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NAVORD REPORT

6296

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THE CHEMICAL ANALYSIS OF DESENSITIZED BOOSTER AND
DETONATING CORD COMPOSITIONS (U)

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U. S. NAVAL ORDNANCE LABORATORY
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THE CHEMICAL ANALYSIS OF DESENSITIZED BOOSTER AND
DETONATING CORD COMPOSITIONS (U)

MAR 59 14P Glover, Donald J. ; Heitzmann,
Martin W. ; Morris, Walter W. ;
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DESCRIPTORS: ANALYSIS, GRAPHITE, MATERIALS (U)

THE CHEMICAL ANALYSIS OF DESENSITIZED BOOSTER AND
DETONATING CORD COMPOSITIONS (U)

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ABSTRACT: A booster composition containing RDX, calcium stearate, polyisobutylene and graphite was analyzed using the selective solubilities of RDX in acetone, calcium stearate in acetic acid and polyisobutylene in toluene. Known mixtures of RDX containing 1.5% calcium stearate and 0.5% polyisobutylene were separated quantitatively with recoveries of 100.1%, 98.5% and 98.8%, respectively, of the amounts present.

A detonating cord composition consisting of RDX with 1 to 3% of calcium stearate can be readily analyzed by selective solution of the RDX in acetone.

Recommended procedures are given for the analysis of the booster explosive, CH-6, and the detonating cord composition.

CHEMISTRY RESEARCH DEPARTMENT
U. S. NAVAL ORDNANCE LABORATORY
White Oak, Silver Spring, Maryland

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10 March 1959

Methods of analysis are described in this report which were devised for quality control of two explosive formulations developed at the Naval Ordnance Laboratory. These methods are believed suitable for inclusion in military specifications. Funds for this project were allocated under Task 301-664/43006/08.

MELL A. PETERSON
Captain, USN
Commander

Albert Lightbody
ALBERT LIGHTBODY
By direction

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CONTENTS

	<u>Page</u>
INTRODUCTION -----	1
RESULTS AND DISCUSSIONS -----	1
Detonating Cord -----	1
Booster Composition -----	5
RECOMMENDED PROCEDURE -----	6
Detonating Cord Composition -----	6
Booster Composition -----	8
REFERENCES -----	14

Illustrations

TABLE I. ANALYSIS OF RDX-CALCIUM STEARATE MIXTURES -----	2
TABLE II. ANALYSIS OF DETONATING CORD EXPLOSIVE-	3
TABLE III. ANALYSIS OF THREE COMPONENT MIXTURE (Z-93) -----	7
TABLE IV. ANALYSIS OF RDX-CALCIUM STEARATE- POLYISOBUTYLENE MIXTURE -----	10
TABLE V. ANALYSIS OF THREE COMPONENT MIXTURE (Z-93) -----	11
TABLE VI. ANALYSIS OF FOUR COMPONENT MIXTURE (X-253) -----	12
TABLE VII. ANALYSIS OF FOUR COMPONENT MIXTURE (X-255) -----	13

THE CHEMICAL ANALYSIS OF DESENSITIZED BOOSTER AND DETONATING CORD COMPOSITIONS (U)

INTRODUCTION

A booster composition composed of desensitized RDX was successfully developed at the NOL as a heat resistant improvement over tetryl in order to minimize accidental explosions attributed to the rapid decomposition, or "cook-off", of tetryl (1).

In addition to the above task, a detonating cord composition containing desensitized RDX was developed for use in the Talos missile. The Talos missile also was designed to use the RDX booster composition in the explosives system to reduce the possibility of a "cook-off".

Analytical methods, described below, were developed for quality control of these formulations and for inclusion in ordnance specifications (2).

RESULTS AND DISCUSSIONS

Detonating Cord

Because RDX is soluble in acetone to the extent of 11.5g/100ml at 40°C and about 18g/100ml near its boiling point (3), an extraction of RDX from calcium stearate seems attractive providing calcium stearate is relatively insoluble in acetone.

