Analysis of CL-20 in Environmental Matrices: Water and Soil

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Abstract

Analytical techniques for the detection of 2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazatetracyclo(5.5.0.05,9.03,11)dodecane (CL-20) in water and soil are developed by adapting methods traditionally used for the analysis of nitroaromatics. CL-20 (a new explosives compound) is thermally labile, exhibits high polarity, and has low solubility in water. These constraints make the use of specialized sample handling, preparation, extraction, and analysis necessary. The ability to determine the concentrations of this new explosive compound in environmental matrices is helpful in understanding the environmental fate and effects of CL-20; understanding the physical, chemical, and biological fate of CL-20; and can be used in developing remediation technologies and determining their efficiency. The toxicity and mobility of new explosives in soil and groundwater are also of interest, and analytical techniques for quantitating CL-20 and its degradation products in soil and natural waters make these investigations possible.

Introduction

The environmental consequences of the industrial production of such well-known energetic materials as hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX), and 2,4,6-trinitrotoluene (TNT) are well-documented. The recently synthesized compound hexanitrohexaazaisowurtzitane (HNIW) and 2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazatetracyclo(5.5.0.05,9.03,11)dodecane (also known as CL-20) is the subject of numerous advanced research and development studies seeking an alternative to currently used explosives (1,2). Several synthesis routes for CL-20 have been developed (1,3–7). The structures of CL-20, TNT, and RDX are illustrated in Figure 1. CL-20 is referred to as a caged compound because it resembles two RDX rings joined at several carbon atoms.

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CL-20 has been investigated in terms of its crystal and molecular structure (1,2,8). CL-20 exists in six different polymorphs (labeled α to ζ), and phase transitions occur easily. The phase transitions may affect reactivity and may be influenced by temperature, organic solvents, and chemical impurities (8–11).

Several studies have been carried out to evaluate the potential of CL-20 for military applications, which include the determination of explosive performance, sensitivity, and response to one-dimensional shock loading (9,11–13). CL-20 compounds exhibit higher energy and density than HMX or RDX, which are monocyclic nitramines (8). Propellants and explosive formulations using CL-20 are expected to have better performance in terms of specific impulse, bum rate, ballistics, and detonation velocity because of this higher energy and density. In addition to improved performance, CL-20 meets stringent munitions sensitivity requirements. Because CL-20 contains no halogens, its combustion products are more environmentally acceptable than those derived from the combustion of propellants made with ammonium perchlorate.

The widespread, high-level interest in CL-20 has resulted in an

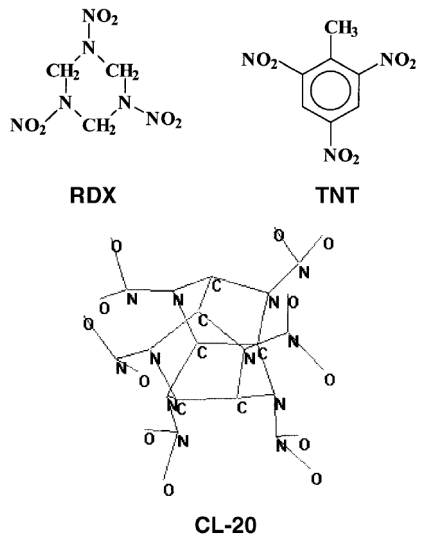

Figure 1. Molecular structures of CL-20, TNT, and RDX.

increase in its industrial production up to several thousand pounds per year. To date there have been no studies on the environmental impact of CL-20 and its degradation products. Concerns regarding the environmental fate of CL-20 are arising because of the potential for deposition within soil or water systems resulting from CL-20 manufacturing and the loading and use of munitions containing CL-20. Before full-scale production begins, however, a thorough investigation of CL-20's environmental fate, transport, and effect along with remedial alternatives for the cleanup of CL-20 in soils and waters is warranted. The ability to monitor CL-20 at levels of environmental interest in soils and waters is a necessary tool for the performance of such studies.

High-performance liquid chromatography (HPLC) methodology was used to investigate and develop sample preparation and techniques for the analysis of CL-20 in various matrices. The results of this study provide analytical methods for the determination of CL-20 and its degradation products that will be needed for future environmental research.

