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MEMORANDUM REPORT ARBRL-MR-03121

THE DETERMINATION OF HEXAHYDRO-1, 3,
5,-TRINITRO-S-TRIAZINE (RDX) AND OCTAHYDRO-
1, 3, 5, 7-TETRANITRO-S-TETRAZOCINE (HMX) BY
HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

J. Omar Doali
Arpad A. Juhasz

August 1981

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
BALLISTIC RESEARCH LABORATORY
ABERDEEN PROVING GROUND, MARYLAND

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) jmk A method is described, using high performance liquid chromatography, to determine quantitatively hexahydro-1, 3, 5-trinitro-s-triazine (RDX) and octahydro-1, 3, 5, 7-tetranitro-s-tetrazocine (HMX) in a mixture and as propellant ingredients. Separations are achieved on a Micro Pak-CN column, 250-mm long x 2.1-mm internal diameter, using a mobile phase of 30-percent methylene chloride/20-percent acetonitrile/50-percent hexane. Under these conditions, the two compounds are separated in approximately ten minutes.		

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I. INTRODUCTION

The determination of octahydro-1, 3, 5, 7-tetranitro-s-tetrazocine (HMX) and hexahydro-1, 3, 5-trinitro-s-triazine (RDX) in mixtures and especially the determination of RDX as an HMX contaminant has been a continuing problem because of the structural similarities of the compounds. This has been an especially important problem because it has been demonstrated that the impure compounds exhibit lower thermal stability than the pure compounds¹. The structures and pertinent physical properties of these nitramines are presented in Appendices A and B.

Due to the difficulties involved in the analyses, a variety of techniques including wet chemical procedures and instrumental methods have been applied to the problem with varying degrees of success. Reviews²⁻⁴ are available which discuss the various techniques. Recently, a gas chromatographic method⁵ and an infrared spectrophotometric method⁶ were developed to perform the analyses. However, both procedures are only applicable to the determination of RDX.

Within the Ballistic Research Laboratory, interest developed in procedures to determine RDX and HMX due to the advent of low vulnerability propellants containing these nitramines. Additionally, an on-going project involving thermal decomposition studies of RDX and HMX necessitated the development of procedures to determine the purity of the parent

¹J. Stahls, "Report on the Basic Chemistry of RDX and α - and β -HMX and Its Application", (U) Defense Standards Laboratories Report 400 (Australia), November 1970, (CONFIDENTIAL).

²H.J. Scullion, "A Survey of the Methods Available for the Analysis of HMX/RDX Mixtures", Ministry of Defense, UK Army Department, Chemical Inspectorate Memorandum No. 169, 1965.

³Earle F. Reese, "Resume of Analytical Methods for the Determination of RDX and HMX 1943-1965", Picatinny Arsenal Technical Report No. 3309, December 1965.

⁴Mortimer Schwartz and Elizabeth A. Mark, "Quantitative Spectrophotometric Determination of Hexahydro-1, 3, 5-Trinitro-s-Triazine in Octahydro-1, 3, 5, 7-Tetranitro-s-Tetrazocine", *Anal. Chem.*, 38, pp. 610-612, April 1966.

⁵M.L. Rowe, "Determination of Hexahydro-1, 3, 5-Trinitro-s-Triazine in Octahydro-1, 3, 5, 7-Tetranitro-s-Tetrazocine by Gas Chromatography", *J. Gas Chromatography*, 5, pp. 531-532, October 1967.

⁶J.W. Grindlay, "Determination of Hexahydro-1, 3, 5-Trinitro-s-Triazine (RDX) in Octahydro-1, 3, 5, 7-Tetranitro-s-Tetrazocine (HMX) by Infrared Spectrophotometry", *Anal. Chem.*, 44, pp. 1676-1678, August 1972.

compounds. Previous experience in the analysis of energetic materials using high performance liquid chromatography (HPLC)⁷ indicated that these nitramines could be separated and possibly determined in a straightforward manner. This report presents the results obtained using an HPLC procedure as a solution to the problem of nitramine analysis.

II. EXPERIMENTAL

The investigation was performed using a DuPont Model 830 Liquid Chromatograph equipped with a fixed wavelength (254 nm) ultraviolet detector. A Spectra Physics Minigrator monitored the output of the detector and produced a printout of the retention times and peak areas. A Micro Pak-CN column (250 mm x 2.1 mm i.d.) was used to achieve the separations. The mobile phase solvents were acetonitrile (UV), methylene chloride, and hexane (UV) obtained from Burdick and Jackson Laboratories, Inc.

The other important instrumental conditions are summarized below:

Mobile Phase: 30-percent CH_2Cl_2 /20-percent CH_3CN /50-percent hexane

Mode: Normal (constant composition)

Pressure: 2.75 MPa, 400 psig

Flow Rate: 0.5 mL/min

Temperature: Ambient (21-22°C)

Under these conditions, the separation of HMX and RDX was complete in approximately ten minutes as shown in Figure 1. Injections were made using a Disc Instruments sample valve which had an internal volume of 10 μL . This valve has demonstrated a repeatability of better than one percent relative.

