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COMPARISON OF THE SENSITIVITIES OF BATCH AND CONTINUOUS PROCESS COMPOSITION B

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NOVEMBER 1978

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Impact sensitivity	Small scale gap test

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Due to variations found in the sensitivity of continuous process Composition B, a series of laboratory tests was conducted to determine the difference between batch process and continuous process Composition B. Four lots were selected from each method of manufacture. RDX was extracted from a sample from each of the lots. The tests on the Composition B and RDX included α-HMX (alpha) analysis, impact sensitivity test, large scale gap test, small scale gap test, projectile impact, friction sensitivity test, electrostatic sensitivity test,

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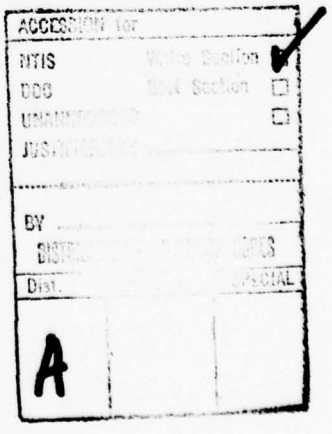
TGA

20. Abstract (Continued)

vacuum thermal stability test, DTA and TGA. The tests results were analyzed and compared to other published data of batch process Composition B and RDX. Based on these results the continuous process Composition B studied herein was considered suitable for military use.

ACKNOWLEDGMENT

The authors wish to acknowledge that the α-HMX analysis was performed by Dr. S. Bulusu and Mr. J. Autera; the large scale and small scale gap tests by Mr. E. Dalrymple; the projectile impact tests by Messrs G. Ziegler and R. Ritchie of the Large Caliber Weapons Systems Laboratory, and the maximum likelihood method analysis by Mr. P. Roediger of the Product Assurance Division, ARRADCOM, Dover, NJ.



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BACKGROUND

Prior to 1969, a batch process was utilized at the Holston Army Ammunition Plant (HAAP), Kingsport, Tennessee, to manufacture RDX (Type II - Acetic Anhydride Process) and Composition B (Comp B). In 1969, as part of the Plant Modernization Program, portions of a batch process line (Line 1 at HAAP) were converted to produce RDX and Comp B by a continuous production method. The prototype batch process line served as a basis for the design and construction of a second generation continuous Comp B incorporation facility.

Among the changes that were incorporated in the continuous line that differed from the batch line were two processing operations. One was the continuous purification and crystallization of RDX, and the other was the precoating of the RDX with the wax desensitizer prior to the addition of the TNT. Figure 1 is a schematic of the continuous process for RDX and Comp B.

Funds to develop and construct this prototype line were furnished by the Office of the Project Manager for Munitions Production Base Modernization and Expansion under the following projects (ref. 1):

Project	Title
2068 (P-15)	Modernization of Nitrolysis Process
4016 (P-16)	Continuous RDX Filtration and Wash
4200 (P-16)	Continuous RDX Recrystallization
4118 (P-16)	Continuous Incorporation Composition B

In 1974-75 HAAP received production orders for large quantities of Comp B. At that time various sections of Line 1 were available but the continuous line was still incomplete. However, in order to meet the production commitments, Holston decided to use the available sections of Line 1, including the continuous RDX recrystallization facility. Subsequently Holston produced, until 15 December 1975, approximately 2.5×10^6 kg of Comp B on this line (ref. 1).

In the strictest sense, the RDX and Comp B produced on this line was prepared without a proper specification since Specifications MIL-R-398C for RDX and MIL-C-401E for Comp B apply only to batch-produced material. It should be noted that both the RDX and Comp B produced on the prototype line met the test requirements given in these specifications.

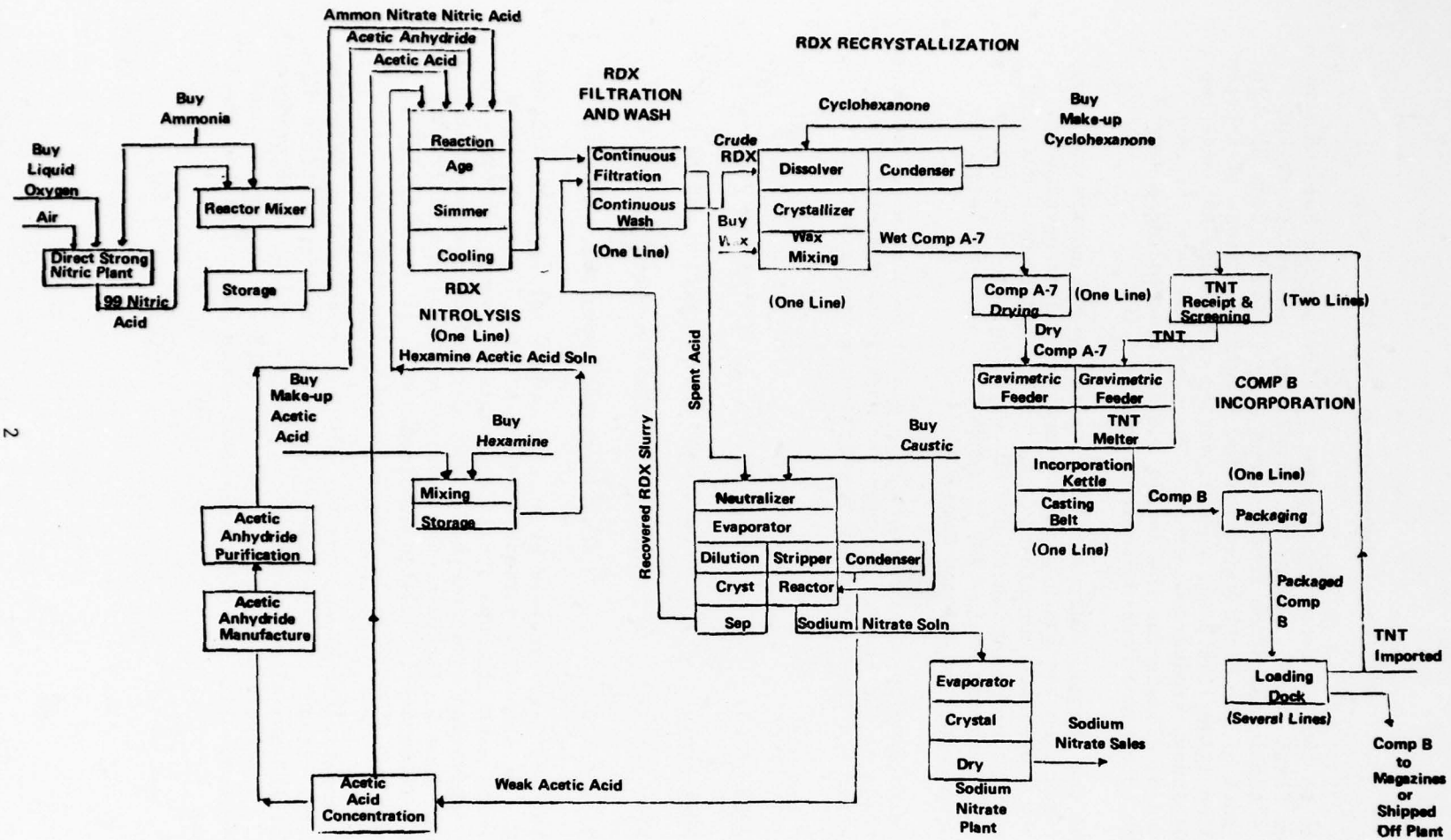

Figure 1. Modern Composition B facilities based on improved Bachman process.

However, problems arose when an increase was noted in the impact sensitivity (lowering of the 50% fire drop height) of RDX lots produced by the continuous recrystallization procedure and subsequently the detection of excessive amounts of α-HMX in that RDX (refs. 1, 2).

INTRODUCTION AND OBJECTIVE

Starting about May 1975, a series of events occurred which culminated with all the Comp B and RDX lots manufactured by the continuous batch procedure being put into a "hold" position. These lots were designated "suspect" until a determination of the suitability of the materials could be made (ref. 2).

These circumstances began in May 1975 with the delivery of a poor quality of cyclohexanone used in the production of RDX. The RDX produced was discolored and, when tested for impact sensitivity, produced a 50% value ranging from 8 to 10 cm as compared to the usual value of 33 cm. Subsequently tighter controls upgraded the quality of cyclohexanone purchased and all batches of RDX manufactured from the poor quality cyclohexanone were destroyed.

However, in June 1975, during the investigation of the poor quality cyclohexanone, HAAP reported that excessive amounts of α-HMX were present in the RDX produced by the continuous recrystallization process. Normally, batch-produced RDX (Type II) contains 5-15% β-HMX which itself has less than 0.1% α-HMX. They also reported the presence of "massive" α-HMX crystals in these RDX lots. Usually α-HMX crystals are needle-shaped and about 10 to 20 μ long and 1 to 2 μ wide. The massive α-HMX crystals were reported to be about 100 to 200 μ long and 20 to 50 μ wide (ref. 2). The preliminary investigation conducted by HAAP also indicated an apparent increase in the impact sensitivity of these RDX batches. These preliminary results implied that the presence of any α-HMX in β-HMX increased the impact sensitivity of the β-HMX (ref. 2).

These circumstances at HAAP were magnified by a JCAP publication (ref. 1) which reported that end items loaded with Comp B had a frequency of malfunction 4 to 8 times higher than the same end items loaded with TNT.

A literature survey was made on the sensitivities of the HMX polymorphs and their effects on RDX. Conflicting data precluded any definite conclusions on the sensitivity of β-HMX vs α-HMX. There was no information on the effect of α-HMX concentration on the sensitivity of RDX, HMX and Comp B. The lack of proper data, the reported increase in impact sensitivity, and the increase in malfunctions in Comp B-loaded items led to the decision that the use of any Comp B lots containing batches of RDX made in Line 1 be deferred until further data was acquired to determine whether these lots were acceptable.

To achieve this, a test program to evaluate the Comp B lots from the recrystallization process was proposed to US Army Armament Command in January 1976 and funding was approved in May 1977.

This report describes the program and the data generated. The sensitivities of continuous process vs batch process Comp B and RDX were assessed and the suitability of the continuous batch Comp B was evaluated for military use.

EXPERIMENTAL TEST PROGRAM

An investigation of the methods used to conduct the impact sensitivity test at HAAP revealed that the precision and discrimination originally thought to be incorporated in the procedure had deteriorated due to human, instrument, and sampling errors. This imprecision nullified any effort to correlate the impact test results with end item malfunctions (ref. 1). The scope of the program had to be broadened to include tests other than the impact sensitivity test alone in order to properly assess the reported increase in the sensitivity due to the presence of α-HMX.

All of the tests selected are among those required in the qualification of explosives for military use as recommended by the Joint Technical Coordinating Group for Munitions Development, Working Party for Explosives, (ref. 3,4,5) and are listed in table 1.

Four lots of Comp B produced by the batch process and four lots of Comp B produced by the continuous process were selected for this program. The four continuous process Comp B lots were among those reported to contain large amounts of α-HMX. The RDX was obtained by extraction from each of the eight Comp B lots using a method furnished by the Los Alamos Scientific Laboratory (appendix A) to preserve the polymorphic form of the RDX. The lot and batch numbers assigned to the Comp B and RDX used in this program are listed in table 2. In this report all references will be made according to the lot number - be it Comp B or RDX, i.e., Comp B lot 53-097 is the Comp B in that lot while RDX lot 53-097 is the RDX extracted from the Comp B in that lot.

In the test program the RDX was subjected to all the tests except the large scale gap test and the projectile impact. The Comp B was used in all the tests except the small scale gap test.

