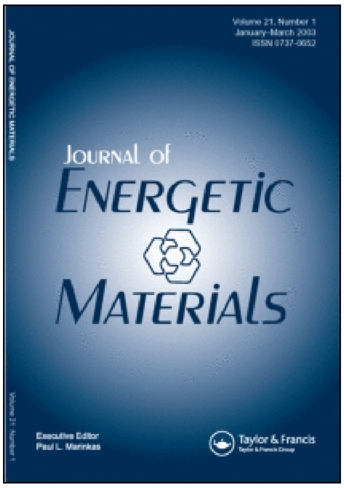

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Analysis of Nitramine and Nitroaromatic Explosives by Micellar Electokinetic Capillary Chromatography (MECC)*

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ABSTRACT

Recently two new explosives, TNAZ, 1,3,3-trinitroazetidine, and CL-20, hexanitrohexaazaisowurtzitane, have attracted interest as possible replacements for or inclusion in various military propellants and explosives. Analysis of these two compounds as well as 12 other nitramine and nitroaromatic explosives could be accomplished by capillary electrophoresis using the technique of MECC, micellar electrokinetic capillary chromatography. Analysis times of less than 10 minutes could be done using this method. Increased sensitivity for some explosives could be accomplished, as well, by using UV detection at 185nm rather than 254nm which is typically used.

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DISCUSSION and RESULTS

TNAZ and CL-20, figure 1, have recently been investigated as possible alternatives to some currently used explosives.[1-3] If such inclusion in military propellants and explosives do occur a method for analyzing for them in the presence of currently used explosives would be necessary. Figure 2 shows the structures of the various explosives currently used in existing propellant formulations and or regulated. Capillary electrophoresis (CE) is one such technique which could be used for the analysis of these compounds. Since all of the compounds are neutral, micellar electrokinetic capillary chromatography (MECC) was used. MECC technique allows for the separation of neutrals by dissolving or diluting the sample into a buffer solution that contains a surfactant which forms a charged micelle. Sodium dodecylsulfate (SDS) is typically used as the surfactant.

Using a borate/boric acid buffer with SDS added a separation of the 12 currently used or regulated nitramine and nitroaromatic explosives as well as TNAZ and CL-20 could be done in less than 10 minutes. Figure 3 is an electropherogram of the separation achieved. Monitoring was done at 254nm which has been used in previous assays for monitoring.[4-5] However with the design of the CE system used it is possible to monitor at 185nm which allows for an improved signal from almost all compounds to be achieved. Figure 4 is an electropherogram of a mix monitored at 185nm (fig. 4A) and 254nm (fig 4B) both scaled the same. As can be seen you get an increase in signal for the nitramine and nitroaromatic explosives by monitoring at 185nm with little increase in noise.

Table 1 is the area count report for each compound at 185nm and 254nm showing that for almost all compounds there was a dramatic increase in peak area and height at the lower wavelength, as expected

EXPERIMENTAL

Instrumentation:

The capillary electrophoresis (CE) system employed was a Quanta™ 4000 (Waters Corp. Milford, MA, U.S.A.) A mercury lamp was used for direct UV detection of the explosives at 254nm and 185nm. AccuSep™ polyimide fused silica capillaries of dimension 50 um I.D. X 60 cm were used throughout. Data acquisition and CE control was carried out with a Waters™ Millennium 2010 Chromatography Manager.

Preparation of Electrolytes:

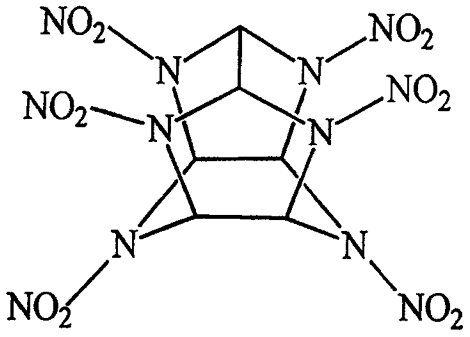
High purity water (Milli-Q, Millipore Corp., Bedford, MA, U.S.A.) was used to prepare all electrolytes and samples. The electrolyte used consisted of a 2.5 mM sodium borate, 12.5 mM boric acid, and 50 mM sodium dodecylsulfate (SDS) solution.