By placing accurately weighed samples of calcium stearate (previously slurried with hot acetone and dried) in medium porosity, sintered-glass crucibles and extracting with 10ml portions of acetone, it was found that calcium stearate was negligibly soluble in acetone. There appeared to be some mechanical loss, for extraction with 60ml of acetone gave a loss of about 5mg from 0.5g samples, while further extraction of these samples with 60ml of acetone, gave a loss of only 0.4mg. Using fine porosity crucibles gave a loss of only 2mg and 0.5mg, respectively, with two 60ml extractions. The weight of the residue was found after drying at 100°C for 40 minutes.

A sample of each component was weighed into a crucible of medium porosity and the mixture was extracted with 10ml portions of acetone at

room temperature. With 3g of RDX, 70ml of acetone were required for complete extraction. Four such mixtures contained 40-100mg of calcium stearate and the recovery was 95-97% (average of 3 mg lost).

Samples prepared at NOL for gap sensitivity measurements were analyzed by extracting with 10ml portions of acetone. Results are given in Table I.

TABLE I
ANALYSIS OF RDX-CALCIUM STEARATE MIXTURES

Sample wt., g Range	Calcium Stearate		% Recovery	Acetone Used, ml
	% Added	Found		
6.5	1.4	1.40	100.0	120
		1.40		
		1.39		
		Av. 1.40		
5.0-6.0	2.0	2.01	99.0	120
		1.98		
		1.95		
		Av. 1.98		
3.0-4.3	2.8	2.67	94.7	160
		2.60		
		2.68		
		Av. 2.65		
2.6-3.6	4.0	3.73	94.3	160
		3.77		
		3.80		
		Av. 3.77		
2.2-4.1	5.6	5.40	96.4	160
		5.45		
		5.36		
		Av. 5.40		

TABLE I (cont'd)

ANALYSIS OF RDX-CALCIUM STEARATE MIXTURES

Sample wt., g Range	Calcium Stearate		% Recovery	Acetone Used, ml
	% Added	Found		
2.0-4.1	8.0	7.67	98.4	160
		8.00		
		7.94		
		Av. 7.87		

The average value for the percent added is 97.1, which agrees with that found by weighing each of the components separately before extraction.

Detonating cords prepared by the Austin and Ensign-Bickford manufacturers were submitted for analysis. There were three cords (70, 120, and 200 grains per foot) from Austin, each of which was divided into two reels; and two cords from Ensign-Bickford (120 and 200 grains per foot), each of which was divided into three reels. Samples from each reel were analysed in duplicate by the method outlined under "Recommended Procedures". Results are given in Table II.

TABLE II

ANALYSIS OF DETONATING CORD EXPLOSIVE

Grains Explosive Per Foot	Reel Number	% Residue	Av.
70	A U S T I N		
	1	1.60	1.57
		1.54	
	2	1.63	1.64
		1.65	
	120	1	1.65
1.66			

TABLE II (cont'd)
ANALYSIS OF DETONATING CORD EXPLOSIVE

Grains Explosive Per Foot	Reel Number	% Residue	Av.
120	2	1.73	1.70
		1.66	
200	1	2.04	2.05
		2.06	
	2	1.85	1.85
		1.85	
ENSIGN - BICKFORD			
120	1	1.36	1.35
		1.34	
	2	1.37	1.36
		1.35	
	3	1.56	1.45
		1.35	
200	1	1.63	1.63
		1.62	
	2	1.65	1.67
		1.68	
	3	1.74	1.75
		1.75	

Acetone is reasonably efficient for separating RDX from 1 to 3% of calcium stearate. A recovery of at least 95% of the calcium stearate added can be attained which is all that is required in this work. For samples with higher percentages of calcium stearate one would expect

a more efficient recovery of this constituent. Any lack of agreement between percent calcium stearate added and found in Table II very likely is associated with the sample submitted for analysis and not with the method of analysis.

Booster Composition

A composition containing RDX with small amounts of calcium stearate, polyisobutylene and graphite was developed for a heat resistant booster explosive. Because RDX could be removed satisfactorily from calcium stearate by acetone extraction, a similar extraction of the booster mixture seemed in order.

Samples of polyisobutylene (PIB) weighing about 0.2g were extracted with 150ml of hot acetone using medium porosity, sintered-glass crucibles. The crucibles plus the residue were dried at 100°C for 40 minutes, cooled, and weighed. A loss of 4-6mg occurred which was considered negligible for this determination.