The α-HMX content in the RDX samples was determined by infrared (IR) spectroscopy. The IR spectra of the eight samples of RDX were run in a KBr matrix. 4.16 mg of each of the RDX samples were mixed

Table 1. Test program to compare batch and continuous process Comp B and RDX.

Test	Test Performance	Number of tests: Batch Comp B	Continuous Comp B	Batch RDX	Continuous RDX	Total No. of tests
1. Extract RDX	Note 1	4	4	-	-	8
2. α-HMX analysis	Note 2	-	-	4	4	8
3. Drop weight impact	a	4	4	4	4	16
4. Large scale gap	b	4	4	-	-	8
5. Small scale gap	c	-	-	4	4	8
6. Projectile impact	d	4	4	-	-	8
7. Friction	e	4	4	4	4	16
8. Electrostatic	Ref 3,4	4	4	4	4	16
9. Thermal vacuum stability	Ref 3,4	4	4	4	4	16
10. Diff. thermal analysis	-	4	4	4	4	16
11. Thermogravimetric analysis	-	4	4	4	4	16
12. Microscopic examination	-	-	-	4	4	3

NOTE 1 - Method to extract RDX received in letter from A. Popolato, LASL, to L.G. Baker, ARRADCOM, 25 May 1977. Method shown in appendix A.

NOTE 2 - α-HMX content determined by infrared analysis of RDX samples.

[a] Test US/Impact/02,03, ref. 5

[b] Test US/Explosive Shock/02, ref. 5

[c] Test US/Explosive Shock/03, ref. 5

[d] Test US/Fragment Impact/01, ref. 5

[e] Test US/Friction/03, ref. 5

Table 2. Lot and batch numbers assigned to Comp B and RDX used in test program.

	Comp B lot number	Comp B batch number	RDX batch number
A.	Batch process samples		
1.	053-97	773794	7RCA-5947
2.	053-99	774177	7RCA-6010
3.	053-5423	370448	3RCA-976
4.	053-5431	370176	3RCA-1036
B.	Continuous process samples		
1.	053-4074	153211	1RCA-292
2.	056-0001	152112	1RCA-30
3.	056-0005	153039	1RCA-243
4.	056-0007	153160	1RCA-267

with 250 mg KBr and pressed into pellets at 18,000 psi. The IR spectra were examined for the characteristic α-HMX band at 710 cm^{-1}.

An examination of the IR spectra reveals that only figures 2 (IR #6 - RDX Lot 056-0001 continuous) and 3 (IR #8 - RDX Lot 056 - 0007 continuous)have a significant amount of α-HMX. These samples contained a total amount of HMX of 17.7% and 12.2%, respectively, in the RDX. The other samples produced spectra as shown in figure 4.

A simple technique using a calibration curve was used to obtain a fairly accurate quantitative estimate of the α-HMX in the IR spectra.

The α-HMX was prepared by a standard method of crystallization from 70% aqueous nitric acid. For calibration purposes, a maximum of 15% total HMX was considered to be in the RDX. Four mixtures made up of 85% RDX and varying amounts of α- and β-HMX were prepared:

	% Composition	
RDX	β-HMX	α-HMX
85	13	2
85	9	6
85	5	10
85	0	15

IR spectra of these samples were obtained and the α-HMX peak at 710 cm^{-1} was measured from the minimum on the low frequency side and plotted against the α-HMX concentration in the mixture as shown in figure 5. This method of obtaining the concentration versus IR absorption can approximate the amount of α-HMX with an accuracy about 2%. Because of the inherent uncertainty in measuring peak heights, the absorbance versus concentration curve would not have assured any better accuracy (ref. 6).

The α-HMX peak heights in figures 2 and 3 when measured against the calibration curve in figure 5 indicated that 17-18% and 2-3% α-HMX, respectively, were present in those two RDX lots. This was checked by the fact that the total amount of HMX in figure 2 (IR #6), as estimated by the ethylene dichloride extraction, was about 13%. Comparing this value to the 17-18% α-HMX found by IR, the

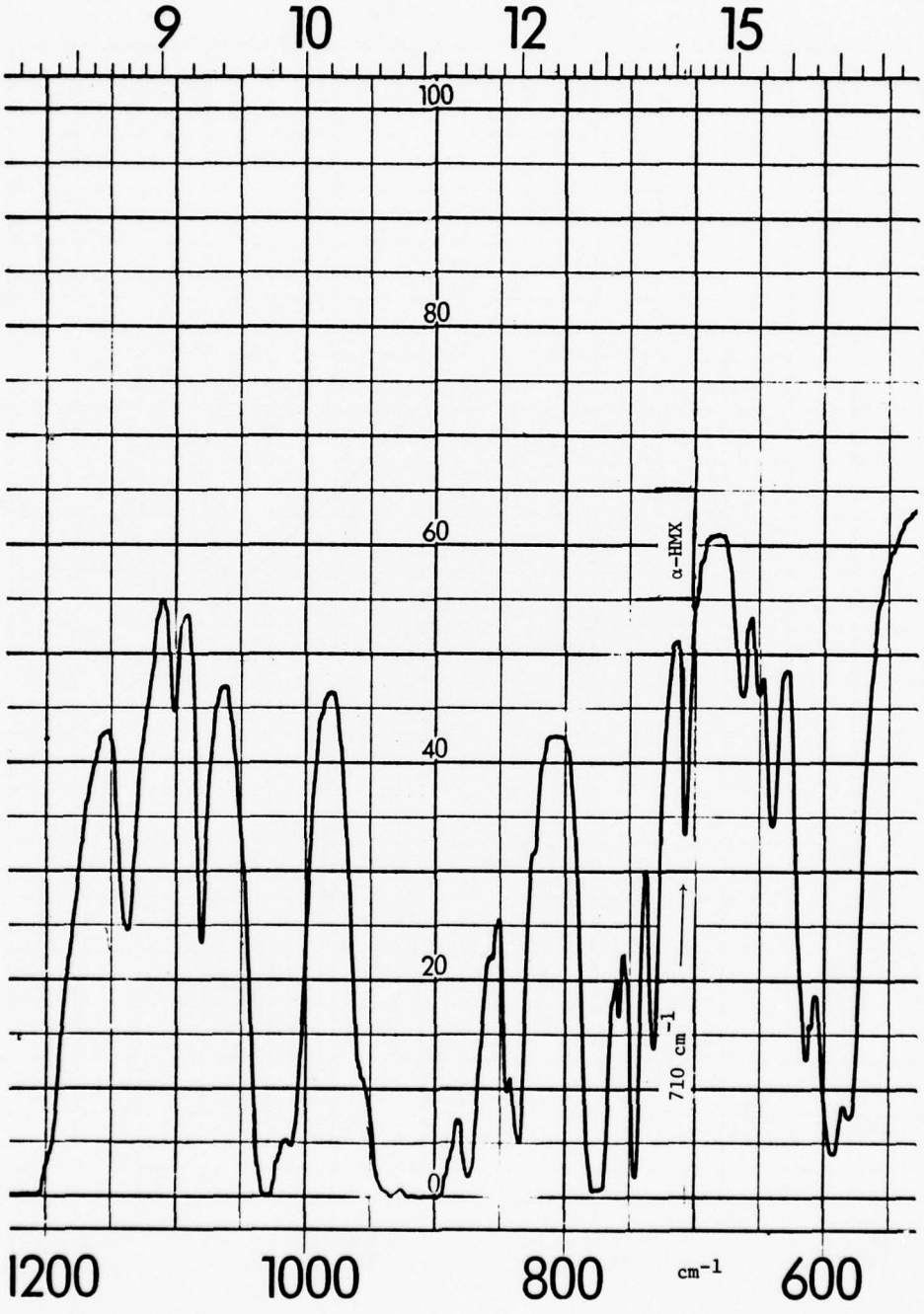

Figure 2. IR #6 (RDX 056-0001, continuous).

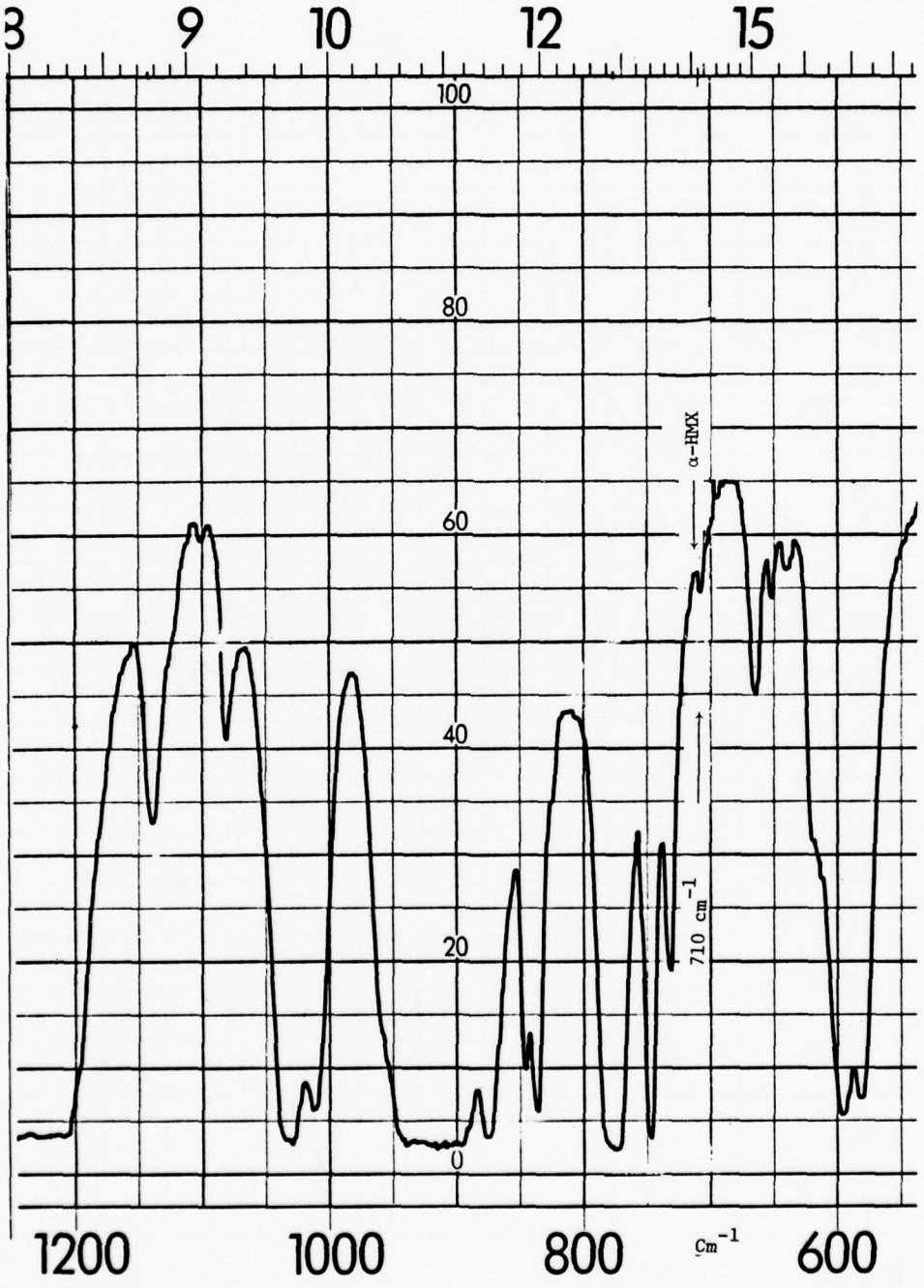

Figure 3. IR #8 (RDX Lot 056-0007, continuous).

