA	MS	Restek Rev Phase Allure C18 column (150 × 3.2 mm; 5 μm)	Methanol/isopropanol/ water or methanol/ water	0.4 mL/min	NR	53
44	TNT, HMX, RDX	Soil	Sonication	Acetonitrile	UV	Nova pak C8 (15 cm × 3.9 mm; 4 μm)	(15:85) isopropyl alcohol/water	1.4 mL/min	254 nm	54
45	HMTD	Explosive mixture	NA	NA	MS	YMC Pro C18 column (150 mm × 2 mm i.d.) and YMC Pro C18gaurd column (10 mm × 2.0 mm)	Water/methanol (95:5)	0.2 mL/min	20 pg/μL i.e., 2ng/100 μL	55

46	RDX, HMX, TNT	Explosive mixture	NA	NA	UV	Capilliary ; HP, 200 μm i.d., 320 μm o.d. 1.5 m length (5 μm) ODS at 28°C	32.5% Acetonitrile/water	1.0 μL/min	254 nm	56
47	RDX, HMX and degradation products	Soil and plant tissue	Sonication for plant tissue	Acetonitrile	PDAD/MS	Supelcosil LC-CN Column (25 cm × 4.6 mm; 5 μm) at 35°C	Gradients of methanol/ water	1.0 mL/min	230 and 254 nm ESI mode, m/Z 40–400 a.m.u.	57
48	RDX, HMX	Sediments	(i) ASE (ii) Soxhlet	For ASE: (i) acetonitrile (ii) acetone/methanol (1:1) For Soxhlet: Acetonitrile	UV	(i) Supelco (25 cm × 4.6 mm) LC-CN column (ii) Supelco (25 cm × 4.6 mm) LC-C18 column	Methanol/ water (60:40,v/v)	1.0 mL/min	12.07 mg/g for RDX and 7.68 mg/g for HMX	58
49	TNT, 2,4 & 2,6-DNT and electrolysis product	Aq. Solution	NA	NA	UV/MS	Reverse phase C_{18} column (25 cm × 4.6 mm i.d.)	Methanolic 60–30% sodium phosphate buffer of pH 2	0.5-1.0 mL/min	254 nm MS detection for non volatile electrolysis products	59
50	Nitro aromatic, nitramine explosives	Water	SPE	Bond Elut ENV	UV-DAD	Microsorb ODS C18 column (25 cm × 4.6 mm i.d.; 5 μm)	50% aqueous methanol and/or aq. 85% propan-2 –ol (isocratic or gradient)	1.5 mL/min	254 nm 50 ppb	60
51	Explosives	Water	SPE	Styrene –divinyl benzene cartridge	UV	C18 length = 25 cm dp=5 micron	Methanol/Water (1:1, v/v), isocratic	1.2 mL/min	245 nm	61
52	PETN, 1,3-DNB, 2,4-DNT, 2,6-DNT, NG, 2-NT, 4-NT, Octagen, PETN, RDX, 1,3,5-TNB, TNT	Water	SPE	Bond Elute SDB (Empore) cartridge	UV	C18	NR	NR	210 nm for NG and PETN 254 nm for blanc	62
53	HMX	Soil	Suspension	water	PDAD	Supelcosil C8 column (25 cm × 4.6 mm i.d.; 5 μm) at 35°C	82% water (v/v) 18% 2-propanol	1.0 mL/min	λ = 254 nm; 25 ppb	63
54	TNT, NB, DNT	Aq. Solu-tion	Filtration	Millipore filters (0.45 μm)	PDAD	Brownlyn Spheri-10 RP -18 (100 mm × 4.6 mm)	Acetonitrile/ water (35:65)	1.6 mL/min	250 nm	64

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TABLE 2
Characteristics of the HPLC methods for the analysis of explosives *(Continued)*

Sr. No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
55	Nitro-aromatic explosives	NR	NR	NR	UV	250/3 Nucleosil 120-3 C18 column at 30°C	Gradients of Methanol/ water	0.35 mL/min	230 nm	65
56	NB, RDX, HMX, 2,4 & 2,6-DNT, 4-NT	Environmental samples	PFET	Electrochemical reduction with carbon electrode	Photoluminescence	1 × 100 mm betabasic C18 column	Mobile phases containing 7% (v/v) of 2-propanol	NA	Low ng range	66
57	Nitro explosives	Water and soil	SPME	CW/TPR fibre and desorption with methanol: Water (1:1) at flow rate 0.2 mL/min	UV	Combination of Res-Elut CN column (3 cm × 4.6 mm i.d.;5 μm) and a Bodensil C-18 column (25 cm × 4.6 mm i.d.; 5 μm)	Isocratic flow of methanol: water (1:1)	NR	220 nm for EGDN, NG and PETN and 254 nm for all other explosives, 5-16 ng/ mL from water & 10-40 μg/Kg from soil	67
58	Nitro-aromatic explosives	Environmental sample	NA	NA	MS	RP-18 column Ultrasep; ES; 250 × 4 mm; 5 μm)	Methanol/ water (41:59 ; v/v) and 5 mM ammonium acetate at pH 5	0.6 mL/min	NA	69
59	RDX, TNT, HMX, TNB, Tetryl, TNT metabolites etc.	Water, substrate & plant tissues	sonication	Acetonitrile at 30°C	^{14}C labeling, LSC	C18 column and CN column	NR	NR	245 nm 0.2–2.0 μg/g FW^{-1} in sediments	71
60	HMX, RDX, TNT, 2-NT, 4-NT, 2,4 and 2,6-DNT, 1,3,5-TNB	Explosive mixture	NA	NA	UV	(25 cm × 4 mm i.d.) at 25°C	Aq. Methanol Isocratic/gradient	1.0 mL/min	254 nm	72
61	RDX, HMX, TNT and other nitro aromatic explosives	Soil extracts			UV	Zorbax 5 SB-C18 column (25 cm × 3 mm i.d.)	Aq methanol (50%)	0.3 mL/min	230 nm	73
62	Nitro aromatic and nitramine explosives	Explosive mixture	NA	NA	UV	Res Elut CN guard column (3 cm × 4.6 mm i.d; 5 μm) connected in series with 5 μm Bodensil C18 column (25 cm × 4.6 mm i.d.) at 20–25°C	Methanol/water (1:1)	1.5 mL/min	254 nm	74