Purification of HMX was achieved by repeated crystallizations from acetone while RDX was purified by sublimation. The compounds were considered pure when a 10 ng injection of either compound resulted in the detection of only one peak at the highest sensitivity of the detector (0.01 AUFS).

⁷ J. Omar Doali and Arpad A. Juhasz, "High Speed Liquid Chromatographic Separations of Thermally Labile High Energy Compounds. Part I Application of High Speed Liquid Chromatography to the Qualitative Analysis of Compounds of Propellant and Explosives Interest", Ballistic Research Laboratory Report No. 1644, Ballistic Research Laboratory, Aberdeen Proving Ground, MD, April 1973. (AD #910482L)

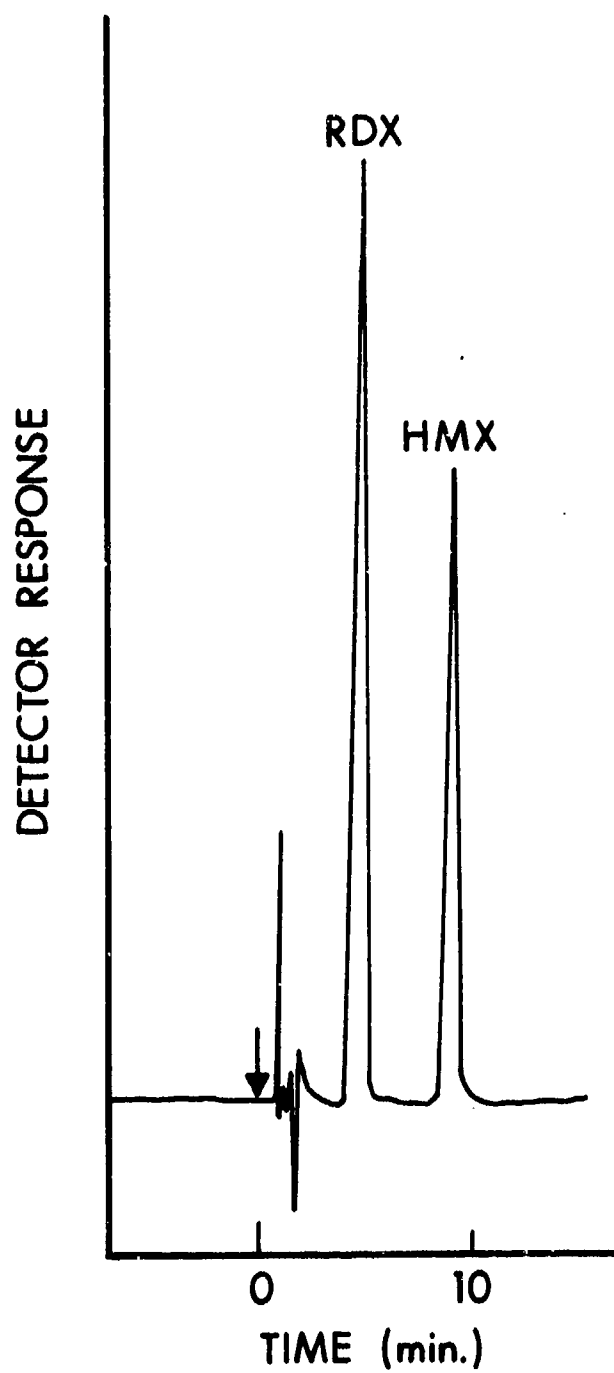


Figure 1. Chromatographic Separation of RDX and HMX

Under these conditions, RDX would be detectable down to at least 3 ng while the detectable quantity of HMX would be slightly higher due to its lower molar absorptivity. The materials which passed this quality control procedure, were then used as standards in all of the analyses.

III. RESULTS AND DISCUSSION

A. Limits of Quantification

Four standard solutions of RDX in acetonitrile were prepared to cover a concentration range of 12 ng to 103 ng per 10 μ L. Injection of these solutions with the detector at its highest sensitivity resulted in the calibration curve shown in Figure 2. The curve is linear with a near zero intercept and a correlation coefficient of 0.999. The 12-ng sample appeared to be the lower limit of quantification due to the detector noise at this high sensitivity. This means that the injection of a 10- μ g sample of HMX would allow the determination of RDX contamination down to approximately 0.1 percent. Though 10- μ g samples were chosen for this investigation, there is no reason why 20- μ g samples could not be used which would increase the quantification of RDX down to approximately 0.05 percent.

The limit of quantification for HMX would be somewhat higher than RDX because of its previously mentioned lower molar absorptivity. However, the detectability of both compounds would be improved if the analyses were performed at a shorter wavelength by using a variable wavelength ultraviolet detector. The limiting factor would be the UV cutoff of the methylene chloride contained in the mobile phase which occurs at approximately 245 nm.

B. Synthetic Sample Analysis

To determine the accuracy and precision of the procedure with respect to small quantities of RDX, a solution was prepared which contained 5.54 μ g of HMX per 10 μ L (99.08%) and 51.6 ng of RDX per 10 μ L (0.92%). Four injections were made into the chromatograph and the resulting peak areas compared with those resulting from the injection of RDX standards. The percent RDX was calculated using the following expression:

$$\%RDX = \frac{W_s \cdot A_x}{A_s \cdot W_t} \cdot 100$$

where W and A are the weights and peak areas respectively, the subscripts refer to the standard (s), the "unknown" RDX (x) and the total sample weight (t). Table 1 shows the mean values ($\bar{x}$), the sample standard deviations (s) and the relative standard deviations resulting from the analysis. The results indicate good accuracy for RDX with acceptable precision for the small RDX peaks.