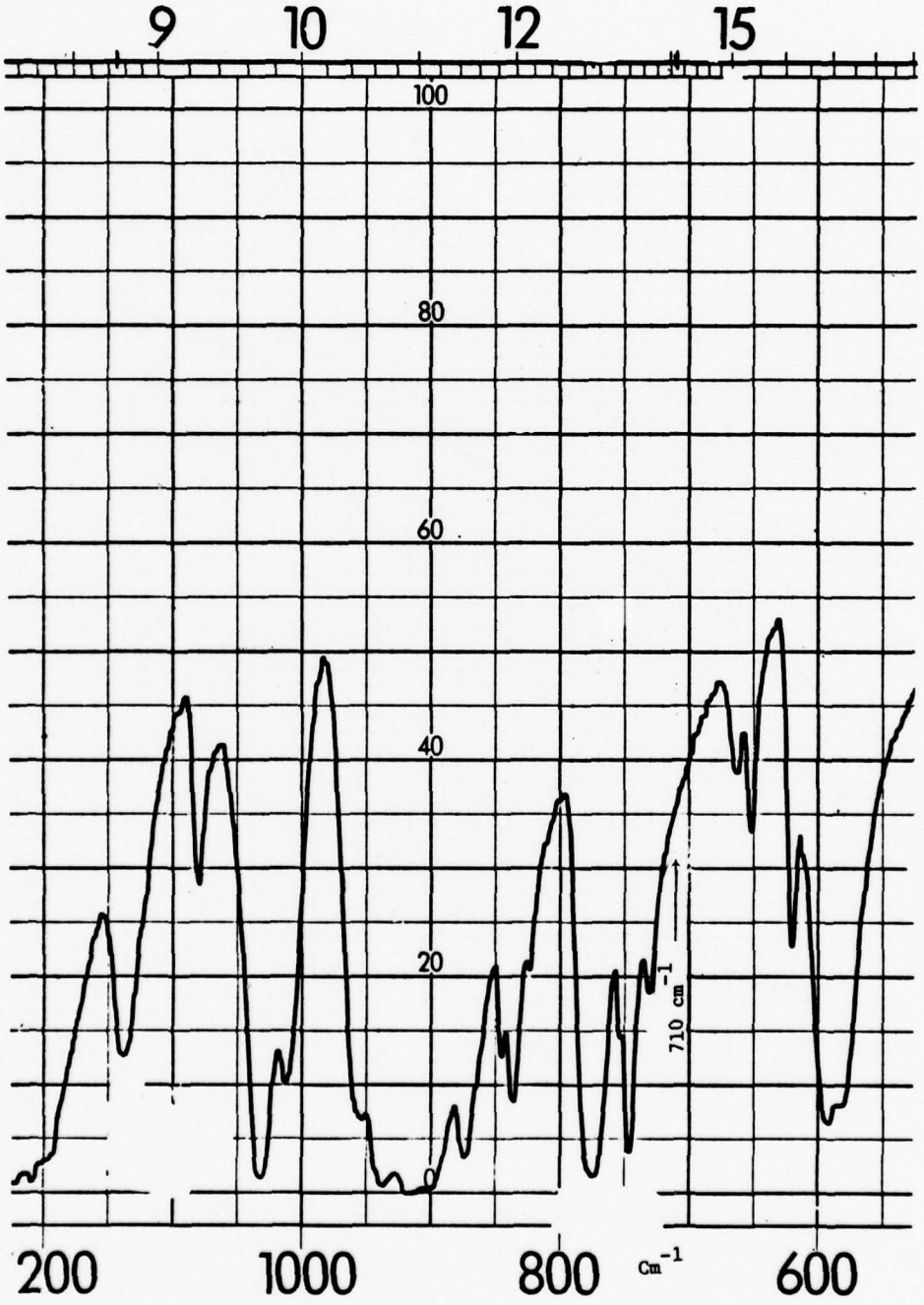

Figure 4. IR #1 (RDX Lot 053-97, batch).

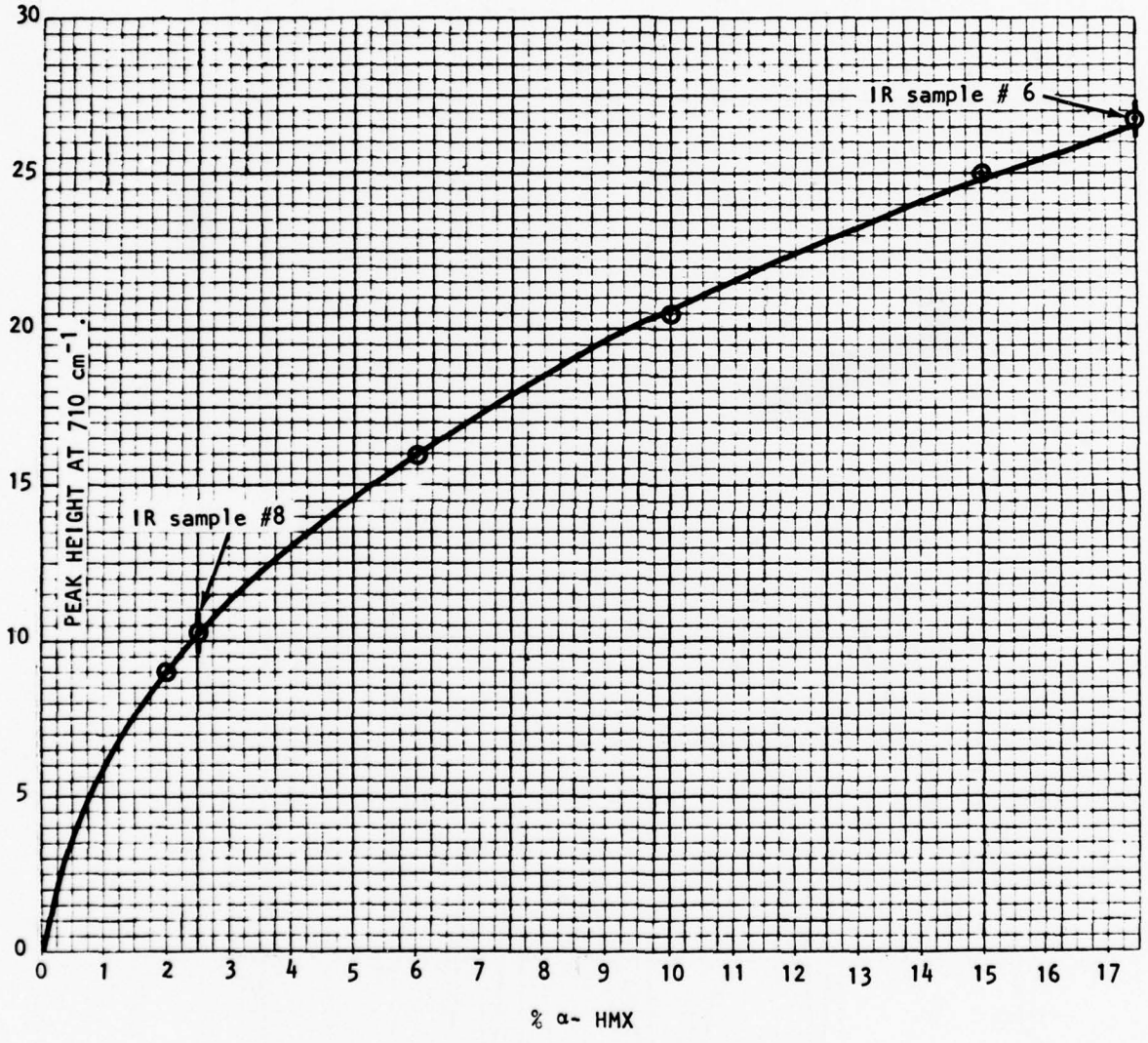

Figure 5. α-HMX peak height vs % α-HMX concentration.

accuracy of the method can be questioned. Therefore, for practical purposes RDX Lot 056-0001 should be considered as containing essentially 12.5 - 17% α-HMX, while RDX Lot 056-0007 contains 2-3% α-HMX in a total of about 17% HMX. Since the lower detection limit of this method is about 2%, the rest of the samples probably contain about 1% α-HMX, if any.

RESULTS

The results of the test program outlined in table 1 are described in the following paragraphs.

1. Drop Weight Impact

The impact tests (refs. 3,4,5) were conducted on the NOL impact tester with Type 12 tools, sandpaper, and a 2-1/2 kilogram drop weight. The Comp B and RDX samples were tested at 68°F and 55% R.H. The results are listed in table 3. The 50% fire point was obtained by the Bruceton up-and-down method. In some instances the tests were re-run if the 50% height was higher or lower than the average and/or the standard deviation seemed large.

2. Large Scale Gap Test

The large scale gap test (refs.3,4,5) was used to determine the response of the Comp B lots to shock and the small scale gap test was used to evaluate the shock response of RDX. The gap test can be used to predict the hazard of sympathetic detonation of one explosive when exposed to a shock wave generated by a second explosive.

The results obtained on the large scale gap test are listed in table 4. Samples from each Comp B lot were cast into pipe sections and radiographically examined for voids and variations in density. Ten acceptable samples from each lot were tested. The average density of the cast samples was 1.67 $\pm$ .01 g/cc.

The tests were begun using a 15.0 x 15.0 x .31 cm steel witness plate but midway through the tests the supply was exhausted and could not readily be replenished. Since a ready supply of 10.0 x 10.0 x .31 cm plates was available, the tests were re-run. All of the results with both plates are shown in table 4. Since the definition of a "go" is the presence of a hole in the witness plate after the test, the change from one plate to the other was not considered significant. The results indicate that the difference in the 50% points (in centimeters) for the two witness plates is not significant.

Table 3. Impact test results on Comp B and RDX lots - batch and continuous process.

Lot number	Comp B		RDX	
	50% point (cm)	σ (cm)	50% point (cm)	σ (cm)
A. Batch process				
1. 053-97	45.50	1.94	37.83	4.34
			32.00	1.80
2. 053-99	42.58	1.25	32.22	1.84
3. 053-5423	48.00	2.07	34.33	3.78
	45.58	2.06	38.85	3.07
4. 053-5431	42.50	4.31	32.95	4.57
	48.86	1.89		
	49.14	3.96		
B. Continuous process				
1. 053-4074	40.42	0.26		
	46.70	5.17	31.14	2.19
2. 056-0001	45.75	3.59	31.20	2.89
3. 056-0005	48.40	4.30	31.58	2.06
	43.41	1.08	32.95	2.80
4. 056-0007	47.08	1.79	40.13	7.43
	40.83	0.95	34.50	1.94
	46.59	2.54		

Table 4. Large scale gap test results on batch and continuous process Comp B lots.

Explosive lot	50% points: 6"x6" (15x15 cm) plate		4"x4" (10x10 cm) plate		Average	
	Inch	(cm)	Inch	(cm)	Inch	(cm)
A. Batch process						
1. 053-97	2.23	(5.66)	2.235	(5.68)	2.232	(5.67)
2. 053-99	-		2.155	(5.47)	2.155	(5.67)
3. 053-5423	2.22	(5.64)	2.22	(5.64)	2.22	(5.64)
4. 053-5431	2.185	(5.55)	2.205	(5.60)	2.195	(5.58)
Avg.	2.213	(5.62)	2.204	(5.60)	2.202	(5.59)
B. Continuous process						
1. 053-4074	-		2.13	(5.41)	2.13	(5.41)
2. 056-0001	2.13*	(5.41)	2.13	(5.41)	2.13	(5.41)
3. 056-0005	2.24	(5.69)	2.275	(5.79)	2.257	(5.73)
4. 056-0007	-		2.26	(5.74)	2.26	(5.74)
Avg.	2.19	(5.55)	2.199	(5.60)	2.195	(5.57)

*Test not completed due to lack of 6"x6"(15x15 cm) plates.