63	2,4-DNT, EGDN, HMX, RDX, PETN, TNT, Tetryl	Water	SPE	Porapak RDX cartridge, Oasis brand cartridge containing Polyvinylpyrrolidone-DVB co polymer and SDB-XC disk cartridge containing styrene –DVB copolymer	PDAD/MS	For LC-UV: (4.6 mm × 150 mm; 5 μm) Supelcosil LC-18-DB For LC-MS: Hyrersil ODS column (2.1 mm × 100 mm; 5 μm)	Methanol/water gradient mixture Methanol/1.0 mM ammonium nitrate mixture; gradient flow	0.80 mL/min 0.3 mL/min	Nitrate esters at 210 nm and nitramine and nitroaromatic at 240 nm	75
64	Hexanitro-hexaazaisowurtzitane				UV	5 μm HP ODS C_{18} column (12.5 cm × 4 mm i.d.)	50% methanol	1.0 mL/min	230 nm	76
65	Explosives including TNT, HMX & RDX	Water and soil	Centrifugation for soil	acetonitrile	UV/ Amperometric	5 μm supelcosil LC-PAH column (15 cm)	Deoxygenated acetonitrile/50 mM phosphate buffer at pH 5 (1:2) containing 18 mM –SDS	1.0 mL/min	230 nm, 9-550 nM, Carbon- electrode at -0.8 V *vs.* Ag/AgCl, with a Pt counter electrode	77
66	TNT, HMX, RDX	Waste water	NA	NA	UV	Supelcosil LC-18 column (25 cm × 4.6 mm i.d; 5 μm.)	Methanol/water (1:1)	1.5 mL/min	254 nm	78
67	RDX, TNT and their metabolites	Plant tissue	Ultrasonic extraction	Acetonitrile	UV	C_{18} and CN reverse phase columns at 30°C	Methanol/acetonitrile	NR	245 nm	79
68	EPA 8330 explosives	Explosive mixture	SPME	50 μm; CW/TPR fibre ; static desorption	UV	5 μm Res –Elut CN column (3 cm × 4.6 mm i.d.) connected in series to 5 μm Bodensil C-18 column (25 cm × 4.6 mm i.d.)	Methanol/Water (1:1)	1.3 mL/min	254 nm	80
69	45 Explosive related compounds	Waste water					Aq. 75% methanol containing 5 mM ammonium acetate	0.6 mL/min		81
70	TNT, HMX	Soil	Solvent extraction	Acetone/water (97:3) for 30 min	UV	Supelcosil LC-CN column	Water/Methanol (1:1)	1.2 mL/min	254 nm	82
71	RDX and nitroso RDX metabolite	Water	SPE	SPE cartridge	MS	Kromasil Reverse phase C8 column (250 × 2 mm)	Isocratic; methanol/water/0.5 M ammonium formate (50:48:2) OR Isopropanol/Water/0.5 M ammonium formate (20:78:2) at 30–32°C	0.2 mL/min	IN ESI Mode: 0.03 μg/L for MNX and 0.05 μg/L for RDX	83

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TABLE 2
Characteristics of the HPLC methods for the analysis of explosives *(Continued)*

Sr. No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
72	TNT and its metabolites	Soil	Ultrasonic extrac-tion	Water/methanol/ethyl-acetate	LSC	Hypersil ODS – C18 column (25 cm × 4 mm ; 5 μm)	Isocratic; Methanol/water (35:65, v/v)‘	1.0 mL/min	254 nm	84
73	TNT and its metabolites	Bacterial culture	Solvent extrac-tion	Methylene chloride	UV	HP LC-18 (15 cm × 4.6 mm	Isocratic flow of methanol/ Water (46:54)	1.0 ml/min	254 nm	85
74	TNT and its metabolites	Compost	Solvent extrac-tion	Methanol	DAD	Nucleosil 120-3 C18 column (3 × 250 mm)	Gradients of methanol and water	—	—	86
75	2,4,6-TNT	Bacte-rial culture	Solvent extrac-tion	Ethyl acetate	NR	Zorbax ODS Rev Phase column (250 mm × 4.6 mm; 5 μm)	Propan-2-ol/ water (1:4)	1.0 mL/min	NR	87
76	EPA Method 8330 explosives				MS	Allure C18 column (high carbon 27%) densly bonded				88
77	27 nitro benzenes, nitrotoluenes and metabolites	Water	NA	NA	UV	A) 5 μm nucleosil RP18 column (15 cm × 4 mm i.d.) B) 5 μm Kromasil column (12.5 cm × 4 mm i.d.)	Aq 45% methanol 70% methanol		254 nm 254 nm	90
78	2-NBA, 2,6-DAT, 2,4-DNBSA, RDX, 4-NP, 3,5-DNBA, 3,4-DNBA, TNT, 1,2-DNB, 4A-2,6-DNT, 2,3-DNT	Water	NA	NA	NMR and UV	Merck RP –select B column (75 mm × 4 mm i.d.; 5 μm) and on Merck RP select B column (125 × 4 mm i.d.; 5 μm)	Methanol/D_2O mixture (45:55) buffered to pH 2.3 by phosphate buffer	0.1 mL/min	600.13 MHz 300 nm	91
79	Tetryl, NB, m-DNB, TNT, 2-NT, 3-NT, 4-NT	Water	SPE	Isolute ENV+ column (200 mg/ 6 ml) conditioned with THF and rinsing with water.	UV	C18 Column (25 cm × 4.4 mm i.d.)	Methanol/water (1:1, v/v)	1.4 mL/min	230 or 254 nm	92
80	23 nitro aromatic compounds including HMX, RDX, TNT, Picric acid	Water	SPE	Three LiChrolut EN cartridges at pH 1.0	(i) PDAD (ii) NMR	MERCK LiChrospher 60 RP-select B C18 column (250 mm × 4 mm i.d; 5 μm) LiChrospher 60 RP-select B C18 column (75 mm × 4 mm i.d; 5 μm)	Methanol/water (9:11) adjusted at pH 2.3 with dihydrogen phosphate buffer Methanol/D_2O (9:11) adjusted at pH 2.3 with dihydrogen phosphate buffer	0.5 mL/min 0.017 mL/min	210 nm 600.13 MHz	93