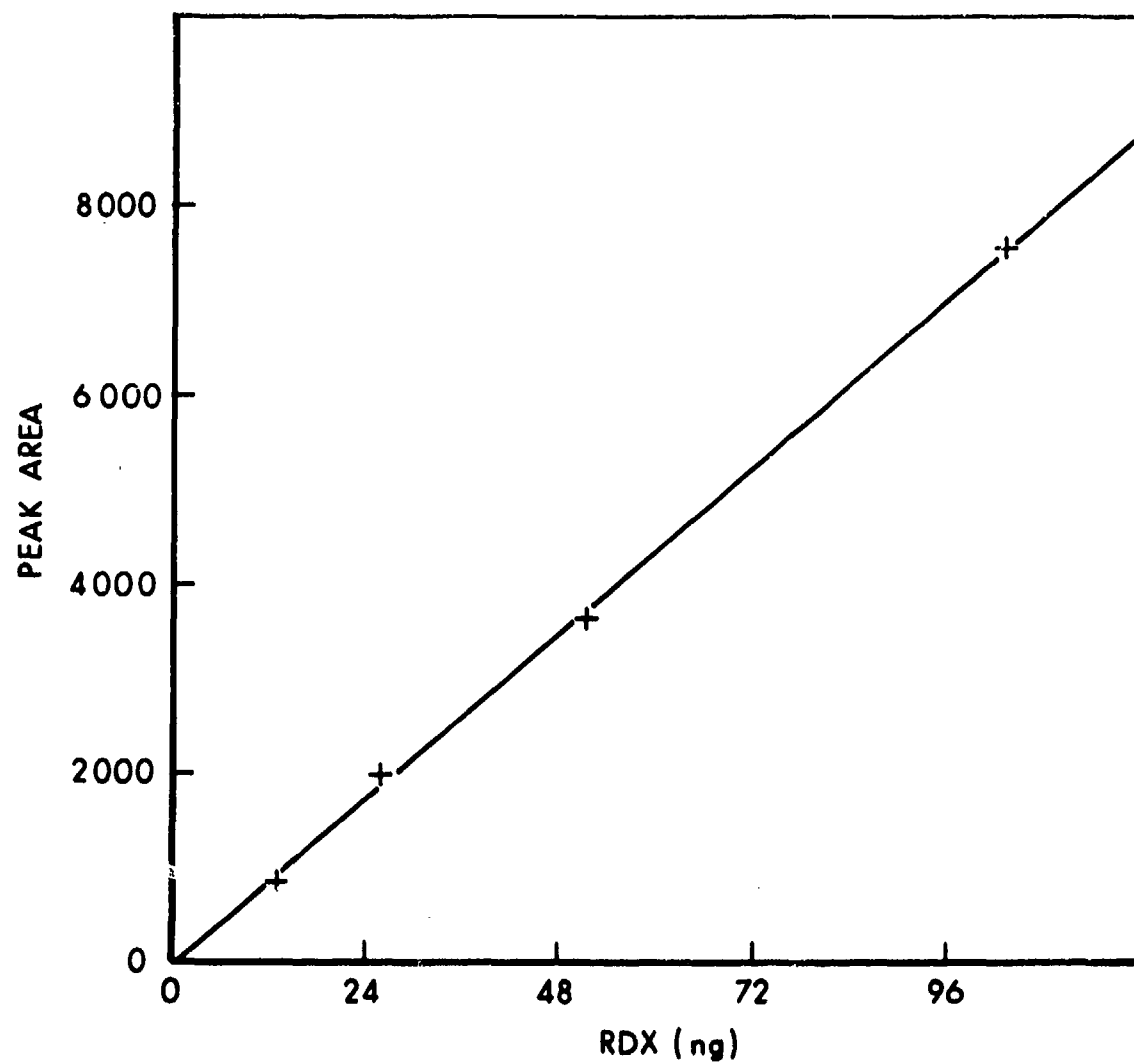


Figure 2. Response of Detector to Small Quantities of RDX

TABLE 1. RESULTS OF SYNTHETIC RDX/HMX MIXTURE ANALYSIS

<u>RDX</u>	
<u>% Added</u>	<u>% Found</u>
0.92	0.91
	0.94
	0.87
	0.90
	$\bar{x} = 0.91$
	$s = 0.03$
	Rel. s = 3.2%

C. Determination of HMX in a Propellant

The propellant chosen for analysis was a low vulnerability formulation PPLA 6379 which had the following nominal composition:

75% HMX
20% Cellulose Acetate
4.5% Nitrocellulose
0.5% Wax
0.3% Ethyl Centralite

Four standard solutions of HMX were prepared to contain from 1.1 μg to 6.6 μg per 10 μL . Injection of the standards in duplicate and measurement of their peak areas resulted in a linear calibration curve with a correlation coefficient of 0.999.