For comparison purposes typical test results obtained by NOL are as follows: TNT has a 50% point of 4.65 cm (1.83 in.) with a pressed density of 1.60 g/cc, Comp B value 6.05 cm (2.38 in.) with a pressed density of 1.66 g/cc, and RDX 8.20 cm (3.23 in.) with a pressed density of 1.64 g/cc.

3. Small Scale Gap Test

The small scale gap test (ref. 5) was conducted on the RDX extracted from the batch and continuous process Comp B lots. Twenty donors and twenty acceptors were loaded and pressed according to the procedure. The zero-gap dent value for all the RDX lots ranged from .190 cm to .208 cm (0.076 in. to 0.083 in.). In each lot the criterion for assessing each shot is set at 50% zero-gap dent. Dent readings below this value ($\sim$ .100 cm (.040 in.)) are recorded as a no-fire, and greater than this value as a fire. The 50% fire point is recorded in table 5 in inches as well as gap decibangs. This value, which is analgous to the decibel used in acoustics, is obtained from the following equation:

$$X = 30 - 10 \log GT$$

where X = initiation intensity in gap decibangs and

GT = observed gap in mils.

For several explosives typical test results have been obtained on the small scale gap test (ref. 5). With the density of each explosive at 92% TMD (theoretical maximum density) the values in gap decibangs are: TNT - 60, RDX - 4.35, and HMX - 3.9.

The results in table 5 indicate that according to the small scale gap test, the RDX extracted from the batch process Comp B is more sensitive than the RDX from the continuous process Comp B. The gap decibang values for the continuous process RDX agree fairly well with the literature value of 4.35.

4. Projectile Impact

The .50 caliber projectile impact sensitivity test (ref. 5). as developed by the Bureau of Mines, was used in this program with some slight modifications to the test. Brass right cylinders, 1.27 cm by 1.27 cm (1/2 in. by 1/2 in.) are fired in a .50 caliber smooth bore gun. The desired projectile velocity is obtained by adjusting the propellant charge. With the weight of the propellant calibrated, the velocity is measured with a 10 megacycle counter chronograph. The start and stop signals are light beams spaced 1/2 meter apart

Table 5. Small scale gap test results on RDX extracted from Comp B lots.

Lot	Average pressed density gm/cm^3	50% Point Inch	50% Point (cm)	Gap decibangs
A. Batch Process				
1. 053-97	1.656	0.406	(1.03)	3.91
2. 053-99	1.655	0.433	(1.10)	3.64
3. 053-5423	1.646	0.423	(1.07)	3.74
4. 053-5431	1.642	0.422	(1.07)	3.75
Average	1.650	0.421	(1.07)	3.76
B. Continuous Process				
1. 053-4074	1.670	0.372	(0.94)	4.29
2. 056-0001	1.673	0.390	(0.99)	4.09
3. 056-0005	1.671	0.377	(0.96)	4.24
4. 056-0007	1.653	0.351	(0.89)	4.55
Average	1.668	0.373	(0.95)	4.29

between the gun and the sample. The measured velocity is a linear function of the square root of the propellant charge.

Only the Comp B lots were so tested. With cast explosives the target samples are 2.54 cm (1 in.) diameter right cylinders, 5.08 cm (2 in.) in height and placed so that the brass projectile strikes a flat end surface. Sound and examination of the debris in the test chamber determine the "go" or "no go" of the test.

The sensitivity of the explosive is expressed as the projectile velocity which produces an initiation in 50% of the trials. The Bruceton up-down technique is used to estimate the 50% point by varying the square root of the propellant weight. These values are listed in table 6. Included in that table are the maximum velocity for a "no-go" and a minimum velocity for a "go".

The batch process Comp B lots produced Bruceton 50% velocity points that were consistent with each other within a range of 30.5 m/sec (100 ft/sec). With the continuous batch Comp B lots the velocity range was about 221.0 m/sec (725 ft/sec) from 876.3 to 1097.2 m/sec (2875 to 3600 ft/sec). A decision was made to retest the continuous process Comp B lots with the high and low velocities (lots 053-4074 and 056-0007). The 50% Bruceton velocities obtained were 1059.1 and 902.2 m/sec (3475 and 2960 ft/sec) respectively.

The projectile impact data also was subjected to an analytical technique in which the data is fitted to a normal or Weibull distribution. Also a determination can be made by the technique as to which distribution has the maximum likelihood (ref. 7) to give the best possible result. The mean values (velocities) for each type of distribution are listed for each lot in table 6. Also the distribution with the maximum likelihood is indicated.

This method was not applicable to the results for two of the lots - batch process Comp B lot 053-99 and continuous process Comp B lot 056-0005. In each instance no overlap occurred - a "no-go" value was not a higher velocity for a "go". For lot 053-99 the highest "no-go" was 966.2 m/sec (3170 ft/sec) while the lowest "go" was 975.3 m/sec (3200 ft/sec). For lot 056-0005 the highest "no-go" was 874.7 m/sec (2870 ft/sec) and the lowest "go" was 883.8 m/sec (2900 ft/sec).

Results from previous projectile impact tests on Comp B and TNT indicate that all the values obtained are comparable. For Comp B - the density was not given - the highest "no-go" was 845.8 m/sec (2775 ft/sec) while the lowest "go" was 899.1 m/sec (2950 ft/sec). For TNT the highest "no-go" was 1059.4 m/sec (3476 ft/sec) while the lowest "go" was 1116.4 m/sec (3663 ft/sec).

Table 6. Projectile impact test results on batch and continuous process Comp B lots.

Comp B lot	Average density g/cc	Projectile impact data: Max. velocity for no-go ft/sec (m/sec)	Min. velocity for go ft/sec (m/sec)	Est. Bruceton 50% ft/sec (m/sec)	Normal distribution value ft/sec (m/sec)	Weibull distribution value ft/sec (m/sec)	Max likelihood
A. Batch process							
1. 053-97	1.662	3180 (969.2)	2975 (906.7)	3075 (937.2)	3092.59 ± 181.20 (942.58 ± 55.22)	3076.10 ± 176.25 (937.55 ± 53.72)	Weibull
2. 053-99	1.665	3170 (966.2)	3200 (975.3)	3175 (967.7)	No overlap		
3. 053-5423	1.665	3200 (975.3)	3100 (944.8)	3125 (952.5)	3143.36 ± 76.90 (958.05 ± 23.44)	3136.24 ± 86.64 (955.88 ± 26.41)	Normal
4. 053-5431	1.669	3170 (966.2)	3100 (944.8)	3175 (967.7)	3147.02 ± 45.20 (959.16 ± 13.78)	3149.53 ± 41.81 (959.93 ± 12.74)	Weibull
			Average	3137.5 (956.3)	3123.0	(951.8)	
B. Continuous process							
1. 053-4074	1.675	3720 (1133.8)	3510 (1069.8)	3600 (1097.2)	3568.05 ± 204.12 (1087.49 ± 62.21)	3553.92 ± 213.53 (1083.18 ± 65.08)	Normal
				3500 (1066.7)			
		3520 (1072.8)	3410 (1039.3)	3475 (1059.1)	3441.05 ± 72.46 (1048.78 ± 22.08)	3433.72 ± 89.88 (1046.55 ± 27.24)	Normal
2. 056-0001	1.679	3180 (969.2)	3110 (947.9)	3150 (960.1)	3153.23 ± 94.73 (961.06 ± 28.87)	3148.54 ± 107.30 (959.63 ± 32.70)	Weibull
3. 056-0005	1.665	2870 (874.7)	2900 (883.9)	2900 (883.9)	No overlap		
4. 056-0007	1.675	2975 (906.7)	2865 (873.2)	2875 (876.3)	2903.63 ± 75.31 (884.99 ± 22.95)	2894.95 ± 92.36 (882.34 ± 28.33)	Normal
				2925 (891.5)			
		3000 (914.4)	2935 (894.5)	2960 (902.2)	2955.47 ± 42.65 (900.78 ± 13.00)	2954.57 ± 46.05 (900.51 ± 14.04)	Weibull
			Average	3118 (950.3)	3194.06	(973.5)	

5. Friction Sensitivity

With the large Picatinny Arsenal friction pendulum (ref. 5), a steel shoe is calibrated to swing over an anvil upon which the explosive sample is placed. The number of swings is calibrated to be 18 $\pm$ 1 before coming to rest. A test consists of ten trials with the steel shoe, except when complete explosion or burning occurs in any trial. The reactions that may occur are designated crackles, sparks, burn and detonation. If burning or detonation occurs, the trials with the steel shoe are discontinued and the steel shoe is replaced with a fiber shoe. If no reactions occur with the steel shoe in ten trials then the explosive sample has passed the friction test. An explosive is also considered to pass the test if, in ten trials with the fiber shoe, there is no more than an almost inaudible local crackling regardless of its behavior when subjected to the action of the steel shoe.

With the RDX samples, a detonation occurred with a batch (RDX 053-5431) and a continuous process (RDX 056-0005) RDX lot. The rest of the RDX samples all produced crackles with the steel shoe. With the fiber shoe all the RDX samples exhibited no reactions.

With the Comp B samples, one batch lot (Comp B 053-99) and one continuous process lot (Comp B 053-4074) passed the steel shoe test. The rest of the samples indicated only crackles, but the tests were switched to the fiber shoe. In this configuration these lots passed the friction test.

Based on the results, all of the batch and continuous process Comp B and RDX samples passed the friction sensitivity test with either the steel or fiber shoe configuration.

6. Electrostatic Sensitivity Test

The electrostatic sensitivity test (ref. 3,4) is designed to discharge energy from a needle electrode through a thin layer ($\sim$50 mg) of explosive to a grounded conductive surface. All 16 samples -- four batch and four continuous process Comp B lots, and four batch and four continuous RDX samples extracted from the Comp B lots -- were subjected to the test. For each sample, 20 consecutive tests were conducted at the 0.25 joule level and no fires were recorded. This is with a .02 microfarad capacitor and a voltage of 5000 VDC.

7. Vacuum Thermal Stability Test

The vacuum thermal stability test (VTS) was conducted at 100°C (ref. 3,4) with a five-gram sample for 48 hours and the amount of gas

evolved for each sample is listed in table 7. The qualification criteria for any explosive to be sufficiently stable for military storage and use is that the VTS value must not be larger than 2.0 ml/gm/48 hrs. All the samples passed the VTS tests and showed no significant variation.

8. Differential Thermal Analysis

The differential thermal analysis (DTA) studies were performed using a duPont 900 Differential Thermal Analyzer at a heating rate of 20°C/minute in a nitrogen atmosphere. The onset and peak values of the endotherms and exotherms were recorded. The results are listed in table 8 and representative DTA thermograms for the Comp B and RDX samples are illustrated in figures 6 and 7.

The DTA curve is dependent on two general types of variables: (a) instrumental factors, and (b) sample characteristics. The instrumental factors are based on the instrument geometry including the heating rate, while the sample characteristics include particle size, packing density, and heating and swelling of the sample.

The samples were subjected to the DTA test as received. A review of the results does not indicate any significant difference between the batch and continuous process Comp B lots or between the batch and continuous process RDX from those Comp B lots.

9. Thermogravimetric Analysis

The change in mass as a function of temperature was obtained for each of the samples by the thermogravimetric analysis (TGA) technique. The volatization of a substance can be followed by the standard non-isothermal thermogravimetric method. By this procedure, decomposition which results in gaseous products is detected, and a quantitative measure of the amount and rate of decomposition at each temperature can be determined. The TGA thermograms are dependent on the same factors as the DTA but are sufficiently reproducible to indicate the temperature-stability ranges of the explosive materials.

The TGA studies were performed with the duPont 950 Thermal Gravimetric Analyzer (TGA), an attachment to the duPont 900 DTA. A 20°C/min heating rate was used, and the temperature at which a 10% weight loss occurred was recorded. The results are listed in table 9 and representative TGA thermograms for Comp B and RDX are depicted in figures 8 and 9.