81	RDX, HMX, TNT, Picric acid, and biodegrade-ation products	Water	Subsampling	NA	UV/ECD	LiChrospher 100 RP 8 column (25 cm × 4 mm i.d.; 10 μm) under isocratic condition	Methanol/phosphate buffer at pH 5.9 (2:3)	0.7 mL/min	254 nm +1100 mV for amperometric detection	94
82	16 nitro explosive compounds	Explosive mixture	NA	NA	UV	Stainless steel column (25 cm × 4.6 mm i.d.) at 30°C	Aq. 55% or 60% aqueous methanol	0.5–2.0 mL/min	254 nm	95
83	19 explosives	Water	SPE	Mixed bed of DVB-ethylvinylbenzene co-polymer (LiChrolut EN) and perfluorinated polyethene (PolyF); desorption at 75°C	UV	Zorbax SB C18 or SB –CN columns (15 cm × 2.1 mm i.d.) at 26°C	Gradient elution with Solvent (A) aq 95% acetonitrile and (B) aq 5% acetonitrile containing 5 mM ammonium trifluoroacetate buffer of pH 2.7	1.0 mL/min	240 and 360 nm LOD is less than 100 ng/L	96
84	TNT	Soil	NR	NR	UV	Primary analysis: Supelco LC 18 Confirmatory analysis: Supelco LC-CN column	Aq. 50% methanol (i) methanol/ water (35:65) (ii) acetonitrile/methanol/ water (23:12:65)	1.5 mL/min	254 nm	97
85	TNT, RDX	Plant tissue and field crop	Solvent extrac-tion/SPE	Ethyl ether; Flurisil Sep-Pak cartridge	UV	(24 cm × 4.6 mm i.d.; 5 μm) Ultrasphere C18 column	Gradients of Acetonitrile /water	1.0 mL/min	254 & 234 nm for TNT, RDX respectively 3.70 ppb	98
86	RDX, HMX, TAX, TNT, Hexyl, TNB and derivatives of DNT, DNB, NT, NP, DNA, DNBS, DNB, DNP etc.	water	LLE/ SPE	In LLE: Dichloro-methane In SPE: styrene-DVB copolymer	MS	LiChrospher 100 RP-18, 15 μm (250 mm × 4 mm i.d.)	Isocratic flow; methanol/water (50:50 to 65:35) at pH 2.2–2.5 on rev phase and added ammonium formate (10 mM) on amino phase	0.6 or 1.0 mL/min	0.3 ng	99

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TABLE 2
Characteristics of the HPLC methods for the analysis of explosives *(Continued)*

Sr. No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
87	TNT, RDX, PETN, NG	Explosives mixture	NA	NA	MS	C18 Capillary column made from l/16-in.o.d., 250- μm-i.d. PEEK tubing; 5 μm	Isocratic and gradient flow of acetonitrile/water	1.0 μL/min and 2.0 μL/min	60 pg for NG, 120 pg for TNT, 200 pg for RDX and PETN	100
88	Nitro- phenol, nitrobenzoic acid	Water	Solvent extraction	Dicloro methane	DAD	RP select B column (25 cm × 4 mm i.d.; 5 μm)	Water/Methanol (11:9) at pH 2.3 with dihydrogen phosphate/H_3PO_4 buffer	0.5 mL/min	190–400 nm; 200 pg for nitro phenol and nitro benzoic acid	101
89	RDX, 2,4-DNT, 2,6-DNT, TNT, picric acid, tetryl, NT, aminonitro-toluenes, NB's and HMX	Waste water	Solvent Extraction	Dichloromethane	UV and NMR	Spherisorb C_{18} -2 (25 cm × 4 mm i.d; 5 μm)	Isocratic; methanol/water or methanol/aq0.25 mM KH_2PO_4 of pH 3 (both 57:43)	0.4 mL/min	420 nm (UV) 600 MHz (NMR) 1–10 μg/mL	102
90	Explosive including TNT and RDX	Water	Preconcentration column	(3.7 cm × 3.2 mm i.d.) packed with 75–100 μm DVB –vinylpyrrolidone co-polymer	UV	ultrasphere C18 column (24 cm × 4.6 mm i.d; 5 μm)	Aq. 50% acetonitrile	1.0 mL/min	254 nm 0.1 ng/mL for TNT and RDX	103
91	Fourteen explosives	Water	SPME	Extraction with 65 μm PDMS/DVB fibre and desorption with aq. 50% acetonitrile	UV	Supelcosil LC8 column (15 cm × 4.6 mm i.d.; 3 μm)	Aq 18% propan-2-ol	1.5 mL/min	354 nm	104
92	Nitro aromatic explosive and degradation products	Water	SPE	SPE cartridge	MS/ PDAD	Water's keystone NA column (250 mm × 4.6 mm i.d.) at 27–29°C	Isocratic flow of Methanol /water (42:58);	0.7 mL/min	230 nm, 10–100 ng/L	105
93	Explosives	Water	SPE	Cartridge containing LiChrolut chloro -methylated ethylvinylbenzene/ DVB copolymer mixed with PolyF (perfluorinated polyethylene) at pH 3.5	DAD-UV	Zorbax SB C18 and zorbax SB CN columns at 18°C	Aq 95% and 5% acetonitrile containing 5 mM ammonium trifluoro acetate adjusted to pH 2.7	–	240 and 360 nm; LOD is less than 200 ng/L(general) For 2A-4NT, NT And HEXYL were 208, 260, and 248 ng/L respectively	106
94	RDX	Soil	Solvent extraction	Acetonitrile	UV	5 mm Supelco C18 (25 cm × 4.6 cm; 5 mm)	Water/methanol (1:1)	NR	254 nm	107

95	Nitro aromatic explosive	Water	Solvent extraction	Dichloro- methane	UV/ECD	Eurospher RP -18 column (25 cm × 4 mm i.d.; 5 μm) at 27°C	Methanol/ 0.01M NaH_2PO_4 buffer of pH 3 (51:49)	1.0 mL/min	LOD for sixteen nitro phenol and six amino nitro aromatic compound were 3.25 ng/mL at 1.2 V and 5.30 ng/mL at 254 nm	108
96	Polynitro explosive	Field sample	NR	NR	UV	Eurospher 80–5 C_{18} column (6–8 cm × 2 mm i.d.; 5 μm) at 45°C	Methanol/water/ 0.1M tetrabutyl ammonium phosphate (pH 6.8) (50:40:10)	0.28 mL/min	230 nm	109
97	Explosive and some metabolite of TNT	Biological tissue and fluid, soil, composts and leachates	For Soil: Ultrasonic bathmixing	Acetonitrile	UV	RP C_{18}/anion mixed mode rev phase/ anion exchange column (150 cm × 4.6 mm i.d.) equipped with guard cartridge (1 cm × 4.6 mm i.d.)	Water/ methanol (9:1) containing 0.015 M potassium phosphate, methanol and acetonitrile	1.0 mL/min	254 nm	110
98	Explosives	Water	SPME			Supelcosil LC8				111
99	TNT, RDX	Water	Column con-centration	DVB-vinyl pyrrolidone copolymer (75–100 μm) packed resin bed of (3.7 cm × 0.32 cm)	UV	Beckman ultrasphere C_{18} column (24 × 0.46 cm i.d.; 5 μm) ODS	Isocratic flow; acetonitrile/ water (50:50;v/v)	1.0 mL/min	254 nm, 0.10 ng/ mL for TNT and RDX (10 mL sample)	112
100	14 explosives	Water	SPE	Cartridge of Porapak RDX ultraclean DVB-pyrrolidone at 10 mL/min	UV/ PDAD	Nova –pak C8 column (15 cm × 3.9 mm i.d.)	Aq. 18% propan-2-ol	1.0 mL/min	254 nm LOD< 0.125 μg/L	113
101	Nitro aromatic and nitramine explosives	Explosive mixture	—	—	UV	Supelcosil LC-8 column (15 cm × 4.6 mm i.d.; 5 μm) and supelcosil LC-18 column (25 cm × 4.6 mm i.d.; 5 μm) as confirmatory column				114
102	Explosives	Water and soil	Ultrasonic Extraction	methanol	PDAD	RP 18 (25 cm × 4 mm i.d.; 5 μm)	Methanol/ water (9:11)	0.8 mL/min	50–200 ng/L in water and 50 μg/Kg in soil	115