Next, mixtures of RDX (10g), calcium stearate (0.15g), and PIB (0.10g) were prepared by weighing each of the components into the crucibles. Four such mixtures were extracted, each with 200ml of hot acetone. The crucibles were dried and weighed, showing an RDX extraction of 100.2%. A further extraction with 40ml of acetone gave an additional weight loss of only 2-6mg.

As the PIB is applied by dissolving it in toluene, this solvent is used for the extraction. Because PIB dissolves slowly, the toluene is heated to boiling before extraction. PIB precipitates out as soon as the solution comes through the filter. This is eliminated by first heating the crucible, either by placing it in boiling toluene for two minutes or heating it in an oven at 100°C for 5 minutes. Extraction of 0.2g of PIB requires 85ml of toluene for quantitative removal.

Finding a suitable solvent for calcium stearate became somewhat of a problem. Hot, concentrated sulfuric acid dissolved calcium stearate, but the filtration was too slow. Although sulfuric acid does not dissolve PIB, any acid which was left in the crucible (after washing with water) caused some charring of the PIB during the drying operation.

Hot pyridine was found to be a good solvent for calcium stearate. Only 4 to 8mg of PIB dissolved in 125ml of pyridine.

Calcium stearate had a strong tendency to clog the sintered-glass crucible during the filtration. This was overcome by heating the crucible prior to filtration by placing it at a depth of about one-half inch in boiling pyridine.

A satisfactory procedure was devised using acetone, pyridine and toluene for the separation of RDX, calcium stearate and polyisobutylene in the booster composition as shown in Table III. Calcium stearate was extracted before the PIB in Table III, as previous work showed a rather low recovery for calcium stearate if the order were reversed.

The toxicity and very disagreeable odor of pyridine made its replacement desirable. Hot glacial acetic acid was found to dissolve calcium stearate more efficiently than pyridine. Two calcium stearate samples of 0.2g each were extracted with two 12ml portions of hot acid. We removed 99.8% of the calcium stearate by this treatment. Also, we found that only a fraction of a milligram of PIB was dissolved along with the calcium stearate. In this respect glacial acetic acid effected a better separation of calcium stearate from PIB than pyridine.

Five mixtures of RDX, calcium stearate and PIB were prepared by weighing each component separately into crucibles. These mixtures were extracted with 200ml of hot acetone, 62 and 97ml of hot glacial acetic acid and 150ml of hot toluene by the recommended procedure given later in this report. Results of the separation are given in Table IV.

RECOMMENDED PROCEDURE

Detonating Cord Composition

Weigh accurately a sample of about 10g into a 30ml medium porosity, sintered-glass crucible (previously weighed). Crucible plus sample = w_1 .

Place the crucible containing the sample on a suction flask and fill with hot acetone (25ml, 55°C) and apply suction. Repeat this procedure 7 more times (total acetone = 200ml). Discard the acetone solution.

Dry the crucible in an oven at 100°C for 40 minutes. Cool in a desiccator and weigh (w_2). The percent RDX is found by:

$$\frac{(w_1 - w_2)}{\text{weight sample}} \times 100$$

TABLE III
ANALYSIS OF THREE COMPONENT MIXTURE (Z-93) (a)

Sample Weight, g	RDX		Calcium Stearate		PIB	
	Grams	%	Grams	%	Grams	%
10.001	9.805	98.0	0.1562	1.56	0.0400	0.40
10.007	9.812	98.0	0.1561	1.56	0.0381	0.38
10.036	9.842	98.0	0.1547	1.54	0.0380	0.38
10.017	9.825	98.1	0.1535	1.53	0.0381	0.38
10.001	9.802	98.0	0.1528	1.53	0.0465	0.47
10.003	9.801	98.0	0.1552	1.55	0.0464	0.46
10.003	9.802	98.0	0.1553	1.55	0.0464	0.46
10.004	9.798	97.9	0.1569	1.57	0.0496	0.50
	AV.	98.0	AV.	1.55	AV.	0.43

*Made up to contain: RDX, 98.0%; Calcium Stearate, 1.50%; PIB, 0.50%.

