

UNCLASSIFIED

AD NUMBER	
AD021247	
CLASSIFICATION CHANGES	
TO:	unclassified
FROM:	confidential
LIMITATION CHANGES	
TO:	Approved for public release; distribution is unlimited.
FROM:	Distribution authorized to U.S. Gov't. agencies and their contractors; Administrative/Operational Use; 15 SEP 1953. Other requests shall be referred to Office of Naval Research, Arlington, VA 22203.
AUTHORITY	
ONR ltr, 10 Apr 1961; ONR ltr, 26 Oct 1977	

THIS PAGE IS UNCLASSIFIED

CONFIDENTIAL

45109

AD No. 21 247
ASTM FILE COPY

NAVY DEPARTMENT
OFFICE OF NAVAL RESEARCH
WASHINGTON, D. C.


15 September 1953
Report No. 737
(Quarterly)
Copy No. 1

EXPLOSIVES RESEARCH



Contract N7onr-46208

Aerojet-General CORPORATION
 A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY
 AZUSA, CALIFORNIA



CONFIDENTIAL
SECURITY INFORMATION

ASTM

CONFIDENTIAL

15 September 1953

Report No. 737
(Quarterly)

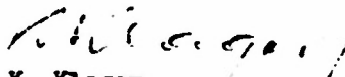
EXPLOSIVES RESEARCH

Contract N7onr-46208

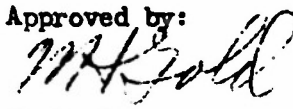
Written by:

L. T. Carleton
M. B. Frankel

Approved by:


K. Klager
Senior Organic Chemist
Solid Engine and
Chemical Division

Approved by:


M. H. Gold
Principal Chemist
Solid Engine and
Chemical Division

No. of Pages: 62

Period Covered:

1 May through 31 July 1953

This document contains information affecting the national defense of the United States within the meaning of the Espionage Laws, Title 18, U.S.C., Sections 793 and 794. The transmission or the revelation of its contents in any manner to an unauthorized person is prohibited by law.

AEROJET-GENERAL CORPORATION

Azusa, California

CONFIDENTIAL

CONFIDENTIAL

Report No. 737

CONTENTS

	<u>Page</u>
Contract Fulfillment Statement _____	v
Initial Distribution of this Report _____	Follows Appendix
I. SUMMARY _____	1
II. TECHNICAL PROGRESS: SYNTHESIS OF NEW HIGH EXPLOSIVES _____	4
A. Introduction _____	4
B. Preparation of Aliphatic Polynitrocarbamates _____	4
C. Preparation of Postnitrated Aliphatic Polynitrocarbamates _____	8
D. Preparation of Aliphatic Polynitronitrates _____	10
E. Preparation of Aliphatic Polynitrosamine Salts _____	13
F. Preparation of Samples for Evaluation by the Naval Ordnance Laboratory _____	14
III. DESENSITIZATION OF RDX _____	18
A. Investigation of Combinations of Polydinitrobutyl Acrylate, Plasticizers, and Surface-Active Agents _____	18
B. Surface-Active Agents Containing Nitro Groups _____	23

Table

I Aliphatic Polynitrocarbamates _____	7
II Postnitrated Aliphatic Polynitrocarbamates _____	11
III Aliphatic Polynitronitrates _____	12
IV Aliphatic Polynitrosamine Salts _____	15
V NOL Preliminary Tests on High Explosives _____	17
VI Compatibilities of Systems of Polynitrobutyl Acrylate, Plasticizer, and Surface-Active Agent _____	20

Page iii

CONFIDENTIAL

CONFIDENTIAL

Report No. 737

CONTENTS (cont.)

<u>Table</u>		<u>Page</u>
VII	Impact Stabilities of Systems Composed of Polydinitrobutyl Acrylate, Plasticiser, and Surface-Active Agent _____	21
VIII	Miscellaneous Desensitisation Experiments _____	22

APPENDIX - SPIA Data Sheets

1,3-bis(3',3',3'-Trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine

1,3,5-tris(3',3',3'-Trinitropropyl)-hexahydro-1,3,5-triazine

2,2,5,7,7,9,12,12-Octanitro-5,9-diaza-tridecane

2,2,4,7,7,10,12,12-Octanitro-4,10-diaza-tridecane

N,N'-bis(3,3,3-Trinitropropyl) oxamide

N,N'-Dinitro-N,N'-bis(trinitropropyl) oxamide

1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane

1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diaza-decane

CONFIDENTIAL

Report No. 737

CONTRACT FULFILLMENT STATEMENT

This quarterly report is submitted in partial fulfillment of
Contract N7onr-1.6209.