(Continued on next page)

TABLE 2
Characteristics of the HPLC methods for the analysis of explosives *(Continued)*

Sr. No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
103	HMX, EGDN, RDX, NG, TNT, TETRYL, DNT, PETN, HNS, TNT TENAC, HEXYL, NQ, TNTAB	Explosive mixture, real sample	Extraction for real sample	Acetone	MS	YMC-SP6 - P18 C18 Column (10 cm × 2 mm i.d.)	Gradient elution of acetonitrile/water over 10 min	200 μl	pg range	116
104	Explosives	Waste water	Solvent extraction/SPE	Dichloromethane; Quartz fibre coated with PDMS; adsorption and desorption with Tenax TA	UV	Spherisorb ODS 2 column (25 cm × 4 mm i.d.; 5 μm) with gaurd column (1 cm × 4 mm i.d.)	Aq. 51% methanol	0.8 mL/min	254 nm	117
105	Explosives	Post blast debris	NR	NR	Derivatisation and TEA chemilumine-sensce (UV-Vis)	Lichrospher RP 18	Aq 50% methanol	NR	540 nm; 25–50 ppb for nitrate esters, 30–100 ppb for nitramines	118
106	TNT, RDX, HMX	Water	(i) Salting -out solvent extraction (ii)SPE (iii) membrane SPE	For (i) NaCl ; acetonitrile For (ii) Porapak P cartridge at 10 mL/min For(iii) Empore styrene –divinyl at 70–100 mL/min	NR	LC 18 (25 cm × 4.6 mm i.d.; 5 μm)	Aq 50% methanol	1.5 mL/min	0.05–0.30 μg/L	119
107	Explosive				UV	YQG-C 17H 38 (15 cm × 4 mm i.d.; 5 μm)	100%, 95%, 90%, 85%, 80% and 75% of methanol	0.5 mL/min	230 nm	120
108	Organic nitrate, nitramine and nitro toluene explosive	Post blast debris	Solvent extraction	Methanol	MS				50–500 pg	121

109	Tetryl and its metabolite	Plant	Solvent extraction	Dichloromethane	UV	(24 cm × 4.6 mm i.d.; 5 μm) of ODS	Gradient elution of aq 35% to 100% acetonitrile over 30 min	NR	264 nm	122
110	TNT, 2.4-DNT, Glyceryl trinitrate	Explosives	Solvent extraction	Chloroform	UV	LiChrosorb Si 60 column (15 cm × 4 mm i.d.; 5 μm)	Hexane/ propan-2-ol (19:1)	—	230 nm	123
111	NG, EGDN, DNT, PETN, RDX, HMX, TNT, Tetryl	Explosive mixture	NA	NA	Derivatization with azo dye	Nova pak RP C18 column or Merck select B RP8	Aq 50% methanol (w/w) or acetonitrile:water (35/65, w/w)	0.7 mL/min	540 nm, 100 pg	124
112	Nitramine, nitro and nitra explosives,nitro pnenols	Waste water	SPE/ LLE	In SPE: water adjusted to pH 9 with 0.02M NaOH and treated with NaCl and cartridge pack with Amchro RP 18 In continuous LLE: DCM/ethyl ether in (23 hrs) In discontinuous LLE: with toluene or DCM at pH 9	UV	For nitramine/nitro and nitra amino toluene explosives: Spherisorb C18 -2 column (25 cm × 4 mm i.d.) For nitrophenol: LiChrosorb RP 18	Aq. methanol Gradient elution with methanol containing 0.1% acetic acid			125
113	Tetryl and its transformation products	Soil	Soxhlet extraction	Methanol	UV	Beckman Ultrasphere ODS column (24 cm × 4.6 mm i.d.; 5 μm)	Gradient elution with acetonitrile/ water over 30 min	NR	265 nm	126
114	Explosives	Soil	Ultrasonic extraction	Acetonitrile	UV	For qualitative measurement: Microsorb C18 (25 cm × 4.4 mm; 5 μm) in series with a column of supelcosil LC-PAH (25 cm × 4.6 mm) For quantitative measurement: Supelco LC 8 (3 μm)	Gradient elution with methanol/ water 70.7% water, 27.8% methanol and 1.5% methanol	2.0 mL/min	244 nm 244 nm	127
115	HMX, RDX, TNT 1,3,5-TNB, 1,3-DNB, 2,4-DNT, Tetryl	Soil	Ultraso-nic extraction	Acetonitrile	UV				254 nm, 1 μg/g to 1000 μg/g	128

(Continued on next page)

TABLE 2
Characteristics of the HPLC methods for the analysis of explosives

Sr.No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
116	TNT, RDX and related compounds and degradation products	Soil	Ultrasonic extraction	Acetonitrile	UV	LC-18 (25 cm × 4.6 mm i.d.; 5 μm) and confirmation on simallar column of LC-CN	Aq 50% methanol	1.5 mL/min	254 nm 0.03-1.27 μg/g	129
117	Nitro aromatic explosives including NT, NB, Nitro phenol, nitro aniline derivatives	Water	Column concentration	Amberlite –XAD 2/4/8 (1:1:1)	PDAD	(25 cm × 4 mm) packed with LiChrosorb RP 18 or LiChrospher RP 18 (5 μm)	Methanol/ water (9:11)	0.8 mL/min	General 50 ng/L and 0.1–20 μg/L for mono, di and trinitro toluene and nitrotoludines	130
118	Nitro glycerine, centralite	Double base powder	—	—	PDAD	Zorbax ODS column (25 cm × 4.6 mm)	70% aq. methanol	0.8 mL/min	254 nm	131
119	TNT, TNB, RDX, HMX	Soil	Soxhle/ ultrason-ic bath/ Mechanical shaker/ homogenizer-sonicator	Acetonitrile or methanol	UV	For separations: Supelco 25 cm × 4.6 mm; 5 μm LC-8 column For confirmation: (i) Supelco 25 cm × 4.6 mm LC-CN column and Supelco 25 cm × 4.6 mm LC-18 column	Water/methanol/ acetonitrile (50:38:12) Water/Methanol (1:1)	1.5 mL/min	254 nm	132
120	RDX	Blood serum, urine	Column concentration	Bond Elut C18 column	UV	Bondpak C18 (30 cm × 3.9 mm; 10 μm)	Aq. 36% methanol	1.8 mL/min	240 nm 0.1 mg/L	133
121	Diphenyl amine	Cloth-ing debris and hand swabs extracts	Solvent extraction/ column concentration	Acetonitrile; column (3 cm × 0.8 mm) of Amberlyst	Oxidative/ reductive ECD	NR	NR	NR	10 pg for oxidative detection and 1000 pg for reductive detection	134
122	Metabolite of TNT	Urine and blood serum	Solvent extraction	Toluene and methylene chloride	UV/MS	For Toluene extracts of human and rat urine : Microcolumn (10 cm × 2.1 mm; 5 μm) containing LiChrosorb RP 8 For methylene chloride extracts: (10 cm × 4.6 mm; 5 μm) containing LiChrosorb RP 8	Aq. acetonitrile or methanol/ acetonitrile/water in various proportion Aq. acetonitrile	120–130 μL/min 1.0 mL/min	214 nm 0.1 ng/mL and MS detection 214 nm and MS detection	135