Experimental

The methods used to detect CL-20 in a variety of environmental samples require matrix-specific sample preparation, separation by reversed-phase HPLC, and ultraviolet detection. The analytical systems described in this report were identical to those that are required to perform U.S. EPA SW-846 Method 8330 for the determination of explosives in waters and soils (14). Samples resulting from research projects are often limited in mass and volume. The method developed for the analysis of CL-20 in these matrices was designed to reduce the amount of material that a laboratory would have to acquire in order to analyze for CL-20. The instrumental methods will be outlined.

Analytical system

The equipment used in the sample preparation included a centrifuge and centrifuge tubes (3000 rpm), syringes and filters, volumetric flasks of various sizes, automatic pipettes, and autosampler vials.

The HPLC system consisted of a Waters (Milford, MA) 610 fluid unit pump capable of achieving 6000 psi, a Waters 717 plus autosampler including a 200-µL loop injector, a Waters 486 tunable UV absorbance detector monitored at 245 nm, a Waters 410 diode-array UV absorbance detector, and Millennium 2.1 chromatography software (Waters). A Supelco (Bellefonte, PA) LC-18 reversed-phase HPLC column (25 cm × 4.6 mm, 5 µm i.d.) (catalog #5-8298) was used as the primary column, and a Supelco LC-CN reversed-phase HPLC column (25 cm × 4.6 mm, 5 µm i.d.) (catalog #5-8231) was used as a confirmation column. The use of both the C-18 and CN columns allowed this system to produce interference-free determinations. The appropriate precolumn, Novapak C-18 catalog #WAT015220 or Novapak CN (catalog #WAT020800) (Waters), was used.

Sonication extractions were performed using a temperature-controlled ultrasonic bath (the temperature did not exceed 30°C). The filtration system used for sample preparation consisted of a disposable LurLoc syringe and disposable 0.50-µm Teflon filter cartridges. The solid-phase cartridges used for sample concentration were Waters SepPak Vac cc (500 mg) Porapak RDX cartridges (catalog #WAT047220).

Reagent-grade inorganic chemicals were used in all tests. Unless otherwise indicated, all reagents conformed to the specifications of the Committee on Analytical Reagents of the American Chemical Society. The solvents used in this method were acetonitrile (CH_3CN, HPLC grade) and methanol (CH_3OH, HPLC grade). Calcium chloride ($CaCl_2$) was used as an aqueous solution at 5 g/L. The water used was organic-free reagent water (18 mΩ, Milli-Q). The HPLC mobile phase (1:1, v/v, methanol–reagent water) was prepared by measuring 500 mL of each and combining them prior to filtration. A vacuum filtration system from Millipore (Bedford, MA) with 0.22-µm filters was used for degassing the mobile phase and removing particulate matter.

Matrix

This method can be used for the determination of CL-20 in a broad range of matrices. It easily accommodates the measurement of CL-20 in water samples ranging from distilled water and seawater to heavily contaminated wastewaters. The mass quantitation of extractable CL-20 in soil is also possible. In order to obtain the method detection limits (MDLs) that are attainable for other explosives compounds, samples were prepared using solid-phase extraction (SPE) to provide a concentrated extract for HPLC analysis. It is also possible to attain MDLs of extractable CL-20 from soil that are analogous to the current U.S. EPA SW-846 Method 8330 MDLs for common explosives-based compounds.

Sample preparation

High concentrations in water

Water samples were prepared for the analysis of CL-20 in water without a concentrative sample preparation step by adding 5 mL acetonitrile to 5 mL of sample. Method blanks were generated by adding 5 mL acetonitrile to 5 mL of Milli-Q water, and laboratory control samples were prepared by spiking 5 mL Milli-Q water with 5 mL acetonitrile. The samples were then filtered using a 0.45-µm Millipore Millex-SR Teflon filter and placed in an autosampler vial for analysis. All samples were refrigerated at 4°C.

Low concentrations in water

For the analysis of CL-20 in water at lower levels with a concentrative sample preparation step, water samples were extracted by an SPE procedure using a vacuum manifold and solid-phase cartridges (Waters SepPak Vac cc (500 mg) Porapak RDX). The cartridges were conditioned with 10 mL of acetonitrile followed by 15 mL of Milli-Q water. After conditioning, 500 mL of the sample was passed through the cartridge until no sample was visible in the cartridge (to dryness). The samples were then eluted off the cartridges using 5 mL of acetonitrile and were collected in centrifuge tubes. Method blanks were prepared by passing 500 mL of Milli-Q water through the cartridge, and laboratory control samples were prepared by passing 500 mL of spiked Milli-Q water through the cartridge. The concentrated extract was diluted to 1:1 (v/v) with reagent-grade water.