Chemicals:

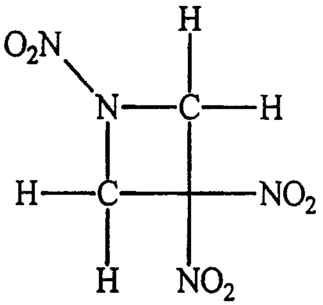
All chemicals used for the preparation of the electrolytes were of ACS grade or better. SDS was obtained in high purity form (electrophoresis grade) from Millipore Corporation (Bedford, MA, U.S.A.). Standards were prepared from a 1.0 mg/mL concentrated standard of all 12 common explosives (Accustandard, New Haven, CT, U.S.A.) and diluted into the running buffer. CL-20 was received as a gift from Dr. Jimmie Oxley of the New Mexico Institute of Mining and TNAZ was a gift from Dr. Tom Archibald of Aerojet Corp.

CONCLUSIONS

As seen in the previous examples capillary electrophoresis can be used for the analysis of TNAZ and CL-20 in the presence of 12 common explosives in less than 10 minutes. Capillary electrophoresis offers a fast and easy method for analyzing for these explosives. Further, monitoring at 185nm allows for increased detection of these explosives.



CL-20



TNAZ

Figure 1. Structure of CL-20, hexanitrohexaazaisowurtzitane, and TNAZ, 1,3,3-trinitroazetidine.

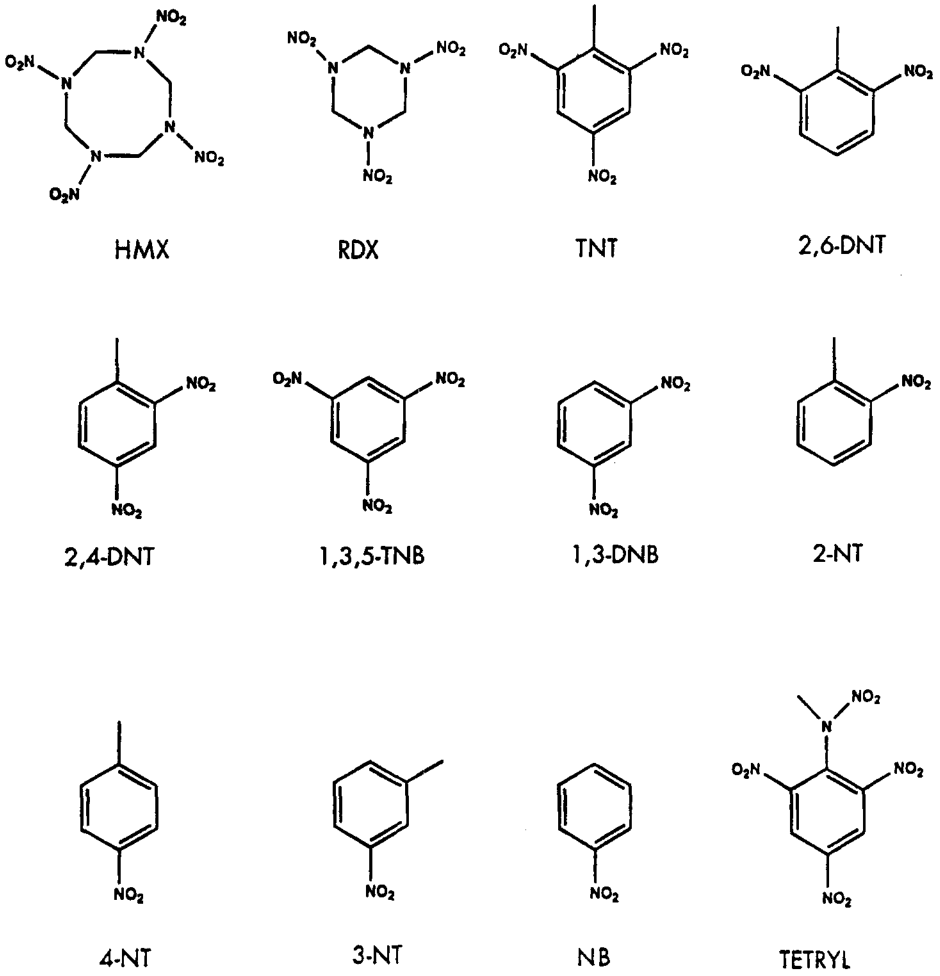


Figure 2: Structure of 12 currently used or regulated explosives used in this analysis.