123	Diphenyl amine, centralite	Explosive residue	—	—	UV/ECD	C18 column (10 μm)	Water/ 85% phosphoric acid/acetonitrile (100:3:97)	—	Picogram level	136
124	TNT, RDX, HMX, PETN	Explosive mixture	NA	NA	UV	(25 cm × 0.5 cm O.D.) column at 30°C	Aq. methanol	1.0 mL/min	254 nm	137
125	Glyceryl 1,2 and 1,3-dinitrate	Cotton wool and hand swab	Solvent extraction/ Column concentration	Methanol/ (3 cm × 0.6 mm) of charcoal	HMDE	(15 cm × 4.5 mm; 3 μm) ODS – Hypersil At 40°C	Deoxygenated methanol/ aq 0.035 M phosphate of pH 3 (20:17)	—	0.9 ng for 1,2-dinitrate and 1.5 ng for 1,3-dinitrate	138
126	Nitro aromatic explosives	NA	NA	UV	UV	(15 cm × 4 mm) column of 3-(10-methyl-9-anthryl) propylsilane as stationary phase	Aq 80% methanol	1.0 mL/min	254 nm	139
127	TNT and its metabolite	Urine	Solvent extraction	Toluene	UV-MS	(10 cm × 2.1 mm) of Brownlee RP 8	Aq 37% acetonitrile or methanol/acetonitrile/ water (10:9:31)	120 μL/min	214 nm 0.1 ng/mL	140
128	TNT, Tetryl, RDX, HMX, PETN, DNT Nitroglycerine, picric acid, ammonium nitrate	Hand swab	Wiping	Acetone	MS	(25 cm × 4.6 mm I.D.) zorbax C_8	Aq 25% to 75% methanol containing 0.1 M ammonium acetate	1.4 mL/min	200 pg for TNT and 5 ng for ammonium pictate	141
129	TNT and its metabolite	Blood	Solvent extraction	Dichloro-methane	UV/MS	(10 cm × 4.6 or 2 mm) of Brownlee RP 8 (5 μm)	Aq 25% or 40% acetonitrile	1.0 mL/min	214 nm	142
130	Nitro based high explosive	Post blast debris	—	—	Photolysis-ECD	Radial Pak C18 cartridge (10 cm × 5 mm; 5 μm)	Aq 35% to 60% methanol containing 0.2M NaCl; isocratic		120 to 250 pg for aromatic nitrocompounds, nitramines and nitrate ester	143
131	Explosive	Water	Solid sorption	Porapk resin and Amberlite resin	ECD	—	—	—	1.0 μg/L	144
132	RDX, HMX and their acetyl derivatives	Water	NA	NA	UV	(25 cm × 4.6 mm i.d.) of zorbax C_8 (6 μm)	Gradients of Aq. 20% methanol (A) and Aq. 80% methanol(B)	1.2 mL/min	254 nm	145
133	Nitroglycerine and other Explosives	Explosive mixture, clothing extracts and explosion debris	Column conc./ solvent extraction for NG	(i) Porapak T reusable column (2.2 cm × 1 mm) (ii) Disposable column (5 cm × 1mm) of eg. Porapak T, chromosorb 104 and charcoal (iii) Acetonitrile/water (100:5;v/v) for solvent extraction	PMDE	(15 cm × 4.5 mm) of ODS –hypersil (3 μm) at 40°C	Deoxygenated methanol-0.035M (pH 3)	1.0 mL/min	Pendent mercury drop electrode (3mg) maintained at a potential of 0.9 V vs. Ag/AgCl.	146

(Continued on next page)

TABLE 2
Characteristics of the HPLC methods for the analysis of explosives *(Continued)*

Sr. No.	Analyte	Matrix	Preconc. Tech.	Fibre/cart/ condition	Detector	Column/ Temp	Mobile Phase	Rate of Flow	Wavelength/ LOD	Ref
134	TNT and its metabolite	Biological fluid	NR	NR	UV/MS	RP-8 column	Acetonitrile-water mixtures at various relative concentrations	1.0 mL/min	214 nm; LOD with MS: 100 ng/ μL to 1 μg/ μL LOD with UV: 1 to 10 ng/ μL	147
135	Nitramine, NT, nitrate ester explosives	Water	(i) Solvent extraction (ii) Adsorption	(i) Dichloromethane (ii) Porapak resin column for adsorption	UV/ECD	(i) For UV: Zorbax ODS reverse phase column (25 cm $\times$ 4.6mm i.d.,7 μm) (ii) For ECD: Spherisorb ODS column (25 cm $\times$ 4.6 mm i.d.,5 μm)	(i) Aq. methanol 1-propanol- 0.025 M sodium acetate, 0.025 M monochloroacetic acid (30:70, v/v)	1.5 mL/min 2.0 mL/min	210 & 254 nm 1 g/L	148
136	HMX, RDX, TNT 2, 6&2, 4-DNT, Tetryl	Soil	Solvent extraction	Acetonitrile	UV	10 m C18 Radial Pack cartridge	Aq 40% methanol	2.0 mL/min	254 nm; 0.45 ppm for HMX and 4.59 ppm for tetryl	149
137	TNT, DNT, Tetryl, NG, ISDN and other nitro compounds	Expl-osives Mixture	NA	NA	Photolysis-ECD	Biophase C_{18}, 25 cm $\times$ 4.6-mm i.d.; 10 μm, Perkin-Elmer Fast-LC C_{18}, (10 cm $\times$ 4.6-mm i.d.; 3 μm, Waters) Bondapak C_{18}, (25 cm $\times$ 4.6-mm i.d.; 10 μm) and various in-house slurry packed C_8 or C_{18} reversed phase columns.	50/50 MeOH/0.1 M NaCl	0. 6 mL/min or 1.4 mL/min	25 ppb for RDX, Tetryl and TNT, 200 ppb for NG and 125 ppb for ISDN	150
138	Ethanediol mono nitrate (I) and monomethy-lamine nitrate (II)	Post blast debris	For (I): adsorption For (II): Solvent extraction	(I) Charcoal (II) heptane	UV UV	μ Bondapak C18 column and μ Bondapak CN column	Aq 50% acetonitrile/ Chloroform (7:3), Hexane/ propan-2-ol (9:1) or gradients of aq 49% to 70% acetonitrile		200, 214 or 354 nm	151
139	NG, DEGN, TNT, Tetryl, 2,4-DNT, RDX, PETN, Ammonium niotrate	Explosives mixture	NA	NA	UV/MS	(10 cm $\times$ 4.6 mm i.d.) containing LiChrosorb RP 8 (10 μm)	Aq 50% methanol or aq 50% acetonitrile	1.0 mL/min	214 nm	152