A review of the results of the TGA thermograms reveals that no significant differences were evident between the batch and continuous process materials - both the Comp B and RDX.

Table 7. Results of 100°C vacuum stability tests conducted on batch and continuous process Comp B and RDX.

5 grams/48 hrs

Explosive lot		Volume of gas evolved - ml
I. Comp B lots		
A. Batch Process		
1. 053-97		0.42
2. 053-99		0.44
3. 053-5423		0.45
4. 053-5431		0.50
	Average	0.45
B. Continuous Process		
1. 053-4074		0.50
2. 056-0001		0.54
3. 056-0005		0.51
4. 056-0007		0.51
	Average	0.52
II. RDX (extracted)		
A. Batch Process		
1. 053-97		0.37
2. 053-99		0.39
3. 053-5423		0.38
4. 053-5431		0.37
	Average	0.38
B. Continuous Process		
1. 053-4074		0.42
2. 056-0001		0.39
3. 056-0005		0.43
4. 056-0007		0.42
	Average	0.42

Table 8. DTA results on batch and continuous process Comp B and RDX.

Heating rate: 20°C/min

Explosive lots	Endotherm Onset - °C	Endotherm Peak - °C	Exotherm Onset - °C	Exotherm Peak - °C	Remarks
I. Comp B					
A. Batch process					
1. 053-97	73	77	196	249	
2. 053-99	73	77	192	254	
3. 053-5423	72	78	190	254	
4. 053-5431	74	77	193	247	
B. Continuous process					
1. 053-4074	72	79	187	247	
2. 056-0001	73	78	190	252	
3. 056-0005	68	78	190	248	Small change on endotherm @ 77°C
4. 056-0007	83	78	193	254	
II. RDX					
A. Batch process					
1. 053-97	183	198	205	244	Small endotherm @ 190°C
2. 053-99	183	200	206	240	
3. 053-5423	183	199	207	248	Small endotherm @ 193°C
4. 053-5431	177	197	205	245	Small endotherm @ 191°C

Table 8 (Continued)

Heating rate: 20°C/min

Explosive lots	Endotherm Onset - °C	Endotherm Peak - °C	Exotherm Onset - °C	Exotherm Peak - °C	Remarks
B. Continuous process					
1. 053-4074	180	200	207	250	Small endotherm @ 193-197°C
	177	201	215	249	
2. 056-0001	186	198	206	253	Small endotherm @ 190-195°C
3. 056-0005	180	198	205	251	Small endotherm @ 193°C
	183	197	205	250	Small endotherm @ 193°C
4. 056-0007	173	200	209	251	
	183	199	210	257	

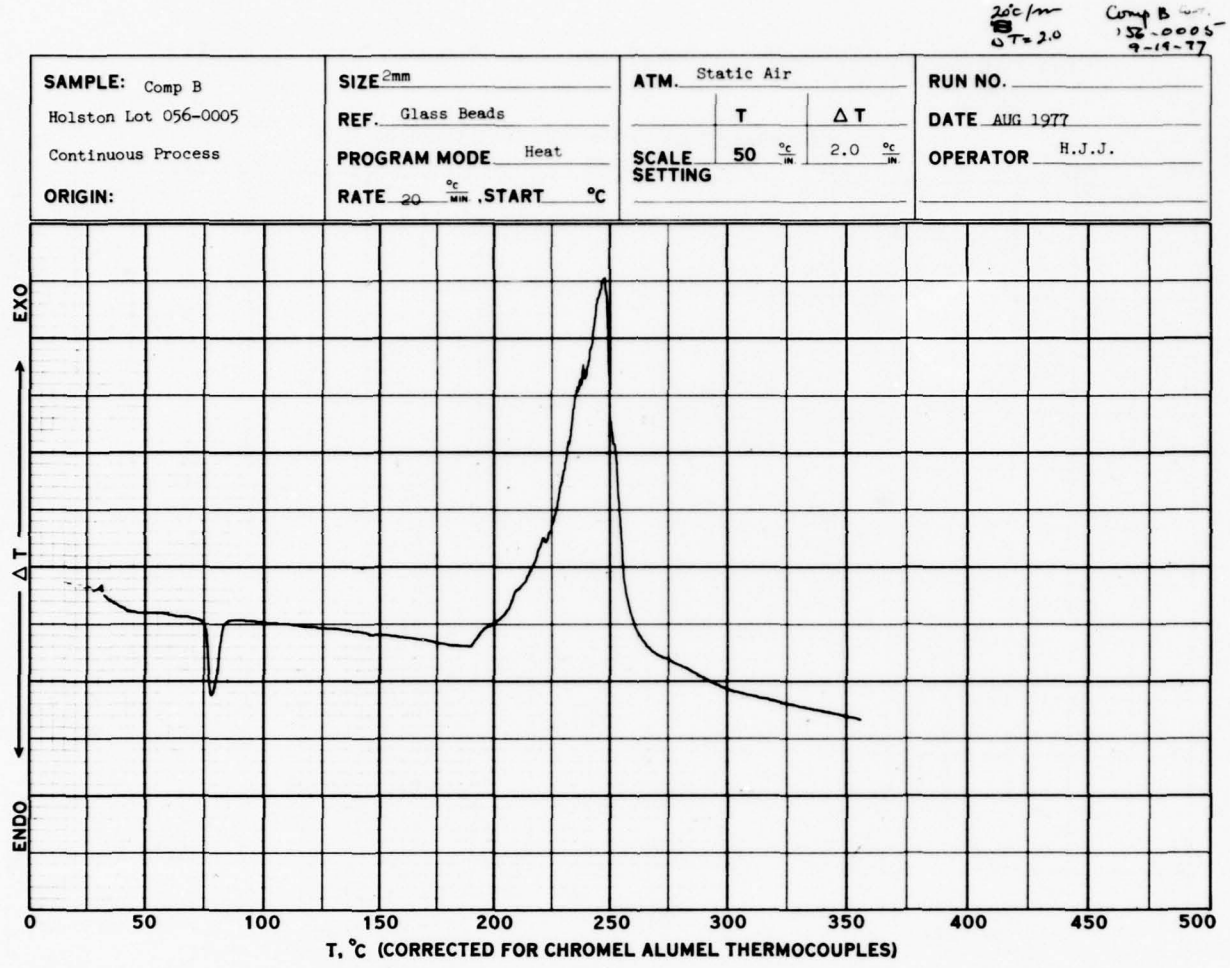

Figure 6. DTA thermogram - Comp B Lot 056-0005 continuous process.

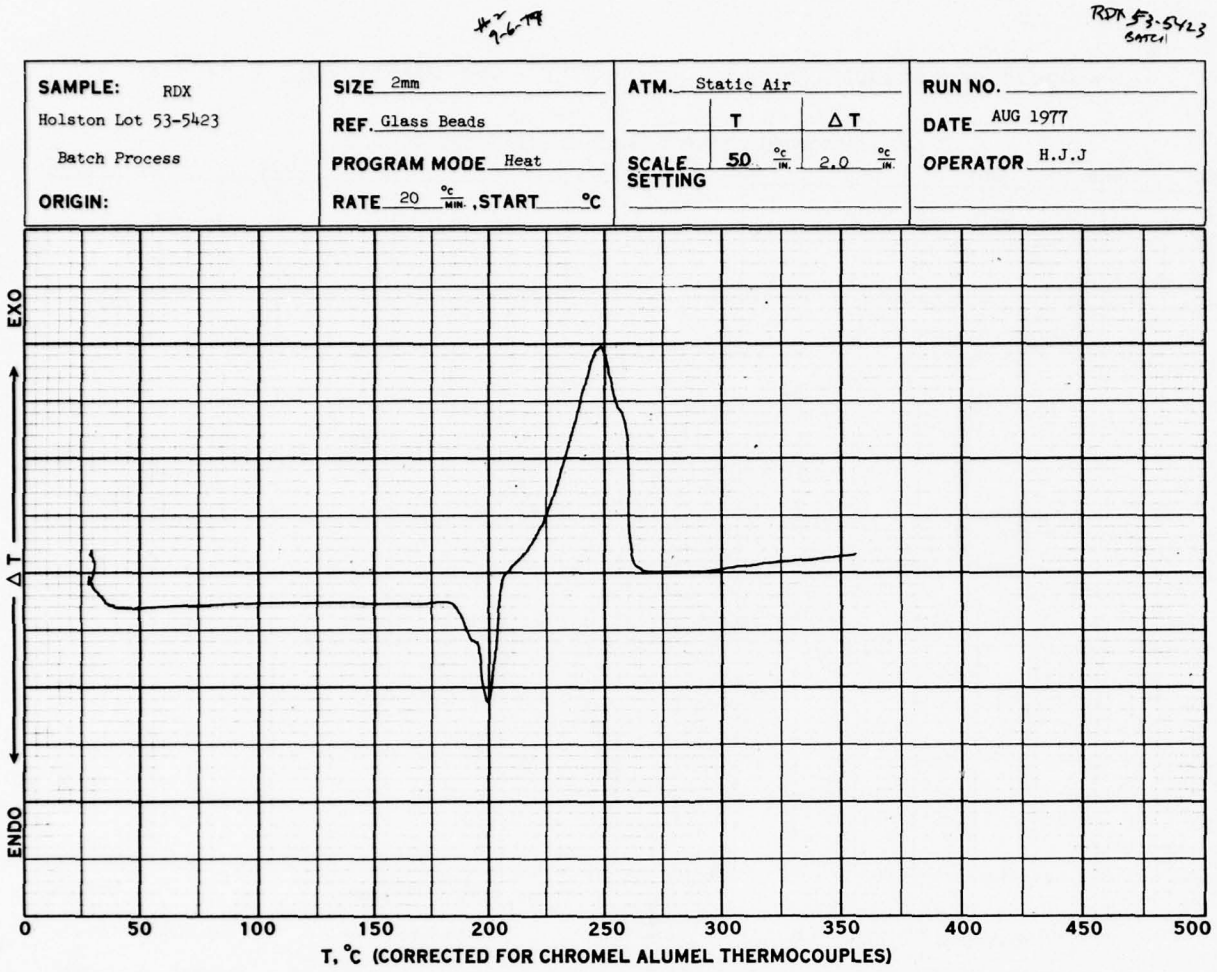

Figure 7. DTA thermogram - RDX from Lot 053-5423 batch process.

Table 9. TGA results on batch and continuous process Comp B and RDX lots.