Page v

CONFIDENTIAL

CONFIDENTIAL

Report No. 737

I. SUMMARY

A. This quarterly report is submitted under Contract N7onr-46208 and covers the period 1 May through 31 July 1953.* The objectives of the contract are threefold:

1. Synthesis and physical studies of new high-energy polynitro compounds as potential explosives.
2. Desensitization of RDX with materials that will not lower the oxygen balance appreciably.
3. Development of bomb and shell casings made of an explosive plastic.

B. The more important results and conclusions are presented below:

1. Seven polynitrocarbamates were synthesized:
 - a. 2,2,9,9-Tetranitro-4-oxa-5-keto-6-aza-decane (I) from 3,3-dinitrobutyl isocyanate and 2,2-dinitropropanol.
 - b. 1,1,1,8,8-Pentanitro-3-oxa-4-keto-5-aza-nonane (II) from 3,3-dinitrobutyl isocyanate and 2,2,2-trinitroethanol.
 - c. 2,2,9,9,9-Pentanitro-4-oxa-5-keto-6-aza-nonane (III) from 3,3,3-trinitropropyl isocyanate and 2,2-dinitropropanol.
 - d. 1,1,1,8,8,15,15,15-Octanitro-4,12-diaza-5,11-diketo-6,10-dioxa-pentadecane (IV) from 3,3,3-trinitropropyl isocyanate and 2,2-dinitro-1,3-propanediol.
 - e. 1,1,1,8,15,15,15-Heptanitro-3,13-dioxa-4,12-diketo-5,8,11-triaza-pentadecane (V) from 3-nitro-1,5-pentane diisocyanate and 2,2,2-trinitroethanol.
 - f. 1,1,1,8,8,10,12,12,19,19,19-Undecanitro-3,17-dioxa-4,16-diketo-5,10,15-triaza-nonadecane (VI) from 3,3,5,7,7-pentanitro-5-aza-1,9-nonane diisocyanate and 2,2,2-trinitroethanol.
 - g. 1,1,1-Trinitro-3-oxa-4-keto-5-aza-hexane (VII) from methyl isocyanate and 2,2,2-trinitroethanol.

* Previous work on this contract was covered in Aerojet Reports No. 512, 536, 562, 589, 621, 637, 660, 682 and 711.

CONFIDENTIAL

CONFIDENTIAL

I Summary, B (cont.)

Report No. 737

2. Seven postnitrated polynitrocarbamates were prepared:

- a. 2,2,6,9,9-Pentanitro-4-oxa-5-keto-6-aza-decane (VIII)
by the nitration of I.
- b. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane (IX)
by the nitration of II.
- c. 2,2,6,9,9,9-Hexanitro-4-oxa-5-keto-6-aza-nonane (X)
by the nitration of III.
- d. 1,1,1,4,8,8,12,15,15,15-Decanitro-4,12-diaza-5,11-diketo-6,10-dioxa-pentadecane (XI) by the nitration of IV.
- e. 1,1,1,5,8,11,15,15,15-Nonanitro-3,13-dioxa-4,12-diketo-5,8,11-triaza-pentadecane (XII) by the nitration of V.
- f. 1,1,1,5,8,8,10,12,12,15,19,19,19-Tridecanitro-3,17-dioxa-4,16-diketo-5,10,15-triaza-nonadecane (XIII) by the nitration of VI.
- g. 1,1,1,5-Tetranitro-3-oxa-4-keto-5-aza-hexane (XIV) by the nitration of VII.

3. Four aliphatic polynitronitrates were prepared by the nitration of the corresponding alcohols:

- a. 4,4-Dinitro-1,7-heptanedinitrate (XV)
- b. 4,4,6,8,8-Pentanitro-1,11-undecanedinitrate (XVI)
- c. 4,4,6,6,8,8-Hexanitro-1,11-undecanedinitrate (XVII)
- d. 4,4,6,8,8-Pentanitro-6-aza-1,11-undecanedinitrate (XVIII).

4. Six polynitroamine salts were prepared by treating the corresponding isocyanates with dilute nitric or hydrochloric acid.