Soils

Soil samples were prepared for analysis by the following procedure. The soil sample was thoroughly mixed to achieve maximum homogeneity prior to subsampling. Approximately 5.0 ± 0.5 g of wet sample was weighed into a 20-mL glass vial with a Teflon-lined cap and the weight was recorded. Samples and associated quality-control samples were spiked with surrogate and matrix spiking solutions. Acetonitrile (10 mL) was added, and using a vortex mixer samples were swirled for 1 min and then placed in a cooled ultrasonic bath for 18 h. After sonication, samples were allowed to settle for 30 min. Representative aliquots (5 mL) of supernatant were removed using 5-mL pipettes with disposable tips and placed into 20-mL vials. Portions (5 mL) of a calcium chloride solution (5% by weight) were added to the 5-mL samples of supernatant. The resulting samples were then filtered and analyzed by HPLC.

In order to determine the percentage solids of the original soil samples, representative samples (2–4 g) of the wet material were placed in disposable weigh dishes and the weights were recorded. Samples were dried at 104–105°C until they maintained a constant weight. The sample weight was recorded after the samples cooled to room temperature.

Concentration ranges

The tested concentration range depended on the matrix in which CL-20 and its degradation products were being measured. Standards that were dissolved in organic solvents and injected directly into the HPLC could be tested in the concentration range of 0.04 to 4.0 µg/mL. Natural waters spiked with standards could be tested in the concentration range of 0.1 to 20 µg/mL using the high-level method and 0.5 to 200 ng/mL using the low-level method. Clean soils spiked with standards could be tested in the concentration range of 0.10 to 20 µg/g. The testable concentration range will vary considerably depending on the matrix encountered. Samples that contain high concentrations of other contaminants may have much higher background levels and detection limits may be considerably higher.

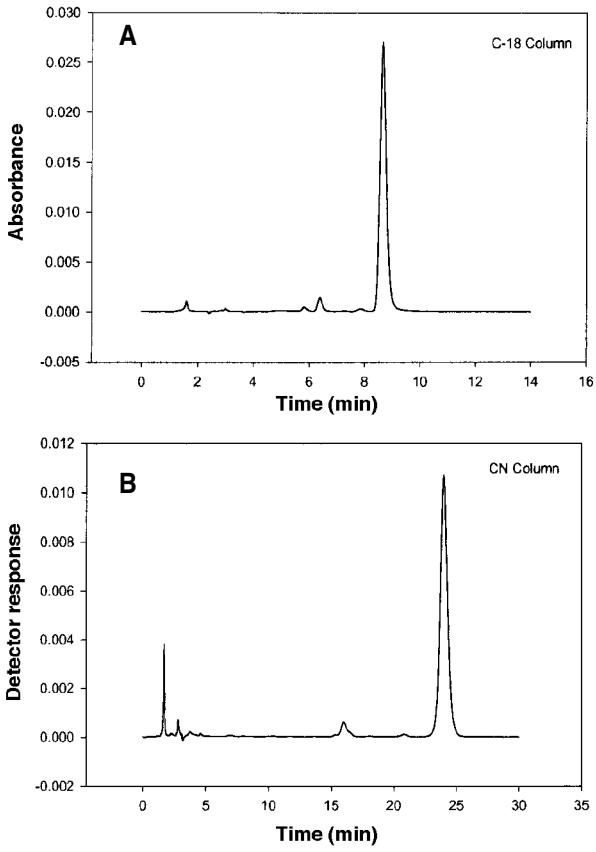

Figure 2. Chromatograms for CL-20 using the (A) C-18 and (B) CN columns.

Interferences

There is always a possibility that an extract may contain a compound that absorbs UV light at the wavelength used for CL-20 detection and thus would elute from the analytical column at a similar time as CL-20. However, the use of both C-18 and CN columns (which have dissimilar retention characteristics) allowed this chromatographic system to significantly reduce the frequency of interferences on both columns. A comparison of the signal amplitude for peaks with the appropriate retention times for CL-20 served to identify interfering compounds in specific extracts.