140	NG, TNT, PETN, RDX, HMX, Tetryl	Expl-osive de-bris,explosive residue	Solvent extraction	Acetonitrile	UV	RP cartridge in a radial –compression module	Aq. 70% acetonitrile	1.0 mL/min	214 nm 0.01 μg	153
141	HMX, TAX, RDX, TNT *etc*.	Waste water	SPE	Sep-pak C_{18} cartridge	UV	C18 radial compression column (10 cm × 8 mm; 10 μm)	By gradient of aq 25% methanol-aq 80% methanol from (19:1) to (1:1) during 30 min	—	240 nm 100 ng for each analyte and 0.2 μg/mL in the sample	154
142	Explosives	Post explosion residue	—	—	—	Nucleosil 10 C_{18}	30% methanol	—	—	155
143	PETN, EGDN, RDX, HMX, Picric acid, NG, Tetryl, TNT, NB, NG, DNT, 2-NT, 3-NT, 4-NT, DNB	Explosives Mixture	NA	NA	ECD	ODS-Hypersil (15 cm × 4.5 mm; 3 μm)	Methanol/ Aq. pottasium phosphate (100:86,v/v) [0.025 M, pH 3.0]	1.0 mL/min	7–49 pg per 20 μL	156
144	PETN, EGDN, RDX, HMX, Picric acid, NG, Tetryl, TNT, NB, NG, DNT, 2-NT, 3-NT, 4-NT, DNB	Handswab extracts	Microfilter extraction	Alumina and ODS	ECD	ODS-Hypersil (15 cm × 4.5 mm; 3 μm)	Methanol/ Aq. pottasium phosphate (100:86,v/v) [0.025 M, pH 3.0]	1.0 mL/min	−1.0 V v/s Ag/AgCl	157
145	Polynitro explosives	Explosives	NA	NA	UV	C18 Raqdial Pak A cartridge	40, 50 or 70% methanol	2.0 mL/min	254 nm 0.2 μg/mL to 3.332 mg/mL	162
146	Nitro aromatic, nitramine, and nitrate ester explosive	Explosives mix-ture and gun shot residues	NA	NA	UV/Reductive ECD	LC column (25 cm × 0.46 cm; 5 μm) with C_{18} and C_8 biophase	For UV: 0.02 M monochloroacetic acid, 0.0145 M sodium acetate, 0.001 M EDTA, 5% (v/v) ethanol. 17% (v/v) 1-propanol, at pH 3.5,	1.7 mL/min	254 nm for UV, 0.5, 1, 2, and 0.3 picomol for nitro aromatic, nitratmine and nitrate ester explosive compounds, and diphenylamines, respectively.	163
147	RDX, PETN, HMX, NG, EGDN, PEDN, NGu, petrin	Explosives mixture	NA	NA	TEA ana-lyzer	(25 cm × 3.2 mm i.d.) one packed with LiChrosorb Si-60 (10 μm) other packed with LiChrosorb NH_2 packing (10 μm)	Iso-octane/ethanol	1.5 mL/min	1.00 ng level except NG which required 20 ng level	166

NA = Not Applicable.
NR = Not Reported.

initially separated and subsequently decomposed photochemically to hydrogen peroxide, which is finally determined by electrochemical detection. This detection scheme was significantly easier to use than other methods incorporating photochemical decomposition, enzymatic post-column reaction and fluorescence detection for analysis of peroxide-based explosives.

Hilmi et al. (77) analyzed explosives including TNT, HMX and RDX In soil and ground water by liquid chromatography-UV and amperometric detection. Spiegel et al. (94) monitor the degradation process of explosives by HPLC analysis and subsequent UV and amperometric detection. Lewin et al. (108) employed high-performance liquid-chromatographic with UV-electrochemical detection for residues of explosives in water samples around a former ammunition plant. Lloyd et al. (134) analyzed diphenylamine traces in handswabs and clothing debris. Dahl et al. (136) determined black and smokeless powder residues in firearms and improvised explosive devices. The method was used to distinguish between black and smokeless gunpowders with HPLC with electrochemical detection. The procedures were reported to detect black or smokeless gunpowder in explosive residues arising from gunpowder bomb explosions or black gunpowder firearm discharges also.

Lloyd et al. (138) analyzed the glyceryl dinitrates in the detection of skin contact with explosives and related materials of forensic science interest with hanging mercury drop electrode (HMDE). Selavka et al. (143) employed liquid chromatography with photolysis-electrochemical detection of nitro-based high explosives and water gel formulation sensitizers. The reported reactor geometry affords detection limits for aromatic C-nitro-compounds, nitramines and nitrate esters, appreciably better than with conventional tubular reactors. Maskarinec et al. (144) analyzed the samples of environmental water which were applied to Porapak resin and Amberlite resin. The explosive compounds were desorbed into acetone and a portion was analyzed by HPLC with electrochemical detection with high selectivity and sensitivity. Lloyd et al. (146) analyzed the explosive during the micro-column cleanup and recovery techniques for organic explosives compounds and for propellants traces in firearms discharge residues with hanging mercury drop electrodes. Maskarinec et al. (148) determined the munitions components in water by electrochemical detection. Krull et al. (150) analyzed nitro explosives and related compounds via high-performance liquid chromatography-photolysis-electrochemical detection. The reported methods were applied in the analysis of standard explosives by using single- or dual-electrode detection. The responses of the dual-electrode detector were found to be a function of applied working potentials. The dual-electrode technique, which showed greater sensitivity than the single-electrode method was applicable in the analysis of explosives, drugs, nitrate ester compounds, nitro-aromatics, nitro-PAH and similar such derivatives. Lloyd et al. (156) analyzed organic explosives components with electrochemical detection at a pendent-mercury-drop electrode. Lloyd et al. (157) analyzed the clean-up procedures for examination of swabs for explosives traces by high-performance liquid chromatography with electrochemical detection of a pendent-mercury-drop electrode. Bratin et al. (163) determined the nitro-aromatic, nitramine and nitrate ester explosive compounds in explosive mixtures and gunshot residue by liquid chromatography and reductive electrochemical detection.