Booster Composition

Weigh accurately a sample of about 10g into a 30ml medium porosity, sintered-glass crucible (previously weighed). Crucible plus sample = w_1 .

ALL OF THE FOLLOWING OPERATIONS MUST BE CONDUCTED IN A HOOD.

Place the crucible containing the sample on a suction flask and fill with hot acetone (25ml) (55°C) and apply vacuum. Repeat this procedure 7 more times (total acetone = 200ml). Discard the acetone solution.

Dry the crucible from above in an oven at 100°C for 40 minutes. Cool in a desiccator and weigh (w_3). The percent RDX is found by:

$$\frac{(w_1 - w_3)}{\text{Wt. sample}} \times 100$$

Place the crucible on the filter flask. Add 12ml of hot glacial acetic acid (110°C) to the crucible and let stand for 2 minutes, then apply vacuum. Repeat this procedure twice with the same volume of acetic acid, then once with a filling of the crucible (total volume = 60ml). Finally, wash the residue with 25ml of acetone and discard the acetic acid-acetone solution

Dry the crucible in an oven at 100°C for 40 minutes. Cool in a desiccator and weigh (w_5). The percent calcium stearate is found by:

$$\frac{(w_3 - w_5)}{\text{Wt. sample}} \times 100$$

Warm the crucible from above by placing it in the oven for 5 minutes; then place it on the filter flask. Add 25ml of hot toluene (105°C) to the crucible and let stand for 2 minutes and apply vacuum. Repeat this procedure 7 more times (total volume = 200ml). Finally, wash the residue with 25ml of acetone. Discard the toluene-acetone solution.

Dry the crucible in an oven at 100°C for 40 minutes. Cool in a desiccator and weigh (w₇). The percent polyisobutylene is found by:

$$\frac{(w_5 - w_7)}{\text{Wt. sample}} \times 100$$

The percent graphite is found by:

$$\frac{(w_7 - \text{crucible wt.})}{\text{Wt. sample}} \times 100$$

Three booster compositions submitted for analysis were analyzed by the method outlined under recommended procedures. The calcium stearate values appear to be slightly low as indicated by Tables V, VI, and VII. This would be expected as calcium stearate is very slightly soluble in acetone. However, the method appears to be satisfactory for adequate control of calcium stearate in the CH-6 booster composition (2), (4).

In general, the chemical methods reported herein are believed satisfactory for control purposes. They are relatively rapid and require only standard laboratory reagents and equipment.

TABLE IV
ANALYSIS OF RDX-CALCIUM STEARATE-POLYISOBUTYLENE
MIXTURE

	RDX, g		CALCIUM STEARATE, g			PIB, g		
	Present	Found	Present	Found ^(a)	Found ^(b)	Present	Found	
		%		%	% ^(c)		(by diff.)	
9.979	9.985	100.1	0.2019	0.1927	0.1969	0.1796	0.1779	99.0
9.982	9.988	100.1	0.2015	0.1939	0.1991	0.1939	0.1906	98.3
9.978	9.983	100.1	0.2064	0.1993	0.2068	0.2500	0.2445	97.8
9.996	9.998	100.0	0.2037	0.2012	0.2010	0.2196	0.2196	100.0
9.997	10.005	100.1	0.1991	0.1818	0.1937	0.1875	0.1855	98.9
	AV.	100.1			AV. 98.5		AV.	98.8

(a) 62ml of Acetic Acid used.
(b) 97ml of Acetic Acid used.
(c) Based on 97ml extraction.

TABLE V
ANALYSIS OF THREE COMPONENT MIXTURE (Z-93) (a)

Sample Weight, g	RDX		CALCIUM STEARATE (b)		PIB	
	Grams	%	Grams	%	Grams	%
9.950	9.747	98.0	0.1436	1.44	0.0510	0.51
10.005	9.807	98.0	0.1420	1.42	0.0494	0.49
10.016	9.810	97.9	0.1479	1.48	0.0528	0.53
10.026	9.826	98.0	0.1499	1.50	0.0475	0.47
10.013	9.813	98.0	0.1484	1.48	0.0495	0.50
	AV.	98.0	AV.	1.46	AV.	0.50

(a) Made up to contain: RDX, 98.0%; Calcium Stearate, 1.50%; PIB, 0.50%

(b) 80ml Acetic Acid used. After PIB extraction, there was a residue which was soluble in 25ml of hot acetic acid. Weight of residue extracted was: 5.9, 6.3, 4.2, 2.6, and 2.3mg, in the order listed.