Heating rate: 20°C/min

Explosive lots	Weight mg	Start of decomposition - °C	10% weight loss temp. - °C	Deflagration
I. Comp B				
A. Batch process				
1. 053-97	8.85	140	208	@ 240°C
2. 053-99	7.9	157	218	@ 233°C
3. 053-5423	7.2	140	203	@ 228°C
4. 053-5431	8.2	145	208	@ 220°C
B. Continuous process				
1. 053-4074	9.6	140	210	@ 220°C
2. 056-0001	8.6	140	208	@ 222°C
3. 056-0005	8.0	155	218	@ 235°C
4. 056-0007	8.2	130	217	@ 237°C
II. RDX				
A. Batch process				
1. 053-97	6.8	185	233	@ 240°C
2. 053-99	7.4	185	216	@ 217°C
3. 053-5423	6.4	185	218	@ 224°C
4. 053-5431	5.4	200	238	@ 243°C
B. Continuous process				
1. 053-4074	7.8	180	217	@ 220°C
	6.8	180	220	@ 226°C
2. 056-0001	9.2	195	211	@ 215°C
3. 056-0005	7.75	180	218	@ 225°C
4. 056-0007	7.75	175	215	@ 215°C

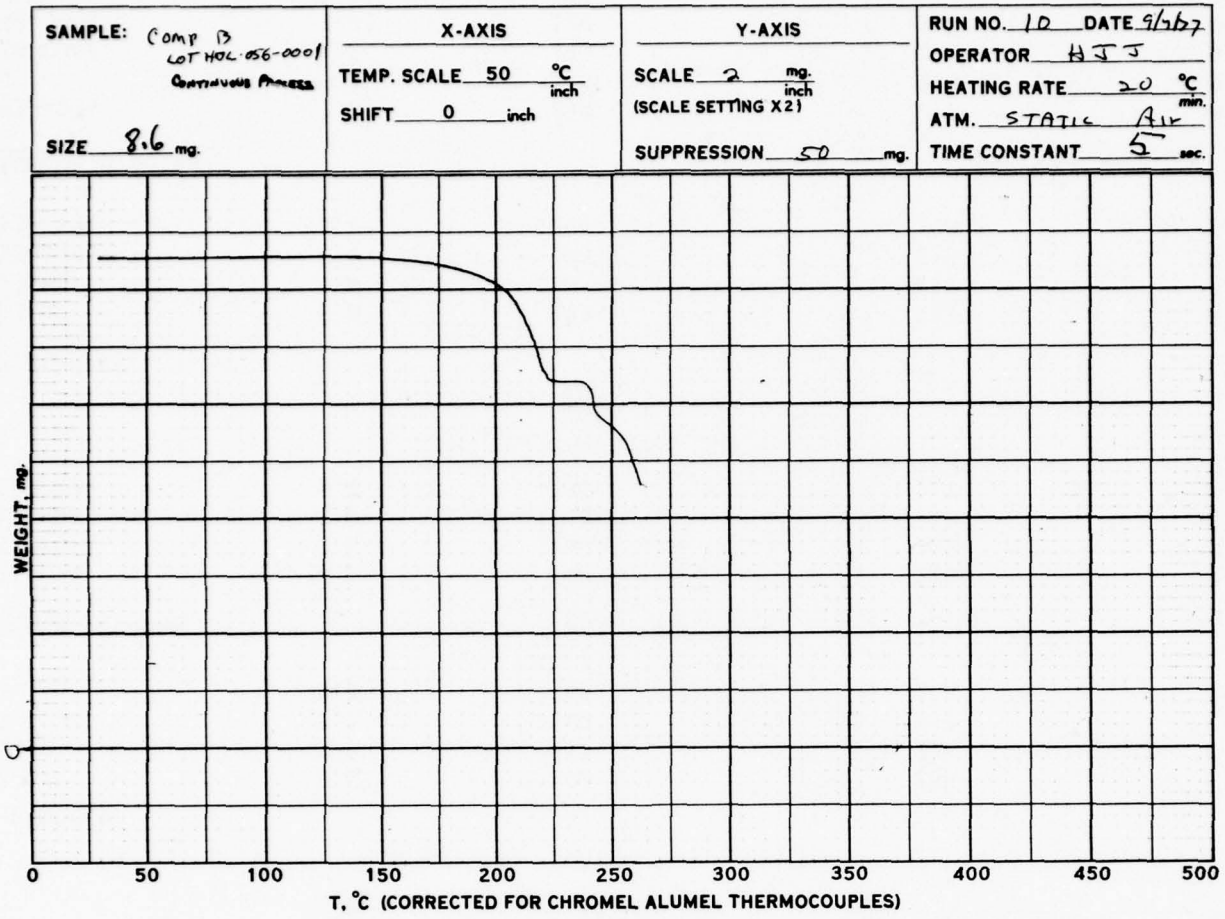

Figure 8. TGA thermogram - Comp B Lot 056-0001 continuous process.

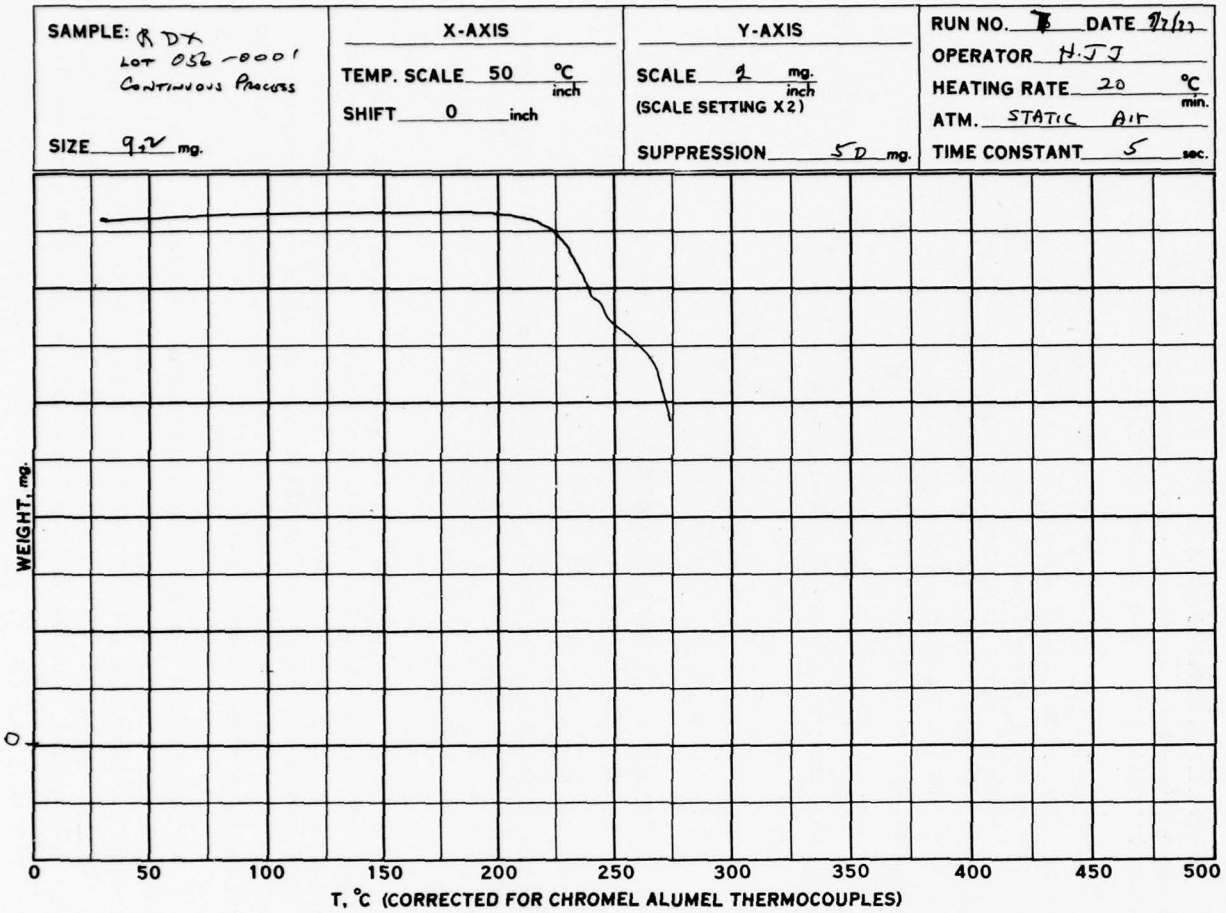

Figure 9. TGA thermogram - RDX from Lot 056-0001 continuous process.

10. Microscopic Examination

The purpose of the microscopic examination was to confirm the size and shape of the α-HMX crystals in the RDX manufactured by the continuous process. The RDX samples were obtained from the same sources that were used to analyze the α-HMX by the IR method.

Several photomicrographs (30 X) were taken of each lot. The photomicrographs shown in this report are representative of each RDX continuous process lot. The visual findings were as follows:

Figures 10, 11: Lot 053-4074 consists mostly of large crystals of RDX. The larger crystals of RDX can be seen in figure 10 while the smaller particles are in figure 11. This was taken to see if any needles, which indicate the presence of α-HMX, were present. There are few, if any, α-HMX needle crystals present. Also it should be noted that broken fragments of large RDX crystals may be included among the smaller needle-like particles.

Figure 12: Lot 056-0001 shows α-HMX needle-like crystals as well as the larger, more equidimensional crystals of RDX. The amount is significant.

Figure 13: Lot 056-0005 does not show any α-HMX needle crystals.

Figure 14: Lot 056-0007 shows that some α-HMX needle-like crystals are present but not in the amount revealed in Lot 056-0001.

As expected, the photomicrographs are in agreement with the IR spectra shown in figures 2 and 3 for the same RDX lots. Figure 13 confirms with figure 2 that the largest amount of α-HMX was present in RDX continuous process Lot 056-0001.

However, the presence of "massive" α-HMX crystals (100-200 μ long and 20-50 μ wide) was not confirmed. Although one of the reasons for the selection of these particular continuous process Comp B lots had been based on the detection of such crystals, no "massive" α-HMX crystals were found in any of the RDX extracted from the continuous process Comp B lots.

An additional visual examination was conducted. Castings of each batch and continuous process Comp B lot were made. One surface on each casting was sanded and then etched with ethyl alcohol to enhance the RDX crystals. The only obvious difference between the batch and continuous process Comp B lots was in the differences in

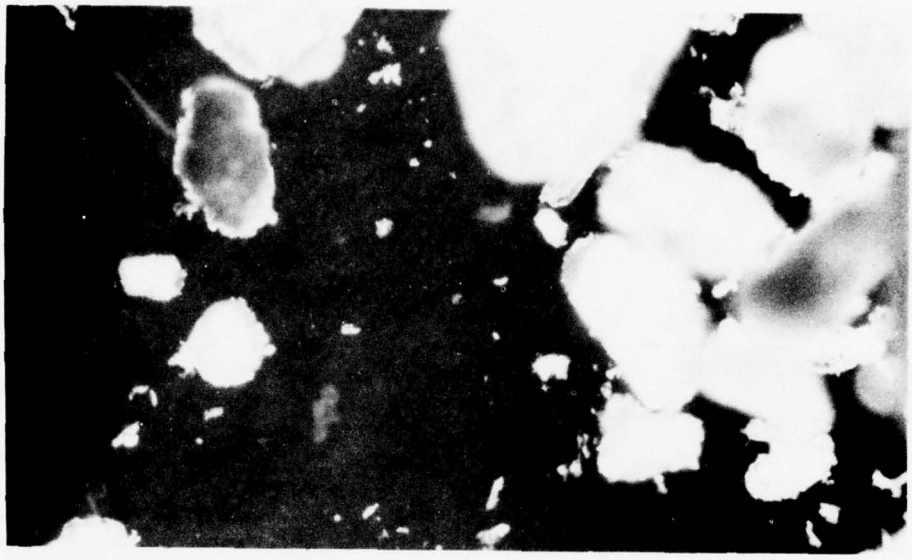

Figure 10. Photomicrograph of continuous process RDX Lot 053-4074 (30X) (large crystals).

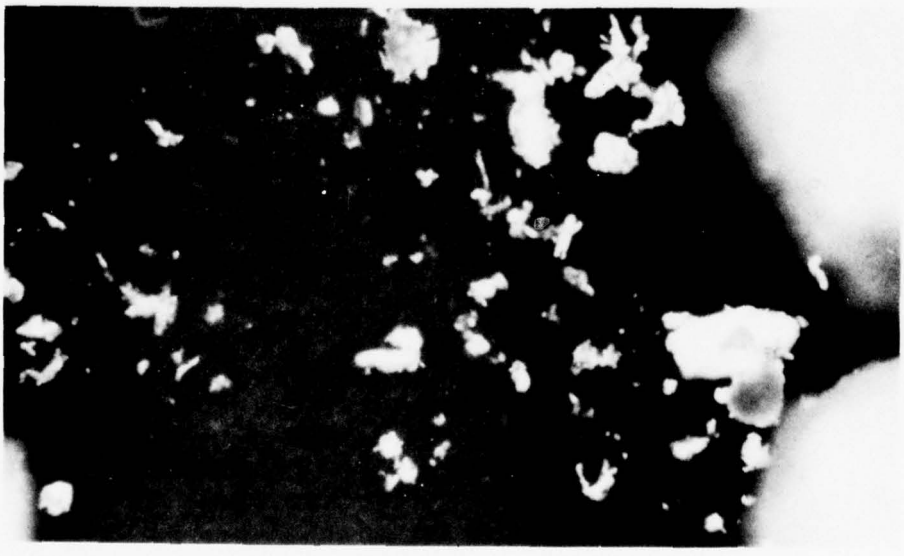

Figure 11. Photomicrograph of continuous process RDX Lot 053-4074 (30X) (small crystals).