Duplicate 500-mg samples of the propellant weighed to ± 0.1 mg were placed in 1-liter volumetric flasks and filled to the mark with acetonitrile. Clear solutions were obtained after the flasks sat overnight with subsequent shaking. Injection of a sample into the chromatograph resulted in only one peak (HMX) eluting after the solvent front as shown in Figure 3. Under these separation conditions, the ethyl centralite would elute at the solvent front. Several injections were made over a period of approximately three hours to determine if any other peaks eluted. There was no evidence of either other peaks eluting from the column or a change in column characteristics.

In order to compensate for any fluctuations in flow rate, a standard was injected prior to each unknown. Three injections of each propellant solution were made and the percent HMX calculated using the same expression used for the RDX determination.

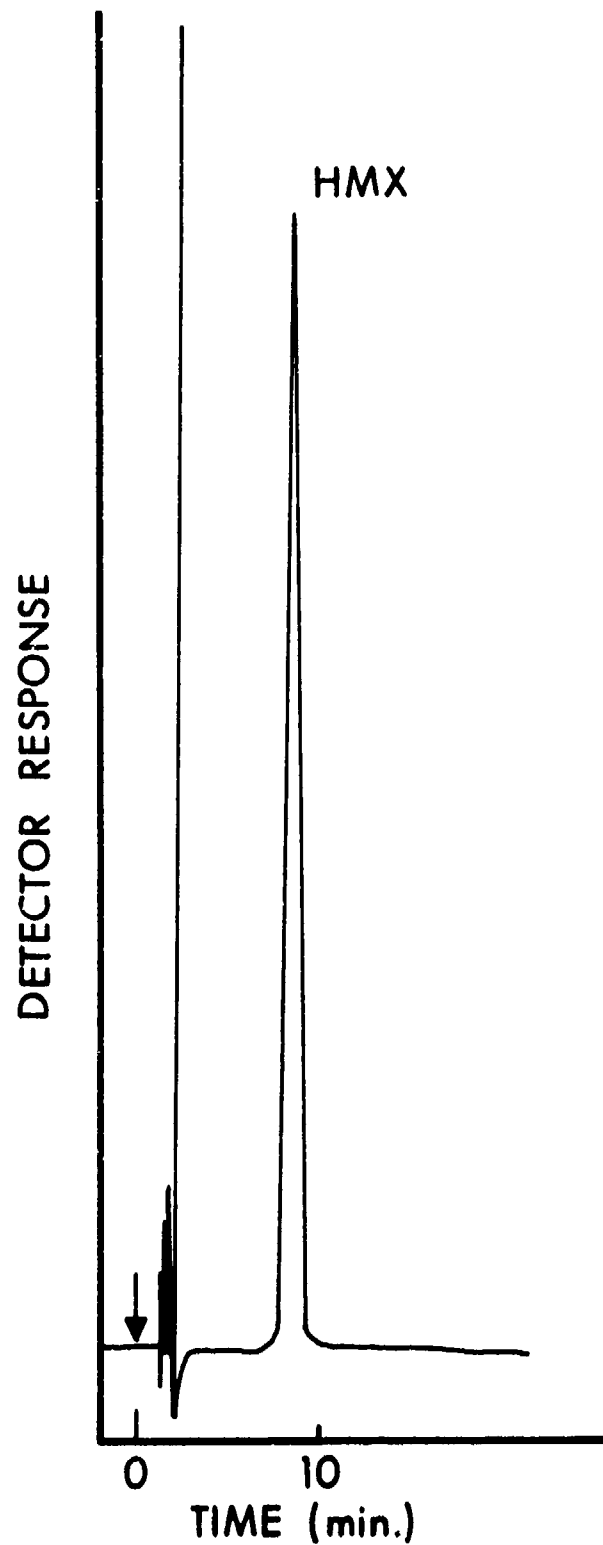


Figure 3. Chromatogram of Propellant Solution

The results as presented in Table 2 indicate good precision and agreed well with the nominal HMX concentration in the propellant. The only complications which may arise in the determination of the nitramines in the new propellants are the variety of binders and additives used in these formulations. Each propellant may have to be approached as a unique problem with respect to achieving sample dissolution and possible interferences due to other propellant ingredients.

TABLE 2. RESULTS OF PROPELLANT ANALYSIS

	%HMX
	74.62
	76.10
	74.62
	73.93
	74.44
	75.45
$\bar{x}$	= 74.86
s	= 0.78
Relative s	= 1.0%

Fortunately, during the course of the analyses, it was found that the results obtained using peak heights agreed with those obtained using peak areas. Consequently, instrumental requirements for the procedure are simplified.

IV. CONCLUSION

The investigation demonstrates that HPLC can be successfully applied to the quantitative determination of HMX and RDX in admixture or separately. The purity of either compound can be determined in a straight forward manner without complications. The procedure has been selected as a tentative method for MIL-STD 286B, Propellants, Solid: Sampling, Examination and Testing and MIL-STD 650, Explosives: Sampling, Inspection and Testing. Appendix C presents the method in its MIL-STD format. The applicability of the method to the determination of HMX in a low vulnerability propellant has also been demonstrated. As a result, a simple quality control procedure is available to determine both of the compounds in the new nitramine propellants.