TABLE VI

ANALYSIS OF FOUR COMPONENT MIXTURE (X-253)^(a)

Sample Weight, g	RDX		CALCIUM STEARATE		PIB		GRAPHITE (Residue)	
	Grams	%	Grams	%	Grams	%	Grams	%
10.004	9.791	97.9	0.1191	1.19	0.0469	0.47	0.0467	0.47
10.021	9.809	97.9	0.1181	1.18	0.0466	0.47	0.0478	0.48
10.007	9.793	97.9	0.1185	1.18	0.0449	0.45	0.0503	0.50
9.982	9.763	97.8	0.1202	1.20	0.0437	0.44	0.0552	0.55
	AV. 97.9		AV. 1.19		AV. 0.46		AV. 0.50	

(a) Prepared at NOL.

TABLE VII
ANALYSIS OF FOUR COMPONENT MIXTURE (X-255)(a)

Sample No.	Sample Weight g	RDX		CALCIUM STEARATE (b)		PIB		GRAPHITE (Residue)		
		Grams	%	Grams	%	Grams	%	Grams	%	
1	10.000	9.764	97.6	0.1385	-----	1.39	0.0468	0.47	0.0510	0.51
2	10.003	9.761	97.6	0.1404	-----	1.40	0.0435	0.44	0.0581	0.58
3	10.013	9.774	97.6	0.1367	-----	1.37	0.0484	0.48	0.0542	0.54
4	10.010	9.779	97.7	0.1310	-----	1.31	0.0491	0.49	0.0508	0.51
5	10.002	9.765	97.6	0.1403	0.1391	1.39	0.0443	0.44	0.0538	0.54
6	10.008	9.758	97.5	0.1516	0.1501	1.50	0.0505	0.51	0.0497	0.50
7	10.003	9.769	97.7	0.1403	0.1386	1.39	0.0487	0.49	0.0464	0.46
8	10.008	9.768	97.6	0.1439	0.1427	1.43	0.0479	0.48	0.0489	0.49
		AV. 97.6		AV. 1.40		AV. 1.40	AV. 0.48		AV. 0.48	AV. 0.52

(a) Prepared at Holston Ordnance Works
 (b) Samples 1, 2, and 3, 62ml Acetic Acid. Sample 4, 100ml Acetic Acid. Samples 5-8:A, 50ml Acetic Acid; B, 75ml (Total) Acetic Acid

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2. Rockets - Boosters
3. RDX
4. Calcium stearate
5. Polyiso-butylene
6. Graphite
7. Explosives - Ch-6
8. Cords, Detonating
- I. Title
- II. Glover, Donald J.
- III. Project

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RDX in acetone.
Recommended procedures are given for the
analysis of the booster explosive, CH-6, and
the detonating cord composition.

1. Missiles - Talos
2. Rockets - Boosters
3. RDX
4. Calcium stearate
5. Polyiso-butylene
6. Graphite
7. Explosives - Ch-6
8. Cords, Detonating
- I. Title
- II. Glover, Donald J.
- III. Project

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Naval Ordnance Laboratory, White Oak, Md.
(NAVORD report 6296)
THE CHEMICAL ANALYSIS OF DESENSITIZED BOOSTER
AND DETONATING CORD COMPOSITIONS (U), by Donald
J. Glover and others. 10 March 1959. 14p.
tables. Project 301-664/43006/08.
A booster composition containing RDX, calcium
stearate, polyisobutylene and graphite was ana-
lyzed using the selective solubilities of RDX
in acetone, calcium stearate in acetic acid and
polyisobutylene in toluene. Known mixtures of
RDX containing 1.5% calcium stearate and 0.5%
polyisobutylene were separated quantitatively
with recoveries of 100.1%, 98.5% and 98.8%,
respectively, of the amounts present.
A detonating cord composition consisting of
RDX with 1 to 3% of calcium stearate can be
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