Figure 12. Photomicrograph of continuous process RDX Lot 056-0001 (30X)

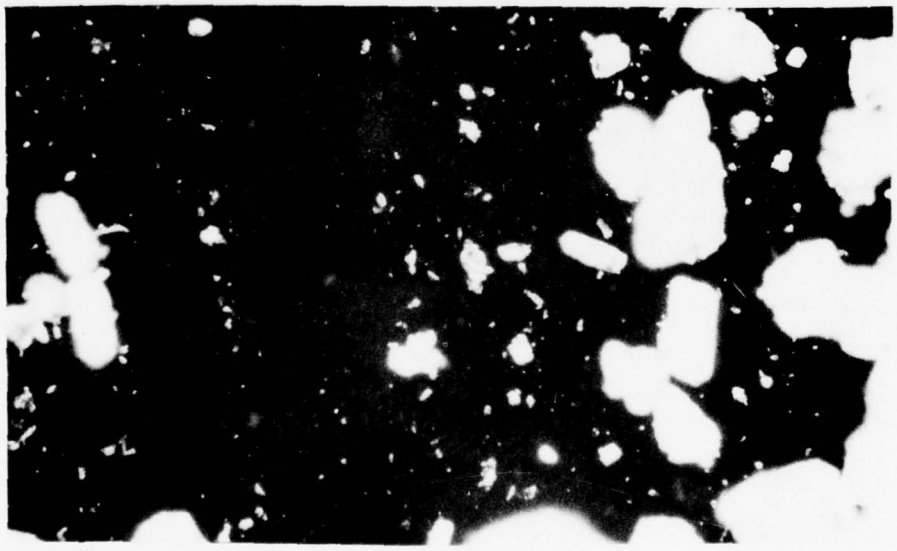

Figure 13. Photomicrograph of continuous process RDX Lot 056-0005 (30X).

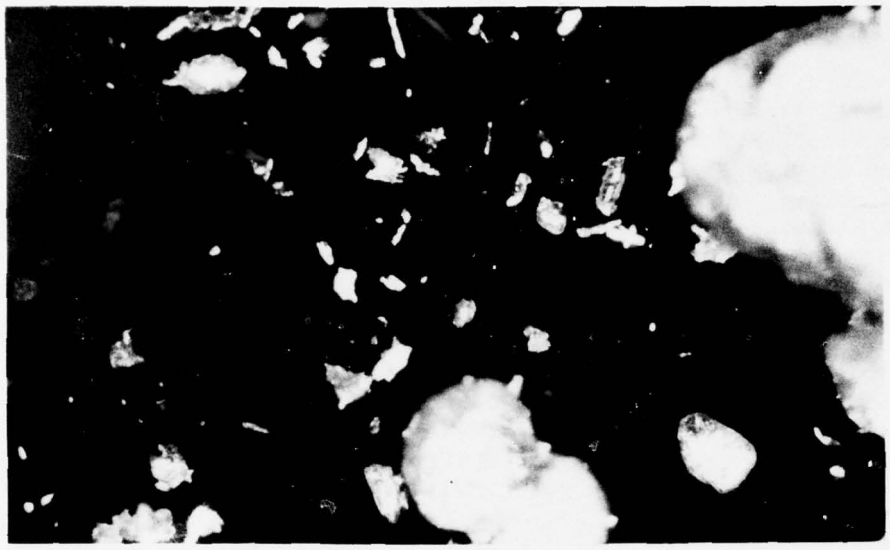

Figure 14. Photomicrograph of continuous process RDX Lot 056-0007 (30X).

the size of the RDX crystals. In each lot the RDX crystals manufactured by the batch process were smaller than the continuous process Comp B lots. The batch process RDX crystals were more consistent and more uniform in size as can be seen in figure 15. In contrast the crystals in the continuous process Comp B lots, as shown in figure 16, were, for the most part, larger, more irregular and the range in particle size much greater (from very large to very small).

DISCUSSION

The literature search had revealed that Blomquist (ref. 8), Johnson (ref. 9), Bachman, et al. (ref. 10), Jeffers (ref. 11), and Cady and Smith (ref. 12) had investigated the impact sensitivities of the HMX polymorphs. The most comprehensive investigations were by Jeffers and Cady and Smith. The work by Jeffers was conducted in the Rotter impact tester where the criterion of a "fire" or "go" was the production of 2 ml of gas. Cady and Smith paralleled Jeffers' work but with an ERL (NOL) Type 12 impact tester.

Some of the findings reported by Jeffers were conflicting. Based on a minimum size for an inclusion in the α-HMX crystal. Jeffers believed that conditions for initiation by adiabatic compression were more favorable for massive α-HMX rather than fine α-HMX. However, fine α-HMX sometimes was found to be more sensitive than massive α-HMX, i.e., the α-HMX formed by heating β-HMX at 190°C. The experiments carried out with α-HMX did show that the massive form was sensitive, but the sensitivity was not necessarily a function of size. However, an interesting result was obtained. After α-HMX had been treated at 130°C for 30 hours, its sensitivity to impact when tested at ambient temperature increased by almost 50%. Under the same conditions β-HMX displayed no change.

Cady and Smith (ref. 12) reported that in crystals of the sizes likely to be encountered in practice, the order of sensitivity of the HMX polymorphs would be $\delta > \gamma > \alpha > \beta$. Using 12B tools, Cady and Smith indicated that α- and β-HMX appear to be in the same sensitivity class. They qualified that statement by indicating that exceptions are not unusual. Although the sensitivity of α-HMX had been reported to increase with increasing thickness of the crystals (refs. 8,10,11), Cady and Smith were not able to confirm that conclusion. Jeffers (ref. 11) also reported exceptions to the trend and stated that the sensitivity of α-HMX as a function of particle size has not been fully resolved (ref. 13).

The available information on RDX-HMX mixtures does not indicate any significant effect on the sensitivity in the proportions of HMX normally found in RDX and subsequently in Comp B. Most of the

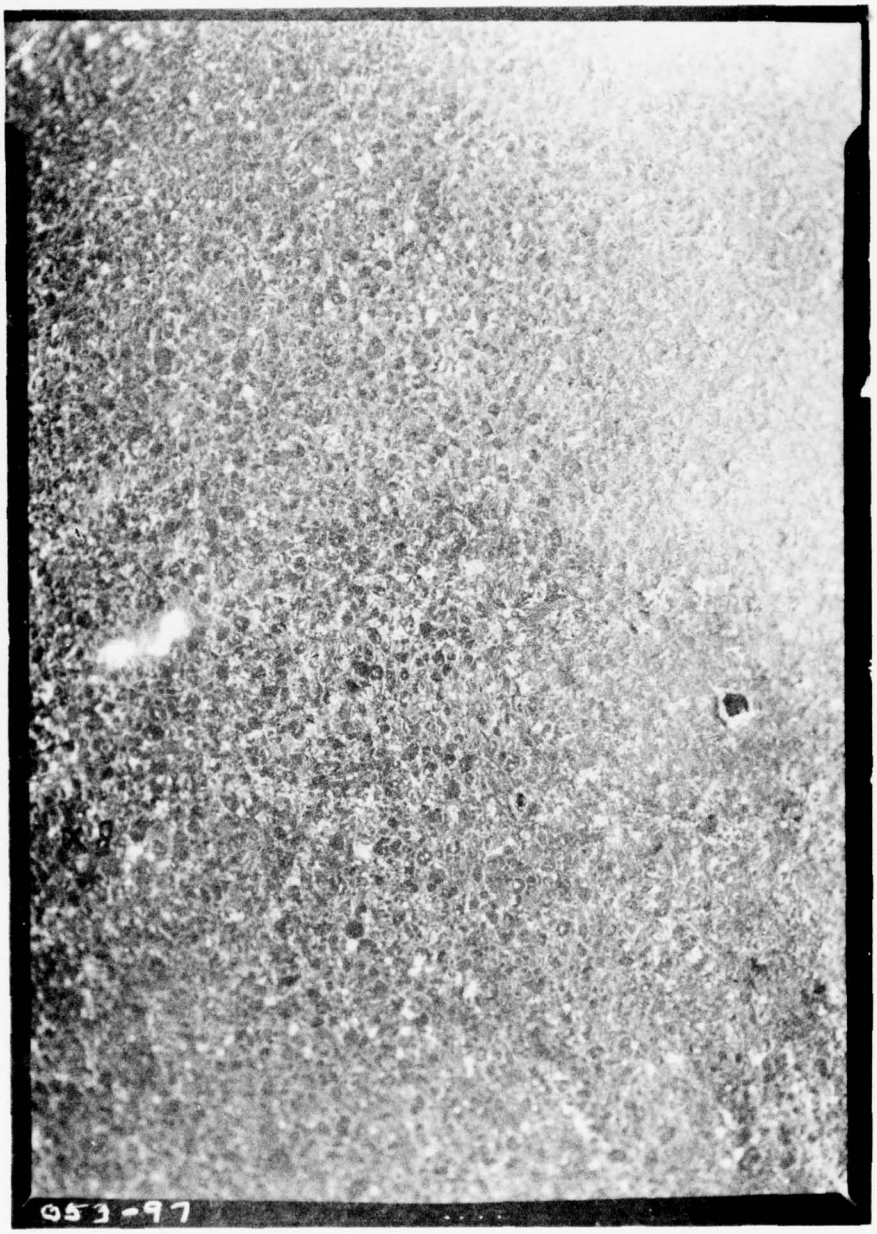

Figure 15. Etched surface casting of batch process Comp B Lot 053-97 (8X).