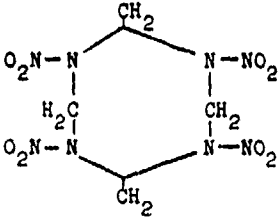
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1. J. Stals, "Report on the Basic Chemistry of RDX and α -and- β -HMX and Its Application", (U) Defense Standards Laboratories Report 400 (Australia), November 1970. (Confidential).
2. H.J. Scullion, "A Survey of the Methods Available for the Analysis of HMX/RDX Mixtures", Ministry of Defense, UK Army Department, Chemical Inspectorate Memorandum No. 169, 1965.
3. Earle F. Reese, "Resume of Analytical Methods for the Determination of RDX and HMX 1943-1965", Picatinny Arsenal Technical Report No. 3309, December 1965.
4. Mortimer Schwartz and Elizabeth A. Mark, "Quantitative Spectrophotometric Determination of Hexahydro-1, 3, 5-Trinitro-s-Triazine in Octahydro-1, 3, 5, 7-Tetranitro-s-Tetrazocine", Anal. Chem., 38, pp. 610-612, April 1966.
5. M.L. Rowe, "Determination of Hexahydro-1, 3, 5-Trinitro-s-Triazine in Octahydro-1, 3, 5, 7-Tetranitro-s-Tetrazocine by Gas Chromatography", J. Gas Chromatography, 5, pp. 531-532, October 1967.
6. J.W. Grindlay, "Determination of Hexahydro-1, 3, 5-Trinitro-s-Triazine (RDX) in Octahydro-1, 3, 5, 7-Tetranitro-s-Tetrazocine (HMX) by Infrared Spectrophotometry", Anal. Chem., 44, pp. 1676-1678, August 1972.
7. J. Omar Doali and Arpad A. Juhasz, "Hight Speed Liquid Chromatographic Separations of Thermally Labile High Energy Compounds. Part I Application of High Speed Liquid Chromatography to the Qualitative Analysis of Compounds of Propellant and Explosives Interest" Ballistic Research Laboratory Report No. 1644, Ballistic Research Laboratory, Aberdeen Proving Ground, MD, April 1973. (AD #910482L)

APPENDIX A

STRUCTURE AND PHYSICAL PROPERTIES OF HMX

The following pages were copied from AMCP 706-177, Engineering Design Handbook, Explosives Series, Properties of Explosives of Military Interest, January 1971.

Composition: % C 16.2 H 2.7 N 37.9 O 43.2 C/H Ratio 0.095				Molecular Weight: (C ₄ H ₈ N ₈ O ₈) 296
		Oxygen Balance: CO ₂ % -21.6 CO % 0.0		
		Density: gm/cc Crystal 1.90		
		Melting Point: °C Capillary method 273 Koffler Micro Hot Stage 280		
		Freezing Point: °C		
Impact Sensitivity, 2 Kg Wt: Bureau of Mines Apparatus, cm 32 Sample Wt 20 mg Picatinny Arsenal Apparatus, in. 9 Sample Wt, mg 23		Boiling Point: °C		
		Refractive Index, n_D²⁰ n _D ²⁵ n _D ³⁰		
Friction Pendulum Test: Steel Shoe Explodes Fiber Shoe Unaffected		Vacuum Stability Test: cc/40 Hrs, at 90°C 100°C 0.37 120°C 0.45 135°C -- 150°C 0.62		
Rifle Bullet Impact Test: Trials Explosions % Partial Burned Unaffected		200 Gram Bomb Sand Test: Sand, gm 60.4		
Explosion Temperature: °C Seconds, 0.1 (no cap used) 380 1 -- 5 327 10 306 15 -- 20 --		Sensitivity to Initiation: Minimum Detonating Charge, gm Mercury Fulminate Lead Azide 0.30 Tetryl		
		Ballistic Mortar, % TNT: 150		
		Treuzl Test, % TNT: 145		
75°C International Heat Test: % Loss in 48 Hrs		Plate Dent Test: Method Condition Confined Density, gm/cc Brisance, % TNT		
100°C Heat Test: % Loss, 1st 48 Hrs 0.05 % Loss, 2nd 48 Hrs 0.03 Explosion in 100 Hrs None		Detonation Rate: Confinement Condition Charge Diameter, in. Density, gm/cc 1.84 Rate, meters/second 9124		
Flammability Index:				
Hygroscopicity: % 30°C, 95% RH (c) 0.00				
Volatility:				