- a. 3,3-Dinitrobutyl ammonium nitrate (XIX)
- b. 3,3-Dinitro-1,5-pentane diammonium dinitrate (XX)
- c. 3-Nitrasa-1,5-pentane diammonium dinitrate (XXI)
- d. 3,6-Dinitrasa-1,8-octane diammonium dinitrate (XXII)

CONFIDENTIAL

I Summary, B (cont.)

Report No. 737

e. 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane diammonium dinitrate (XXIII)

f. 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane diammonium dihydrochloride (XXIV).

5. The following samples were prepared and submitted to the Naval Ordnance Laboratory for evaluation as potential explosives:

- a. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane
- b. 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diazadecane
- c. 4,4,6,8,8-Pentanitro-1,11-undecanedinitrate
- d. 4,4,6,6,8,8-Hexanitro-1,11-undecanedinitrate
- e. 4,4,6,8,8-Pentanitro-6-aza-1,11-undecanedinitrate
- f. 2,2,6,9,9-Pentanitro-4-oxa-5-keto-6-aza-decane
- g. 2,2,6,9,9,9-Hexanitro-4-oxa-5-keto-6-aza-nonane
- h. 1,1,1,4,8,8,12,15,15,15-Decanitro-4,12-diazas,11-diketo-6,10-dioxa-pentadecane
- i. 1,1,1,4,13,16,16,16-Octanitro-4,13-diazas,12-diketo-6,11-dioxa-8-hexadecane
- j. 1,1,1,5,8,11,15,15,15-Nonanitro-3,13-dioxas,12-diketo-5,8,11-triasa-pentadecane
- k. 1,1,1,5,8,8,10,12,12,15,19,19,19-Tridecanitro-3,17-dioxa-4,16-diketo-5,10,15-triasa-nonadecane
- l. 3,3-Dinitrobutyl ammonium nitrate
- m. 3,3-Dinitro-1,5-pentane diammonium dinitrate
- n. 3-Nitrasa-1,5-pentane diammonium dinitrate
- o. 3,6-Dinitrasa-1,8-octane diammonium dinitrate
- p. 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane diammonium dinitrate.

The preliminary evaluation of some of these compounds is described.

CONFIDENTIAL

I Summary, B (cont.)

Report No. 737

6. SPIA data sheets have been completed on the following compounds:

- a. 1,3-bis(3',3',3'-Trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine
- b. 1,3,5-tris(3',3',3'-Trinitropropyl)-hexahydro-1,3,5-triazine
- c. 2,2,5,7,7,9,12,12-Octanitro-5,9-diaza-tridecane
- d. 2,2,4,7,7,10,12,12-Octanitro-4,10-diaza-tridecane
- e. N,N'-bis(3,3,3-Trinitropropyl) oxamide
- f. N,N'-Dinitro-N,N'-bis(3,3,3-trinitropropyl) oxamide
- g. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane
- h. 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diaza-decane.

II. TECHNICAL PROGRESS: SYNTHESIS OF NEW HIGH EXPLOSIVES

A. INTRODUCTION

1. The present explosives program is directed toward the synthesis of new, stable, high-energy polynitro compounds with a preferred oxygen balance on the positive side or very near zero on the negative side.

2. This report describes the synthesis of aliphatic polynitro-carbamates, polynitronitrates, and polynitroamine salts.

B. PREPARATION OF ALIPHATIC POLYNITROCARBAMATES

1. Discussion

a. The preparation of aliphatic polynitrocarbamates for evaluation as high explosives has been previously reported.* This work has now been extended utilizing the new isocyanates and alcohols prepared on the nitropolymer program. The following aliphatic polynitrocarbamates were synthesized.

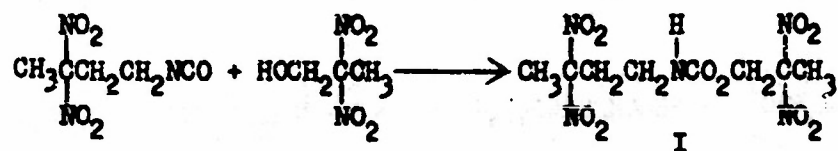
*Aerogel Reports No. 562, 589, and 621.

CONFIDENTIAL

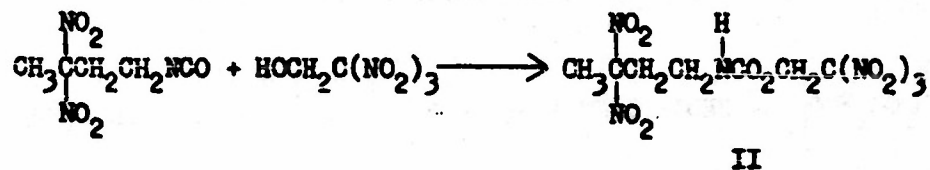
II Technical Progress, B (cont.)