Safety

Many nitramine and nitroaromatic explosives (including CL-20) are suspected carcinogens. Some degradation products of nitroaromatics are more toxic than their parent compounds. The nitrosoamines are a class of organic contaminants that are also known carcinogens (15). The compounds formed during the degradation of CL-20 have not been identified, and the possible health effects of these compounds are unknown. A good laboratory technique and protective equipment are required during the entire analysis as a result of both the safety risk associated with the analyte and the need to minimize background current arising from contamination. Protective equipment includes impermeable latex gloves, safety glasses, and fume hoods. Standards and eluents should be disposed of in accordance with approved regulatory practices.

Results and Discussion

The development of an analytical method for CL-20 determination in waters and soils began by identifying a chromatographic system that could separate CL-20 on both the primary and secondary analytical columns. A standard of CL-20 material was obtained from the Naval Air Warfare Center Weapons Division for spiking into a simulated extract. Figure 2 shows chromatograms acquired with CL-20 prepared with purified standards. These solutions were prepared using reference standards in 50% acetonitrile and 50% distilled water. Baseline separation (separation of the CL-20 from other peaks observed during the analysis in which the baseline was achieved between the analyte peak and other peaks) was achieved on both columns.

The elution times for CL-20 were significantly longer on the CN column than on the C-18 column. Generally, the nonpolar C-18 analytical column allows the most polar compounds to

elute first and the nonpolar compounds to elute later. The CN column contains silica coated by a cyanide derivative, which is more polar than the C-18 column. As a result, the polar compounds are retained on this column while the nonpolar compounds elute more quickly.

The use of two columns with different retention characteristics serves two functions. It helps confirm the peak identified on the primary column by identifying the same compound at the same concentration and the distinctive retention time on the second column. The likelihood of an unknown interfering with the peak-matching retention times is quite high on a single column, but not on two columns with dissimilar solid phases. The dual-column technique also serves to remove known interferences.

Another technique that can be used to confirm that the peaks were diagnostic of CL-20 beyond the confirmation is matching the UV–vis spectrum for the peak with the known spectrum of CL-20. A common analytical instrument used for HPLC detection is the photo-diode-array spectrometer, which is capable of measuring a complete UV–vis spectrum at any point along a peak. The spectra of the CL-20 compound can be obtained during elution using a photo-diode-array detector. Figure 3 contains the UV spectra associated with the peak detected at 245 nm that was separated on the CN column. The figure illustrates a maximum absorbance at 230 nm, and no absorbance was evidenced between 280 and 400 nm.

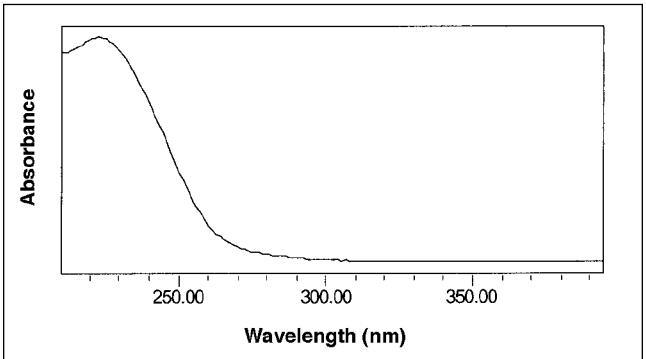

Figure 3. Ultraviolet spectra of CL-20 from a CN column at a 24-min retention time.

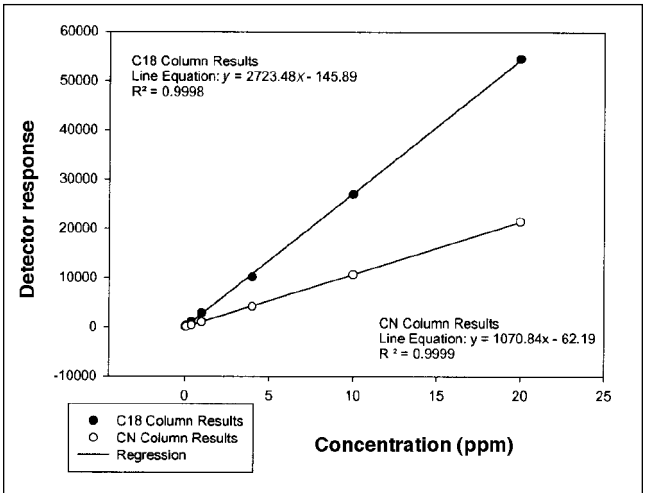

Figure 4. CL-20 calibration curve on a C-18 and CN column.