Booster Sensitivity Test: Condition Tetryl, gm Wax, in. for 50% Detonation Wax, gm Density, gm/cc		Decomposition Equation: Oxygen, atoms/sec (Z/sec) Heat, kilocalorie/mole (ΔH , kcal/mol) Temperature Range, °C Phase		(e) $10^{19.7}$ 52.7 271-314 Liquid
Heat of: Combustion, cal/gm Explosion, cal/gm (e) Gas Volume, cc/gm Formation, cal/gm (e) Fusion, cal/gm		Armor Plate Impact Test: 60 mm Mortar Projectile: 50% Inert, Velocity, ft/sec Aluminum Fineness 500-lb General Purpose Bomb: Plate Thickness, inches 1 1¼ 1½ 1¾		
Specific Heat: cal/gm/°C °C -75 0.153 0 0.228 25 0.248 50 0.266 75 0.282		Recrystallized (g) °C 85 0.288 90 0.290 100 0.295 125 0.307 150 0.315		
Burning Rate: cm/sec		Bomb Drop Test: T7, 2000-lb Semi-Armor-Piercing Bomb vs Concrete: Max Safe Drop, ft 500-lb General Purpose Bomb vs Concrete: Height, ft Trials Unaffected Low Order High Order 1000-lb General Purpose Bomb vs Concrete: Height, ft Trials Unaffected Low Order High Order		
Thermal Conductivity: cal/sec/cm/°C				
Coefficient of Expansion: Linear, %/°C Volume, %/°C				
Hardness, Mohs' Scale: (e) 2.3				
Young's Modulus: E', dynes/cm² E, lb/inch² Density, gm/cc				
Compressive Strength: lb/inch²				
Vapor Pressure: °C mm Mercury				

APPENDIX B

STRUCTURE AND PHYSICAL PROPERTIES OF RDX

The following pages were copied from AMCP 706-177, Engineering Design Handbook, Explosives Series, Properties of Explosives of Military Interest, January 1971.

Cyclonite* (RDX)

AMCP 706-177

Composition: % C 16.3 H 2.7 N 37.8 O 43.2 C/H Ratio 0.095		Molecular Weight: $(C_3H_6N_6O_6)$ 222 Oxygen Balance: CO ₂ % -22 CO % 0.0 Density: gm/cc Crystal 1.82 Melting Point: °C 204 Freezing Point: °C	
Impact Sensitivity, 2 Kg Wt: Bureau of Mines Apparatus, cm 32 Sample Wt 20 mg Picatinny Arsenal Apparatus, in. 8 Sample Wt, mg 18		Boiling Point: °C Refractive Index, n_D^{20} n_D^{25} n_D^{30}	
Friction Pendulum Test: Steel Shoe Explodes Fiber Shoe Unaffected		Vacuum Stability Test: cc/40 Hrs, at 90°C 100°C 0.7 120°C 0.9 135°C - 150°C 2.5	
Rifle Bullet Impact Test: Trials Explosions 100 Partial 0 Burned 0 Unaffected 0		200 Gram Bomb Sand Test: Sand, gm 60.2	
Explosion Temperature: °C Seconds, 0.1 (no cap used) 405 1 316 5 Decomposes 260 10 240 15 235 20 -		Sensitivity to Initiation: Minimum Detonating Charge, gm Mercury Fulminate 0.19* Lead Azide 0.05* Tetryl - * Alternative initiating charges.	
75°C International Heat Test: % Loss in 48 Hrs 0.03		Ballistic Mortar, % TNT: (a) 150 Trauzl Test, % TNT: (b) 157	
100°C Heat Test: % Loss, 1st 48 Hrs 0.04 % Loss, 2nd 48 Hrs 0.00 Explosion in 100 Hrs None		Plate Dent Test: (c) Method A Condition Pressed Confined Yes Density, gm/cc 1.50 Brisance, % TNT 135	
Flammability Index: (d) 278		Detonation Rate: Confinement None Condition Pressed Charge Diameter, in. 1.0 Density, gm/cc 1.65 Rate, meters/second 8180	
Hygroscopicity: % 25°C, 100% RH 0.02			
Volatility: Nil			

*Name given by Clarence J. Bain of Picatinny Arsenal. Germans call it Hexogen; Italians call it T4; British, RDX.

Bonster Sensitivity Test: Condition Tetryl, gm Wax, in. for 50% Detonation Wax, gm Density, gm/cc	Decomposition Equation: Oxygen, atms/sec (Z/sec) Heat, kilocalorie/mole (ΔH, kcal/mol) Temperature Range, °C Phase	(1) 18.5 10 ^{18.5} 47.5 213-299 Liquid																				
Heat of: Combustion, cal/gm Explosion, cal/gm Gas Volume, cc/gm Formation, cal/gm Solution, cal/mol (28-55% HNO ₃) *Assuming cyclonite unimolecular	2285 1280 908 -96 7.169*																					
Specific Heat: cal/gm/°C <table><thead><tr><th colspan="2">°C</th><th colspan="2">°C</th></tr></thead><tbody><tr><td>20</td><td>0.298</td><td>100</td><td>0.406</td></tr><tr><td>40</td><td>0.331</td><td>120</td><td>0.427</td></tr><tr><td>60</td><td>0.360</td><td>140</td><td>0.446</td></tr><tr><td>80</td><td>0.384</td><td></td><td></td></tr></tbody></table>	°C		°C		20	0.298	100	0.406	40	0.331	120	0.427	60	0.360	140	0.446	80	0.384				
°C		°C																				
20	0.298	100	0.406																			
40	0.331	120	0.427																			
60	0.360	140	0.446																			
80	0.384																					
Burning Rate: cm/sec																						
Thermal Conductivity: cal/sec/cm/°C Density, gm/cc	(h) 1.263 1.533	6.91 x 10 ⁻⁴ 6.98 x 10 ⁻⁴																				
Coefficient of Expansion: Linear, %/°C Volume, %/°C																						
Hardness, Mohs' Scale:	2.5																					
Young's Modulus: E', dynes/cm ² E, lb/inch ² Density, gm/cc																						
Compressive Strength: lb/inch²																						
Vapor Pressure: °C	mm Mercury																					