HPLC–NMR Methods

The combination of chromatographic separation techniques with NMR spectroscopy is one of the most powerful and time-saving methods for the separation and structural elucidation of unknown compounds and mixtures. The technique of HPLC-^{1}H-NMR complements conventional methods, such as GC-, HPLC/MS and HPLC/UV, in the analysis of environmental samples. Its comparatively low sensitivity is usually more than compensated by its advantages. It makes the differentiation of isomers quite easy and even allows the elucidation of the structure of unknowns, e.g., degradation products of pollutants. The concentrations of structurally known components can be quantified without previous calibration runs because of the exactly known relative response factors (number of protons in the analyte/number of protons in the standard) relative to an internal standard. This also implies that there is no need to provide reference samples for all compounds of interest. The content of spectral information of NMR-chromatograms makes the NMR spectrometer a highly specific yet universal detector for proton containing analytes.

Strynar et al. (45) analyzed the soil without a history of exposure to explosives incubated with ^{15}N-labeled 2,4,6-trinitrotoluene (TNT) and ^{14}C-TNT and then amended with compost at a 1:2 soil to compost ratio. The mixture of soil and compost was inoculated with methanogens from cattle manure, amended with glucose and starch and incubated under anaerobic conditions. The anaerobic incubation was followed by forced aerobic incubation. At the end of the aerobic phase no TNT was present in the extracts as determined by high-performance liquid chromatography. Radioactive materials associated with humic acid and humin were analyzed by solid-state ^{15}N-nuclear magnetic resonance (NMR) spectrometry. Godejohann et al. (91) analyzed organic explosives compounds in mixtures by HPLC-NMR. Godejohann et al. (93) analyzed the ground water near former ammunition plants for the detection of nitroaromatic high explosives. Compared with HPLC with photodiode-array detection, the method was able to identify more compounds derived from explosives. Preiss et al. (102) analyzed the high explosives, e.g., RDX, TNT with HPLC during comparison of high-field proton nuclear magnetic resonance spectroscopy for the analysis of explosives and related compounds in groundwater samples to the high-performance liquid chromatography (HPLC) method. Despite relatively low sensitivity, proton NMR was reported to be useful because of its high selectivity (Figure 8).

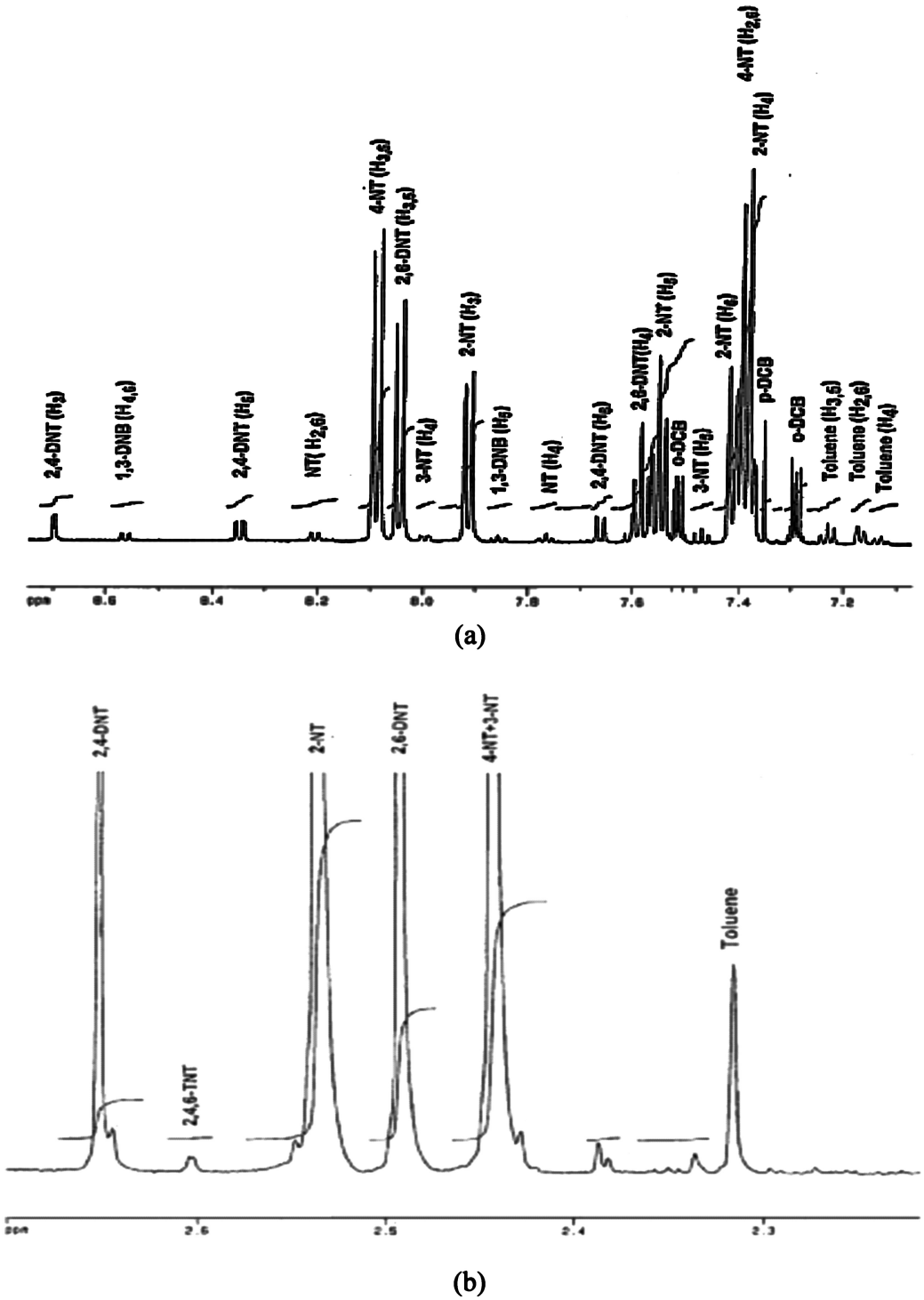

FIG. 8. 600 MHz 1H-NMR partial spectra of the sample from the lower level: (a) shift region of the aromatic protons (7.80–8.74 ppm); (b) shift region of the methyl protons (2.20–2.70 ppm). (With permission from (102).)