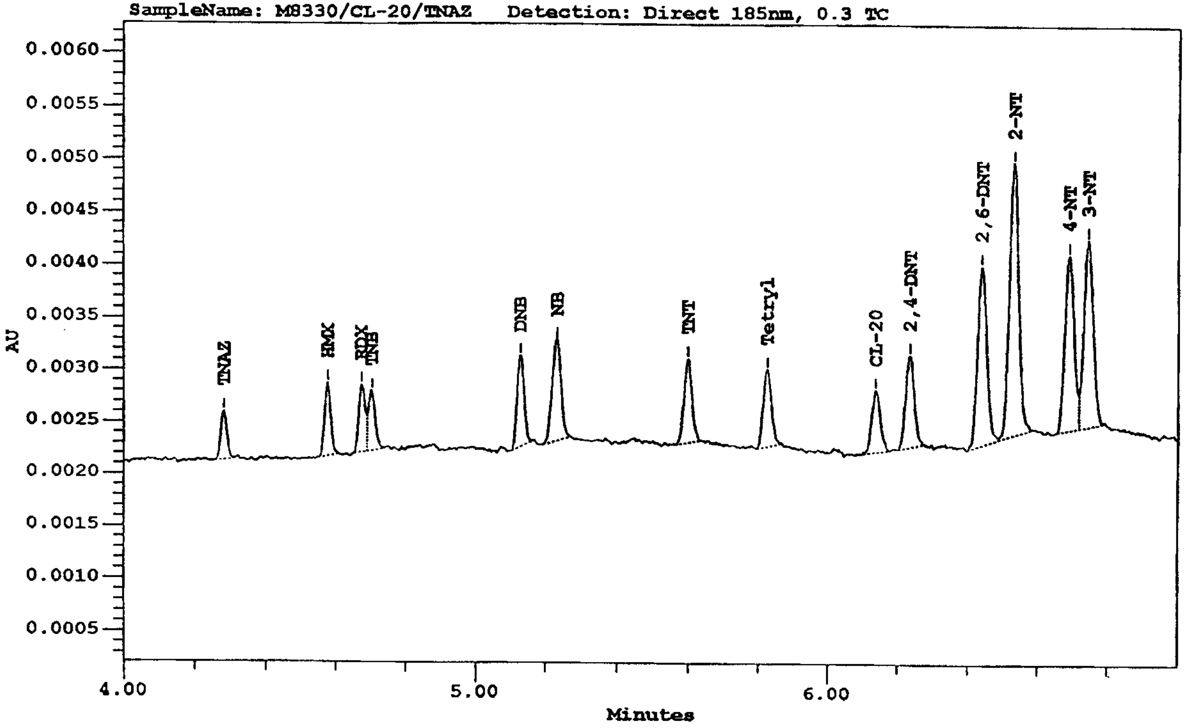


Figure 3: Electropherogram of TNAZ, and CL-20 in the presence of 12 other nitramine and nitroaromatic explosives. All compounds are at a concentration of approximately 10 mg/mL. Conditions as stated in experimental section.