Armor Plate Impact Test: 60 mm Mortar Projectile: 50% Inert, Velocity, ft/sec Aluminum Fineness 500-lb General Purpose Bombs: Plate Thickness, inches 1 1¼ 1½ 1¾	Bomb Drop Test: T7, 2000-lb Semi-Armor-Piercing Bomb vs Concrete: Max Safe Drop, ft 500-lb General Purpose Bomb vs Concrete: Height, ft Trials Unaffected Low Order High Order 1000-lb General Purpose Bomb vs Concrete: Height, ft Trials Unaffected Low Order High Order
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APPENDIX C

Procedure For the Determination of RDX and HMX in MIL-STD Format

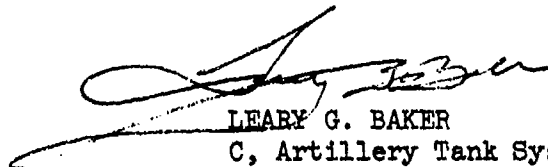
DISPOSITION FORM

For use of this form, see AR 346-15, the proponent agency is TAGCEN.

REFERENCE OR OFFICE SYMBOL DRDAR-QAR-R	SUBJECT Report from Dr. Juhasz - Quantitative Determination of Propellant Ingredients by High Performance Liquid Chromotography (HPLC)
TO Director, PAD	FROM C, DRDAR-QAR
	DATE DEC 13 1978 CMT 1 Mr. Gultz/jt/5626

1. Per your note, dated 6 December 1978, the subject report was reviewed. "Comments, conclusions and implementation actions" are provided below.
2. Subject report contains test procedures (HPLC) for a number of ingredients that are used in propellant formulations (i.e. DNT, DBP, NG, EC, etc.). These methods have already been implemented in MIL-STD-286, Propellants, Solid: Sampling, Examination and Testing.
3. Subject report also, contains test procedures (HPLC) in connection with RDX and HMX. This Command has previously approved these procedures for use by HOLSTON AAP as equivalent test procedures for specification testing of Composition B, RDX and HMX. Action has been initiated to include these procedures in MIL-STD-286 and MIL-STD-650, Explosives: Sampling, Inspection and Testing.

1 Incl
Subject rpt


LEARY G. BAKER
C, Artillery Tank Systems Division

APPENDIX C

RDX AND HMX (LIQUID CHROMATOGRAPHIC METHOD)

1. SCOPE

- 1.1 This method may be used to determine HMX and RDX in a mixture or separately.

2. SPECIMEN

- 2.1 The specimen shall consist of sufficient material to give a final concentration of 1 mg/mL of the major component in a purity determination of either compound as a major ingredient, a final concentration of 0.2 mg/mL to 0.8 mg/mL is required.

3. APPARATUS

- 3.1 High pressure liquid chromatograph (DuPont 830 or equivalent equipped with an ultraviolet detector (254 nm)).
- 3.2 Micro Pak-CN (10 μ particle size) column 250 mm x 2.1 mm i.d. (Varian Instrument Division or equivalent).
- 3.3 Sample injection valve of 10-microliter volume (Disc Instruments Model 704-4-16 or equivalent).
- 3.4 Volumetric flasks as required.
- 3.5 Volumetric pipets as required.

4. MATERIALS

- 4.1 Acetonitrile (UV) (Burdick and Jackson, Inc. or equivalent).
- 4.2 Methylene Chloride (Burdick and Jackson, Inc. or equivalent).
- 4.3 Hexane (UV) (Burdick and Jackson, Inc. or equivalent).
- 4.4 HMX purified by crystallization from acetone or equivalent quality.
- 4.5 RDX purified by sublimation or equivalent quality.
- 4.6 Acetone, reagent grade or equivalent.

5. PROCEDURE

- 5.1 Instrument conditions.
- 5.1.1 Wave length of detector: 254 nm.

- 5.1.2 Column temperature: ambient.
- 5.1.4 Mobile phase: 30% methylene chloride/20% acetonitrile/50% Hexane.
- 5.1.5 Pressure: 2.75 MPa (400 psig).
- 5.1.6 Flow Rate: 0.5 mL/min.
- 5.1.7 Recorder Chart speed: 2.54 mm/min.
- 5.1.8 Under these conditions RDX will elute before HMX, see Figure C-1.
- 5.2 Sample Analysis.
 - 5.2.1 All solutions shall be made using acetonitrile as the solvent.
 - 5.2.2 All weighings shall be ± 0.1 mg.
 - 5.2.3 RDX and HMX used in preparation of standards shall exhibit only one peak when 10 μ g of each compound is injected into the chromatograph with the ultraviolet detector at a sensitivity of 0.01 AUFS.
 - 5.2.4 Determination of RDX contaminant in HMX.
 - a) Prepare RDX standards in volumetric flasks to contain 1.0 mg, 0.50 mg, 0.25 mg, and 0.12 mg per 100 mL. These standards correspond to a concentration of 1%, 0.5%, 0.25%, and 0.12% RDX in a 10- μ g sample of HMX.
 - b) Allow liquid chromatograph to stabilize.
 - c) Set ultraviolet detector to 0.01 AUFS.
 - d) Inject the standards (10 μ L) and obtain a linear calibration curve of peak height vs. weight of RDX.