Determination of the CL-20 concentration in the extracts from soil and water requires a correlation between the detector response and a set of samples with known CL-20 concentrations. This instrument calibration results in a set of paired data for the concentration and detector response that can be plotted and fit to an algebraic correlation. A series of extracts using purified reference standards at known concentrations was prepared and analyzed to determine this correlation. Figure 4 shows the CL-20 calibration curves for the C-18 and CN columns. Excellent linearity was achieved over 3 orders of magnitude of the concentration range (R^2 values for the least-squared linear curve fit were 0.9998 and 0.9983). The difference in slopes can be attributed to the retention time differences between the two columns. Peak heights (instead of peak areas) were used to quantitate chromatographic features because this method yielded more consistent results. The retention time of CL-20 was much longer on the CN column (24 min compared with 8.75 min on the C-18 column). Longer retention times cause chromatographic peaks to broaden, which decreases the peak heights causing a gentler slope for the standard curve. Figure 5 shows a set of chromatograms produced during the preparation of the calibration curves. Retention times were stable throughout the three orders of magnitude in the calibrated concentration range.

Because the use of explosives often results in soil and water contamination in which a number of explosive compounds are present in mixtures, the method must be able to quantitate CL-20 in the presence of these other common compounds. Both

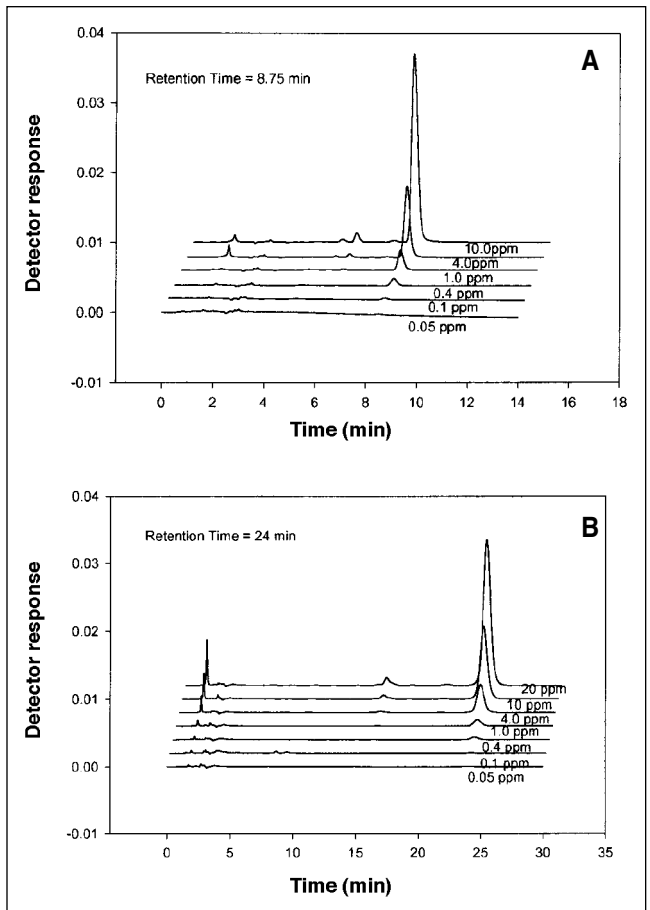

Figure 5. CL-20 calibration on a (A) C-18 and (B) CN column.

TNT and RDX are explosive-based compounds that are common contaminants in both soil and water. Figure 6 shows CN and C-18 chromatograms that exhibit the proposed method's ability to separate the CL-20 from both TNT and RDX. Figure 7 illustrates chromatograms showing the analysis of a mixture of common explosive compounds plus CL-20 using the proposed method on C-18 and CN columns. Separation of CL-20 from the other compounds was excellent on the CN column, but CL-20 coeluted with 1.3-dinitrobenzene (DNB) on the C-18 column. Samples would have to be analyzed on both C-18 and CN columns if DNB was a suspected contaminant.