HPLC-Post Column Derivatization Methods

In most cases the separated sample material is passed on-line to a UV detector where UV light is absorbed by a chromaphore and displayed as a peak on a recording device or computer screen. Other detectors such as fluorescence and refractive index have been used instead of or in tandem with UV detectors to view sample components that may not have a chromaphore to absorb UV light. In some cases UV-absorbing derivatives

are prepared prior to introduction to the column. However, if the UV moiety interferes with the separation, the pre-column derivatization is impractical. Instead the separation must be performed before UV-absorbing derivatives are formed. In such instances, post-column derivatization is a viable option. To perform post-column derivatization, the HPLC must be modified with the addition of a secondary fluid delivery system. Typically this consists of a pump, tubing and fittings and a reaction coil. Derivatizing agent is introduced between the column and detector. So the derivatization process is carried out "on-the-fly," i.e., during transfer of the sample components from the column to the detector. The post-column reaction system mixes the stream of eluant from the HPLC column with a stream of reagent solution. The mixture usually flows through a reactor to allow enough time for the chemical reactions to complete. If the reaction is slow, the reactor may be heated to speed things up. Some reactions need two or more reagents added in sequence. Finally the mixed streams pass into the detector, typically UV/VS absorbance or fluorescence. Of course a practical system requires metering pumps, pulse-dampeners, thermostats, and safety systems to give reliable results. The chemical requirements for post-column derivatization are generic, i.e., Stability of reagent, completeness of reaction, reproducibility, minimal detector response of reagents, solubility. Thus HPLC-post-column derivatization proves to be of great importance in routine analysis.

Schulte-Ladbeck et al. (38) determined the peroxide-based explosives using postcolumn derivatization and UV irradiation with the help of fluorescence detector.

Woltman et al. (66) analyzed nitroaromatic and nitramine compounds by electrochemical reduction combined with photoluminescence following electron transfer. The oxidizing agents were reported to use as a postcolumn reagent for the detection of nitroaromatic and nitramine explosive compounds. After separation, the explosives are reduced electrochemically to oxidizable products and these products react readily with oxidizing agents. The photoluminescence from the latter is used for detection. Kolla et al. (118) adapted dramatization and TEA chemiluminescence to the trace analysis of explosives. Preferred detection was reported by post-column derivatization with sulphanilamide and naphthylethylenediammonium chloride under mercury-lamp irradiation and absorbance measurement. Engelhardt et al. (124) analyzed the nitroexplosives with subsequent post-column derivatization with azo dye.

HPLC-TEA Methods

Lafleur et al. (166) analyzed the explosives at trace levels by high-performance liquid chromatography with a nitroso-specific detector (TEA Analyzer). This method was reported for the identification and determination of explosives and other related compounds possessing thermally labile nitro or nitroxy groups. Use of retention times on the two different columns coupled with a response on the nitrosyl-specific detector serves as a convenient method for identification.

Similarly, Albanbauer et al. (155) analyzed the smoke residues and splinters from brisant explosive detonations used to identify the source and composition of the material. Traces of powder were collected by wiping. The samples were fractionated by HPLC. Neumann et al. (158) used of high-performance liquid chromatography for forensic analysis of explosives glyceryl trinitrate, hexahydro-1,3,5-trinitro-1,3,5-triazine, pentaerythritol tetranitrate in gunpowder and/or explosive mixtures etc. Anspach et al. (160) used solid sorbents for sampling and subsequent hplc analysis of explosives from water. Richardson et al. (161) determined diphenylamine and various nitrated diphenylamines by reversed-phase high-performance liquid chromatography in samples containing exposives. Krull et al. (164,165] trace analyzed the explosives by HPLC - electron-capture detection and in explosive mixture respectively. Prime et al. (167) analyzed the recovery and identification of ethyleneglycol [ethanediol] dinitrate and nitroglycerin in explosion debris using pre-concentration and high-performance liquid chromatography. Sjobom et al. (168) analyzed separation and quantization of nitrocellulose, nitroglycerin, diethyl phthalate and centralite in double-base powder.

CONCLUSION

Thus we can conclude that HPLC is a powerful analytical tool for the analysis of the explosives. Explosives are present in wide complex matrices at the training and testing sites The detection of explosives in different environmental samples such as surface and subsurface soil and water, plant and animal tissues, etc., showed that contamination by explosives is widespread and can reach the water table and also accumulate in plants. The application of the preconcentration methods in combination with HPLC is very useful for their detection at very low concentration level in sub ppb range. It is found that SPME has an over edge in the preconcentration methods over the normal SPE methods. Though, recently in literature, advanced mass spectrometric detection modes are reported for the analysis of the explosives but HPLC-UV system is still promising and offers many advantages over the other systems.

ACKNOWLEDGMENT

The authors are thankful to the DRDO, New Delhi for grant supporting this research work.

ABBREVIATIONS

ADNT	Aminodinitrotoluene
APCI	Atmospheric pressure chemical ionization
ASE	Accelerated solvent extraction
BTDN	Butanetrioldinitrate
BTTN	Butanetrioltrinitrate
CW/TPR	Carbowax/templated resin
DAT	Diaminotoluene
DNA	Dinitroaniline
DNB	Dinitrobenzene

DNBA	Dinitrobenzoic acid
DNBSA	Dinitrobenzenesulphonic acid
DNG	Dinitrglycerine
DNP	Dinitrophenol
DNT	Dinitrotoluene
DNX	Hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine
DPA	Diphenylamine
ECD	Electrochemical detection
EGDN	Ethyleneglycoldinitrate
ESI	Electron spray ionization
FEL	Forensic explosive laboratory
GC	Gas chromatography
HMDE	Hanging mercury drop electrode
HMTD	Hexamethylenetriperoxidediamine
HNS	Hexanitrostilbene
ISDN	Isopsorbidedinitrate
LLE	Liquid liquid extraction
LSC	Liquid scintillation counter
MAE	Microwave assisted extraction
MNG	Mononitroglycerine
MNX	Hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine
MS	Mass spectrometry
NB	Nitrobenzene
NBA	Nitrobenzoic acid
NG	Nitroglycerin
NGu	Nitroguanidine
NMR	Nuclear magnetic resonance
NP	Nitrophenol
NQ	Nitroguanidine
NT	Nitrotoluene
ODS	Octadecylsilane
PA	Polyacrylate
PAED	Photo assisted electrochemical detector
PDAD	Photo diode array detector
PDMS/DVB	Polydimethylsiloxane/divinylbenzene
PETN	Pentaerythritol tetranitrate
PFE	Pressurized fluid extraction
PFET	Photoluminescence following electron transfer
PGC	Porous graphite carbon
PLE	Pressurized liquid extraction
PTFE	Polytetrafluoroethylene
SDS	Sodium dodecyl sulphate
SEX	l-acetyloctahydro-3,5,7-trinitro-1,3,5,7-tetrazocine
SFE	Supercritical fluid extraction
SPE	Solid-phase extraction
SPME	Solid-phase micro extraction
SRS	Scientific research service
TATP	Triacetonetriperoxide
TAX	1-acetylhexahydro-3,5-dinitro-1,3,5-triazine
TEA	Thermal energy analyzer
TENAC	Tetranitroacridone
TNB	Trinitrobenzene
TNT	Trinitrotoluene;
TNTAB	Triazido trinitrobenzene
TNX	Hexahydro-1,3,5-trinitroso-1,3,5-triazine
UV	Ultraviolet

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