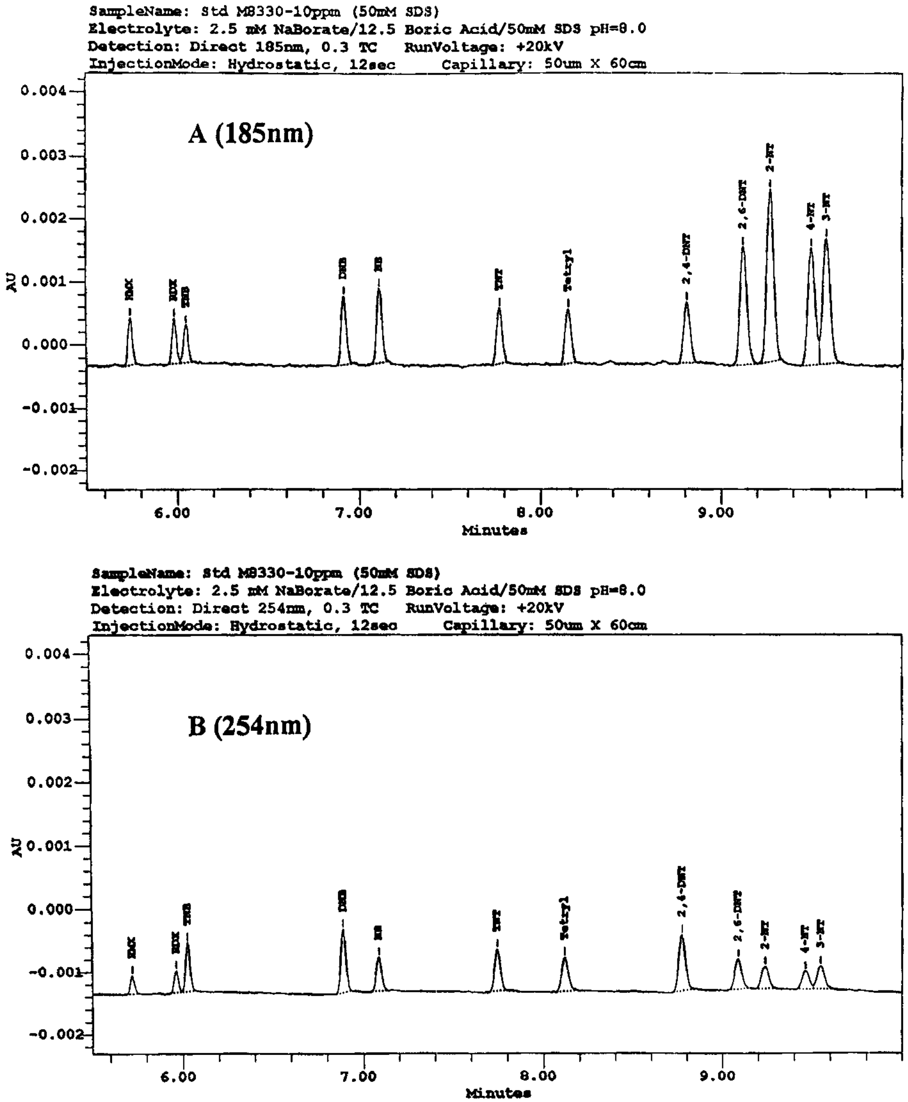


Figure 4: Electropherogram of 12 nitramine and nitroaromatic explosives at 185 nm (A) and 254 nm (B) detection. All compounds are at a concentration of 10 mg/mL. Conditions as stated in experimental section.

Peak_Information

#	Name	Detection	Area (uV*sec)	Height (uV)
1	2,4-DNT	Direct 185nm, 0.3 TC	2451	974
2	2,4-DNT	Direct 254nm, 0.3 TC	2424	912
3	2,6-DNT	Direct 185nm, 0.3 TC	5202	1881
4	2,6-DNT	Direct 254nm, 0.3 TC	1366	496
5	2-NT	Direct 185nm, 0.3 TC	7523	2764
6	2-NT	Direct 254nm, 0.3 TC	1033	358
7	3-NT	Direct 185nm, 0.3 TC	5810	1991
8	3-NT	Direct 254nm, 0.3 TC	1124	376
9	4-NT	Direct 185nm, 0.3 TC	5279	1864
10	4-NT	Direct 254nm, 0.3 TC	836	300
11	DNB	Direct 185nm, 0.3 TC	2215	1094
12	DNB	Direct 254nm, 0.3 TC	2099	1030
13	HMX	Direct 185nm, 0.3 TC	1275	754
14	HMX	Direct 254nm, 0.3 TC	510	307
15	NB	Direct 185nm, 0.3 TC	2491	1196
16	NB	Direct 254nm, 0.3 TC	1154	554
17	RDX	Direct 185nm, 0.3 TC	1261	730
18	RDX	Direct 254nm, 0.3 TC	607	368
19	TNB	Direct 185nm, 0.3 TC	1102	605
20	TNB	Direct 254nm, 0.3 TC	1384	798
21	TNT	Direct 185nm, 0.3 TC	1963	897
22	TNT	Direct 254nm, 0.3 TC	1538	682
23	Tetryl	Direct 185nm, 0.3 TC	2040	883
24	Tetryl	Direct 254nm, 0.3 TC	1417	557
25		Direct 185nm, 0.3 TC	1639	1083

Table 1: Peak area and height counts for each component at 185nm and 254 nm detection.

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