In accordance with the requirements of U.S. EPA SW846, the MDLs and laboratory reporting limits (LRLs) were determined for the quantitation of CL-20 in the three matrices/extracts described in this report. Table I contains the results of seven replicate runs near the data-reporting limit as well as the statistical interpretation of those results. As can be seen, precision was good for the replicate analysis in both water and soil matrices. The MDLs are 0.10 ng/mL for concentrated extracts in water, 17.1 ng/mL for unconcentrated extracts in water, and 33.93 ng/g for extracts in soil. The LRLs were five times the MDL values, or 0.49 ng/mL, 85.48 ng/mL, and 169.64 ng/g, respectively.

The ability to measure CL-20 at low levels in waters and soils is an important tool for studying the environmental fate and risks associated with the introduction of CL-20 into the environment. Applications of the technique identified in this report included studies of the sorption of CL-20 into soils and sediments, soil column studies to determine the rate of mobility of CL-20 in soils, adsorption/desorption studies to determine groundwater migration rates, CL-20 solubility studies in natural waters, studies of the uptake of explosives by plants, toxicity studies for CL-20, and measurement of the rates of the natural

Table I. MDL Statistics for CL-20 in Water and Soil Matrices

		Concentration (ppb)								Average	Standard	MDL	Recovery	LRL
Compound	Column	µg/L	MDL-1	MDL-2	MDL-3	MDL-4	MDL-5	MDL-6	MDL-7	(µg/L)	deviation	(µg/L)	%	(µg/L)
CL-20 water	C-18	60.00	61.00	58.00	59.00	50.00	53.00	50.00	50.00	54.43	4.79	14.37	90.71	71.86
	CN	60.00	63.00	63.00	54.00	48.00	53.00	53.00	52.00	55.14	5.70	17.10	91.90	85.48
CL-20 SPE	C-18	0.60	0.56	0.56	0.56	0.57	0.56	0.53	0.54	0.55	0.01	0.04	92.38	0.21
	CN	0.60	0.60	0.68	0.60	0.60	0.65	0.60	0.60	0.62	0.03	0.10	103.10	0.49
CL-20 soil	C-18	150.00	91.00	94.00	96.00	93.00	92.00	92.00	93.00	93.00	1.63	4.90	62.00	24.49
	CN	150.00	91.00	96.00	120.00	117.00	97.00	110.00	99.00	104.29	11.31	33.93	69.52	169.64

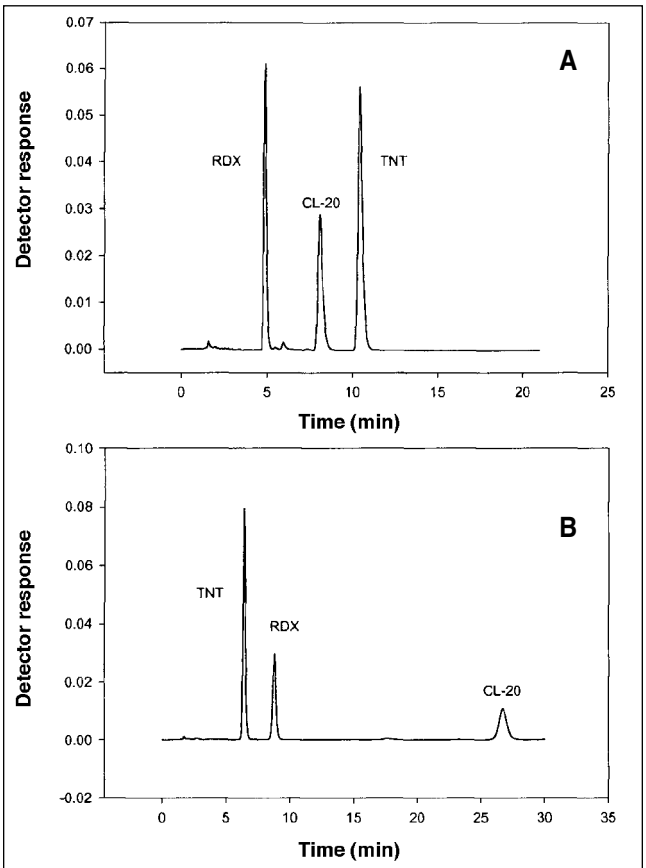

Figure 6. CL-20 separation from TNT and RDX on a (A) C-18 and (B) CN column.