NOTE: Once the linearity of detector response has been determined over the concentration range of interest, it is only necessary to run a standard prior to each analysis.
 - e) Dissolve 250 mg of the HMX sample in a 250-mL volumetric flask.
 - f) Inject standard solution into the chromatograph.
 - g) Make two 10- μ L injections of the HMX solution into the chromatograph.
 - h) Calculate the percent RDX(x) in the sample using the following expression:

$$\%X = \frac{W_s \cdot H_x}{H_s \cdot W_t}$$

Where: W = weight
 H = peak height
 s = standard
 t = sample
 x = unknown

5.2.5 Determination of HMX contaminant in RDX

- a) Follow procedure 5.2.4, substituting RDX for HMX and HMX for RDX where appropriate.

5.2.6 Determination of HMX and RDX as major ingredients.

- a) prepare four standard solutions of each compound to contain 0.2 mg to 0.8 mg per mL.
- b) Adjust the detector attenuator so that all 10-μL injections result in on-scale peaks.
- c) Obtain linear calibration curves of peak height vs. weight of the nitramines.
- d) Select weight of sample to be analyzed such that the weights of nitramines will be within calibration range.
- e) Inject standard solution.
- f) Inject duplicates (10 μL) and calculate quantity of nitramine (x) using the following expression:

$$W_x = \frac{W_s \cdot H_x}{H_s}$$

Where the symbols represent the same parameters described in the previous expression.

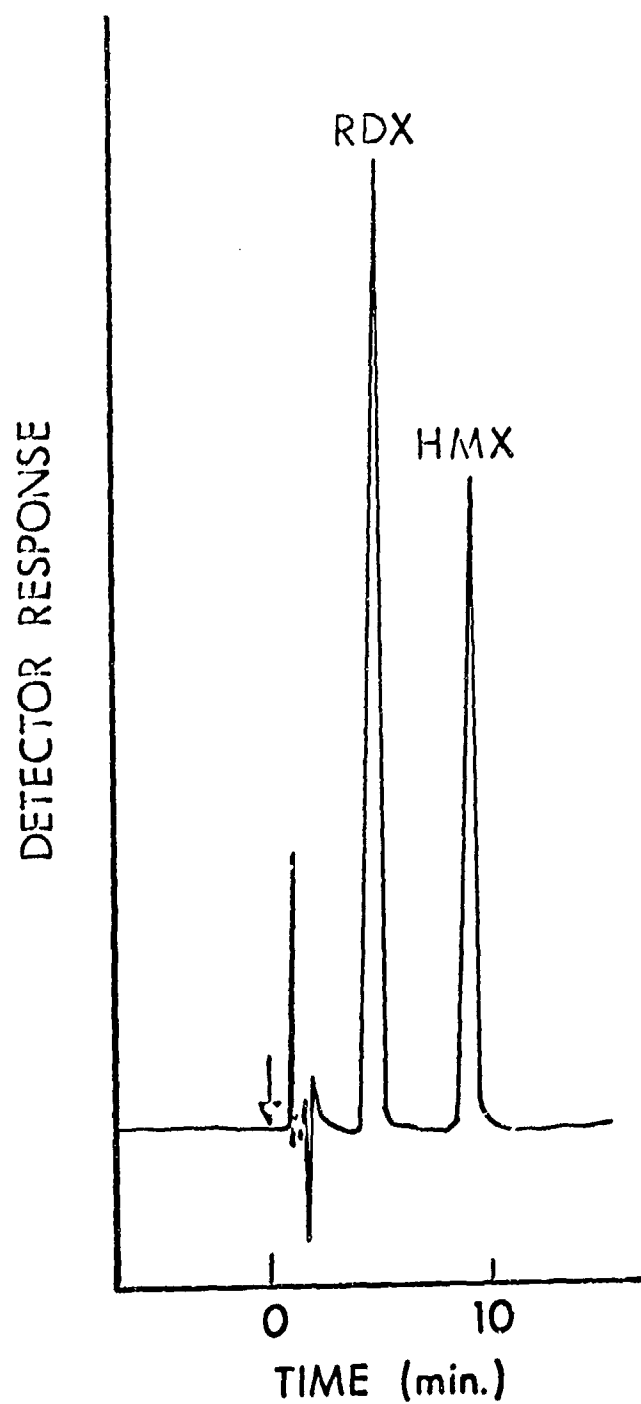


Figure C-1. Separation of HMX and RDX
Column: 250 mm x 2.1 mm i.d. Micro Pak-CN
Mobile Phase: 30% CH₂Cl₂/20% CH₃CN/hexane
Pressure: 2.75 MPa (400 psig). Flow Rate: 0.5 mL/min.

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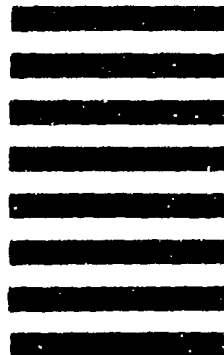


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