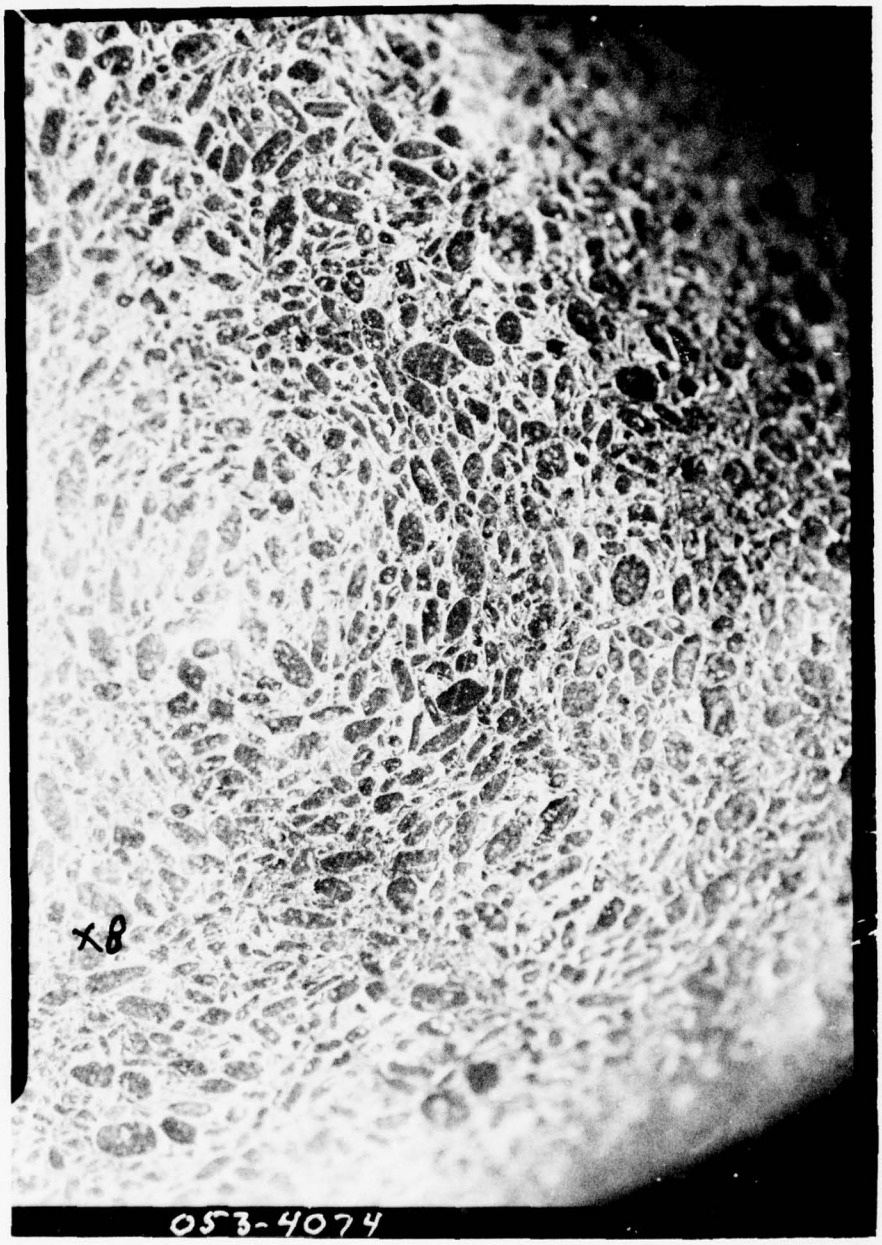

Figure 16. Etched surface casting of continuous process Comp B Lot 053-4074 (8X).

published data were generated during WWII (ref. 9, 10) and the effect of α-HMX was not clearly defined.

All the test data are summarized in table 10. In order to properly assess the data,the results of each test are evaluated so that a step-by-step comparison can be made between the batch and continuous processes. Upon extraction of the RDX from the batch and continuous process Comp B lots, an analysis was conducted on each lot to determine the amount of α-HMX. The results indicated that all of the batch RDX had less than 2% α-HMX, while the results for the continuous RDX revealed that two of the lots had less than 2%, one 2-3% and RDX Lot 056-0001 12-17% α-HMX.

The results on the impact test for the Comp B lots indicated that the average 50% point of the four batch process Comp B lots was 45.42 cm while for the four continuous process Comp B lots the average was 45.01 cm. The difference is not considered significant. For the RDX lots the average 50% point for the four batch process lots is 32.92 cm and for the four continuous process lots the average is 32.98 cm. Again the difference is insignificant.

The large scale gap test was conducted on the Comp B lots. The average 50% gap for the four batch process Comp B lots is 5.59 cm (2.202 in.) while for the four continuous process lots the average is 5.57 cm (2.194 in.). The difference here is not considered significant.

In the small scale gap test which was conducted only on the RDX lots, the results did show a difference between the batch and continuous process lots. The average 50% gap of the batch process lots is 1.07 cm (0.421 in.) which converts to a 3.76 gap decibang while for the continuous process RDX lots the average 50% gap is 0.95 cm (0.373 in.) or a 4.29 gap decibang. In the small scale gap test the batch process RDX lots were more sensitive to shock.

The projectile impact test produced an average of the four 50% Bruceton velocities for the batch process Comp B lots of 956.3 m/sec (3137.5 ft/sec). For the continuous process Comp B lots the average of the four is 350.3 m/sec (3118.0 ft/sec). The velocity of the maximum likelihood distribution for each batch process was averaged and the value was 951.8 m/sec (3123.0 ft/sec) compared to 973.5 m/sec (3194.0 ft/sec) for the continuous process lots. Although the range in the velocities is greater with the continuous process lots producing the highest and lowest impact velocities, the averages for the two processes were within 1-2% of each other, which is well within experimental error.

Table 10. Summary of test data for batch and continuous process Comp B and RDX.

	Composition B								RDX (Extracted From Comp B Lots)							
	Batch Process Comp B Lots				Continuous Process Comp B Lots				Batch Process RDX				Continuous Process RDX			
Composition B Lot Number	053-97	053-99	053-5423	053-5431	053-4074	056-0001	056-0005	056-0007	053-97	053-99	053-5423	053-5431	053-4074	056-0001	056-0005	056-0007
Composition B Batch Number	773794	774177	370448	370776	15321	152112	153039	153160								
RDX Batch Incorporated	7RCA-5947	7RCA-6010	3RCA-796	3RCA-1036	1RCA-292	1RCA-30	1RCA-243	1RCA-267	7RCA-5947	7RCA-6010	3RCA-976	3RCA-1036	1RCA-292	1RCA-30	1RCA-243	1RCA-267
α-HMX Content %									<2	<2	<2	<2	<2	12-17	<-2	2-3
Total HMX %														17.7		12.2
Impact Test-50%Pt-cm	45.50	42.58	46.79	46.83	43.56	45.75	45.91	44.83	34.92	32.22	31.59	32.95	31.14	31.20	32.27	37.32
Average		45.42				45.01				32.92				32.98		
Large Scale Gap Test - Inch	2.232	2.155	2.22	2.195	2.13	2.13	2.257	2.26								
50%Pt (cm)	(5.67)	(5.47)	(5.64)	(5.58)	(5.41)	(5.41)	(5.73)	(5.74)								
Average		2.202 (5.59)				2.194 (5.57)										

Table 10 (Continued)

	Composition B								RDX (Extracted from Comp B Lots)							
	Batch Process Comp B Lots				Continuous Process Comp B Lots				Batch Process RDX				Continuous Process RDX			
Small Scale Gap Test-Inch (cm)									0.406 (1.03)	0.433 (1.10)	0.423 (1.07)	0.422 (1.07)	0.372 (0.94)	0.390 (0.99)	0.377 (0.96)	0.351 (0.89)
Average									0.421 (1.07)				0.373 (0.95)			
Gap Decibangs									3.91	3.64	3.74	3.75	4.29	4.08	4.24	4.55
Average									3.76				4.29			
Projectile Impact ft/sec	3075	3175	3125	3175	3500	3150	2900	2925								
50% Bruceton (m/sec)	(937.2)	(967.7)	(952.5)	(967.7)	(1066.7)	(960.1)	(883.9)	(891.5)								
Average	3137.5 (956.3)				3118 (950.3)											
Average Max. Likelihood	3123.0 (951.8)				3194 (973.5)											
Friction Sensitivity																
Steel Shoe Reaction	Spark	Nil	Crackle	Crackle	Nil	Crackle	Crackle	Crackle	Crackle	Crackle	Crackle	Detonation	Crackle	Crackle	Detonation	Crackle
Fiber Shoe Reaction	Nil		Nil	Nil		Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Electrostatic Test 20@ 0.25J	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil

Table 10 (Continued)

	Composition B								RDX (Extracted From Comp B Lots)							
	Batch Process Comp B Lots				Continuous Process Comp B Lots				Batch Process RDX				Continuous Process RDX			
100°C Vacuum Stability Test																
ml/5gm/48 hrs	0.42	0.44	0.45	0.50	0.50	0.54	0.51	0.51	0.37	0.39	0.38	0.37	0.42	0.39	0.43	0.42
Average	0.45				0.52				0.38				0.42			
Differential Thermal Analysis																
Endotherm, Onset °C	73	73	72	74	72	73	68	73	183	183	183	177	179	186	182	178
Endotherm Peak °C	77	77	78	77	79	78	78	78	198	200	199	197	201	198	198	200
Exotherm, Onset °C	196	192	190	193	187	190	190	193	205	206	207	205	211	206	205	210
Exotherm, Peak °C	249	254	254	247	247	252	243	254	244	240	248	245	250	253	251	254
Heating Rate 20°C/min																
Thermogravimetric Analysis																
Sample Wt.mg	8.85	7.9	7.2	8.2	9.6	8.6	8.0	8.2	6.8	7.4	6.4	5.4	7.3	9.2	7.8	7.9
Decomposition Start °C	140	157	140	145	140	140	155	130	185	185	185	200	180	195	180	175
10% Wt. Loss Temp °C	208	218	203	208	210	208	218	217	233	216	218	238	219	211	218	223
Deflagration Temp °C	240	233	228	220	220	222	235	237	240	217	224	243	223	215	225	225
Heating Rate 20°C/min																

One batch and one continuous process Comp B lot passed the friction pendulum test with the steel shoe. All the rest produced crackles, but each passed using the fiber shoe. With the RDX lots one batch process lot produced a detonation as did a continuous process lot. All the rest produced crackles. However,all the RDX lots passed the test using the fiber shoe. These results agree with other Comp B and RDX values (ref. 5).

The electrostatic test consisted of 20 trials for lots in which a sample is subjected to a discharge of 0.25 joules. No reactions occurred with all the batch and continuous process Comp B and RDX lots.

In the 100°C vacuum stability test all the Comp B and RDX lots produced less than 1 ml gas in 48 hours for a 5 gram sample.

The DTA and TGA thermograms for the batch and continuous process Comp B and RDX lots produced slight differences which were considered to be insignificant.

The photomicrographs confirmed the presence of α-HMX crystals in continuous process RDX Lots 056-0001 and 056-0007. Also the 8X photographs of the etched Comp B castings revealed the presence of larger RDX crystals in the continuous process Comp B lots. However, in either instance, no evidence of massive α-HMX crystals was found.

SUMMARY AND RECOMMENDATIONS

It can be concluded from this study that there is no significant difference in sensitivity between batch and continuous process Comp B and RDX. Therefore the continuous process Comp B in deferred status is suitable for use.

Based on the results obtained it was recommended that all Comp B lots containing continuous recrystallization RDX be released from "Hold" status (Condition Code J) with no restrictions (ref. 14).

It is also recommended that a detailed investigation be conducted to determine the sensitivities of the HMX polymorphs and their effect on mixtures of RDX and Composition B under the various parameters expected under production conditions.

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APPENDIX A

EXTRACTION OF RDX FROM COMP B

(Method received from A. Popolato, LASL, in letter to L.C. Baker, ARRADCOM, d. 25 May 1977)

In order to preserve the polymorphic phase of the RDX present in the Comp B, it is necessary to separate the RDX at room temperature with solvents in which the RDX is either insoluble or only slightly soluble. The most suitable solvent for the extraction of TNT and some of the wax is toluene* saturated with RDX. The extraction should be performed near 20°C.

The choice of equipment and the exact procedure to be used depend upon the quantity of RDX to be extracted. The following procedure should be suitable:

> Weigh the desired quantity of Comp B into a stainless steel container; add an excess of RDX-saturated toluene and agitate the mixture. When the TNT is in solution, filter through a suitable filter to collect the RDX. Wash the RDX in the filter with cold (20°C) RDX-saturated toluene.
>
> At this stage of the separation, some of the wax desensitizer may be mixed with the RDX. The wax can be removed by washing with chloroform,** (this may not be required for your tests).

A representative sample of the dried RDX should be selected and analyzed to determine the residual TNT and wax.

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* The solubilities of TNT and RDX in toluene at 20°C are 55 g/100 g of solvent and 0.020 g/100 g of solvent, respectively.

** The solubility of wax and RDX in chloroform at 20°C is 2.42 and 0.00 g/100 g of solvent, respectively.

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