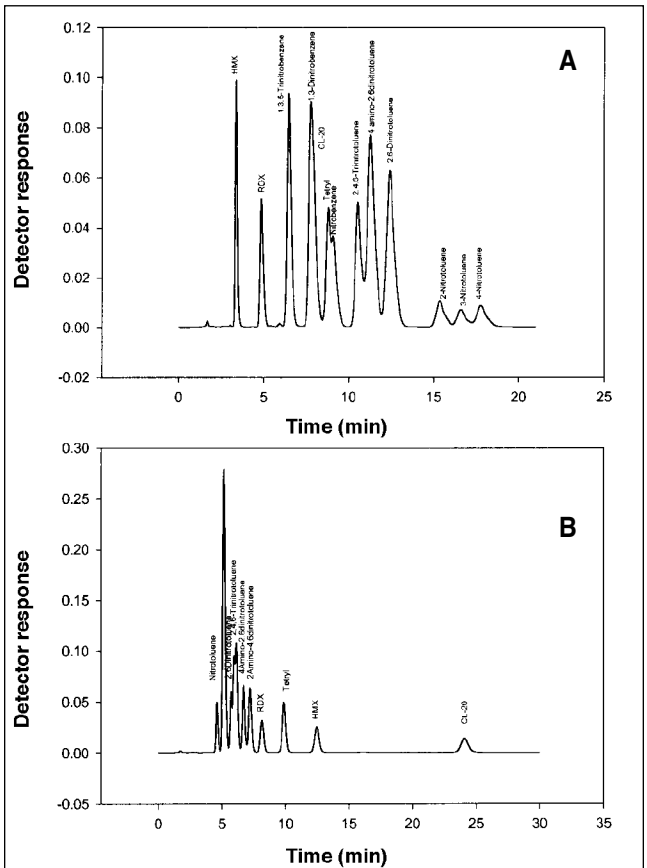

Figure 7. CL-20 separation from common explosives compounds on a (A) C-18 and (B) CN column.

attenuation of CL-20 in waters and soils. The ability to identify the presence of CL-20 and determine its concentration throughout a specific degradation process provides a means of evaluating each specific remediation technology. It is important to have the capability of accurately and reputably measuring CL-20 in the various compartments identified in an experimental matrix. For example, measurement of CL-20 levels in soil sections, influent water, pore water, and effluent water is necessary when studying the mobility of CL-20 in a soil column. Low detection limits are required for compartments that contain small fractions of the total CL-20 present in the entire system.

The ability to measure CL-20 at low levels in waters and soils is also important in studies of technologies for remediating soil and water contaminated with CL-20. These treatments, analogous to those proposed for the remediation of nitroaromatics and nitramines, include the biodegradation of CL-20 (16), thermal processes for the treatment for the mineralization of CL-20 (17), base hydrolysis for CL-20 transformation (18), phytoremediation (the use of plants) to transform CL-20 (19,20), advanced oxidation technologies for CL-20 transformation or mineralization (21), physical separation for the mass reduction of CL-20 contamination (22), and the granulated activate carbon for CL-20 removal from waters.

Conclusion

A means of separation and quantitation of CL-20 in environmental matrices has been developed. This method satisfies the need for analytical techniques to monitor the degradation of CL-20 in remedial systems. The system is based on reversed-phase liquid chromatography for the separation of nitroaromatics and nitramines. C-18 and a CN-bonded silica HPLC columns were used to eliminate common interferences. Contaminant identification was further confirmed by performing a spectral analysis of the compounds upon elution. The method for detecting CL-20 used techniques and equipment common to most analytical laboratories performing explosives detection. Analytically, it was possible to detect CL-20 down to the 500-ppt range. This analytical technique is relatively simple and cost efficient and is expected to be a valuable tool for evaluating CL-20 contamination.

The usefulness of the technique will depend on how CL-20 is used in a weapons system in the future. If its use is similar to that of RDX, TNT, and mixtures of nitramines and nitroaromatics, then the amount of soil and water impacted with this material could be extensive.

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