Report No. 737

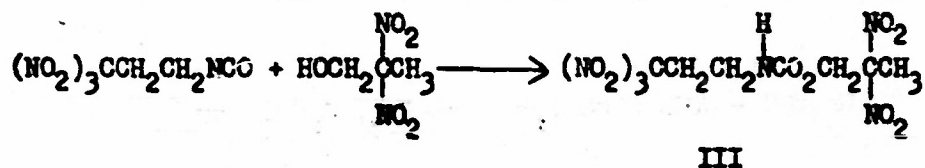
(1) 2,2,9,9-Tetranitro-4-oxa-5-keto-6-aza-decane
(I) from 3,3-dinitrobutyl isocyanate and 2,2-dinitropropanol:



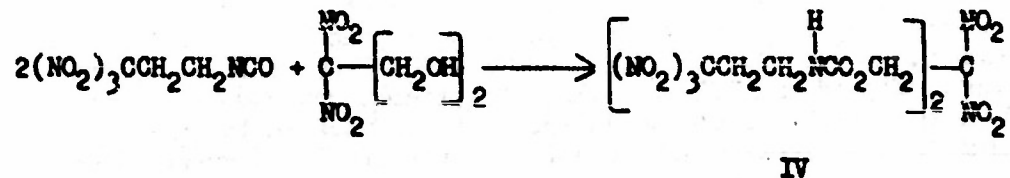
(2) 1,1,1,8,8-Pentanitro-3-oxa-4-keto-5-aza-nonane (II) from 3,3-dinitrobutyl isocyanate and 2,2,2-trinitroethanol:



(3) 2,2,9,9,9-Pentanitro-4-oxa-5-keto-6-aza-nonane (III) from 3,3,3-trinitropropyl isocyanate and 2,2-dinitropropanol:



(4) 1,1,1,8,8,15,15,15-Octanitro-4,12-diaza-5,11-diketo-6,10-dioxo-pentadecane (IV) from 3,3,3-trinitropropyl isocyanate and 2,2-dinitro-1,3-propanediol:

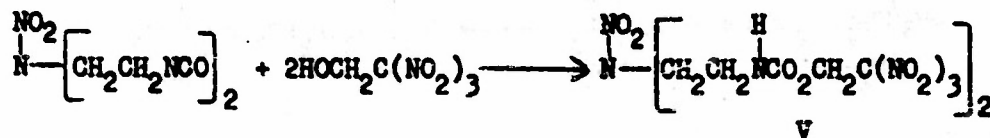


CONFIDENTIAL

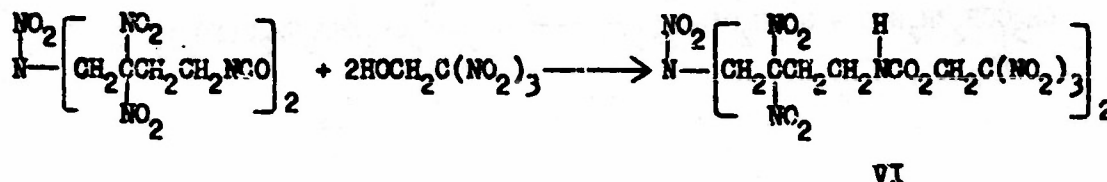
II Technical Progress, B (cont.)

Report No. 737

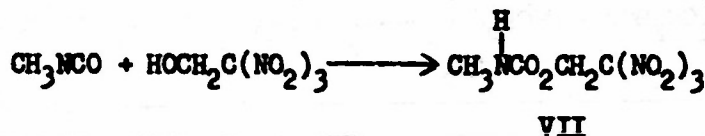
(5) 1,1,1,8,15,15,15-Heptanitro-3,13-dioxo-4,12-diketo-5,8,11-triasa-pentadecane (V) from 3-nitroso-1,5-pentane diisocyanate and 2,2,2-trinitroethanol:



(6) 1,1,1,8,8,10,12,12,19,19,19-Undecanitro-3,17-dioxo-4,16-diketo-5,10,15-triasa-nonadecane (VI) from 3,3,5,7,7-pentanitro-5-asa-1,9-nonane diisocyanate and 2,2,2-trinitroethanol:



(7) 1,1,1-Trinitro-3-oxo-4-keto-5-asa-hexane (VII) from methyl isocyanate* and 2,2,2-trinitroethanol:



2. Experimental

The general procedure employed was to reflux for about 8 hr equivalent quantities of the isocyanate and alcohol in dry, alcohol-free chloroform with a catalytic amount of ferric acetylacetonate. The solution was then evaporated to dryness in vacuo leaving a quantitative yield of product. Compounds III, V, and VI are white crystalline solids and Compounds I, II, IV and VII are oils. No attempt was made to purify the oils, inasmuch as they could be satisfactorily nitrated to give the desired postnitrated polynitrocarbamates. The results are summarized in Table I.

*Collucci, Can. J. Research 23B, 111 (1945).

II Technical Progress, B (cont.)

Report No. 737

TABLE I

ALIPHATIC POLYESTERCARBAMATES

Isocyanate	Alcohol	Polymer/carbamate	Yield %	Re-crystallization Solvent	Formula	Analyses	
						Calculated	Found
$\begin{matrix} \text{NO}_2 \\ \\ \text{CH}_2-\text{CH}_2-\text{CH}_2-\text{NCO} \\ \\ \text{NO}_2 \end{matrix}$	$\begin{matrix} \text{HOCH}_2(\text{CH}_2)_3\text{NO}_2 \\ \\ \text{NO}_2 \end{matrix}$	$\begin{matrix} \text{NO}_2 & \text{H} & \text{NO}_2 \\ & & \\ \text{CH}_2-\text{CH}_2-\text{CH}_2-\text{NCO}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{NCO}_2 \\ & & \\ \text{NO}_2 & & \text{NO}_2 \end{matrix}$	Quant. Oil	--	$\text{C}_9\text{H}_{13}\text{N}_2\text{O}_6$	--	--
$\begin{matrix} \text{NO}_2 \\ \\ \text{CH}_2-\text{CH}_2-\text{CH}_2-\text{NCO} \\ \\ \text{NO}_2 \end{matrix}$	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	$\begin{matrix} \text{NO}_2 & \text{H} & \text{NO}_2 \\ & & \\ \text{CH}_2-\text{CH}_2-\text{CH}_2-\text{NCO}_2-\text{CH}_2-\text{C}(\text{NO}_2)_3 \\ & & \\ \text{NO}_2 & & \text{NO}_2 \end{matrix}$	Quant. Oil	--	$\text{C}_7\text{H}_7\text{N}_2\text{O}_6$	--	--
$(\text{NO}_2)_3\text{CCH}_2\text{NCO}$	$\begin{matrix} \text{NO}_2 \\ \\ \text{HOCH}_2\text{CH}_2\text{NO}_2 \\ \\ \text{NO}_2 \end{matrix}$	$\begin{matrix} \text{NO}_2 & \text{H} & \text{NO}_2 \\ & & \\ (\text{NO}_2)_3\text{CCH}_2-\text{NCO}_2-\text{CH}_2-\text{CH}_2-\text{NCO}_2 \\ & & \\ \text{NO}_2 & & \text{NO}_2 \end{matrix}$	91.7	103-104 Chloroform	$\text{C}_7\text{H}_7\text{N}_2\text{O}_6$	23.31 23.31 23.33	22.98 22.80 22.90
$(\text{NO}_2)_3\text{CCH}_2\text{NCO}$	$\begin{matrix} \text{NO}_2 \\ \\ \text{C}(\text{CH}_2\text{NO}_2)_2 \\ \\ \text{NO}_2 \end{matrix}$	$\begin{matrix} \text{NO}_2 & \text{H} & \text{NO}_2 \\ & & \\ (\text{NO}_2)_3\text{CCH}_2-\text{NCO}_2-\text{CH}_2-\text{C}(\text{NO}_2)_2 \\ & & \\ \text{NO}_2 & & \text{NO}_2 \end{matrix}$	Quant. Oil	--	$\text{C}_{11}\text{H}_{11}\text{N}_2\text{O}_{10}$	--	--
$\begin{matrix} \text{NO}_2 \\ \\ \text{CH}_2-\text{CH}_2-\text{NCO} \\ \\ \text{NO}_2 \end{matrix}$	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	$\begin{matrix} \text{NO}_2 & \text{H} & \text{NO}_2 \\ & & \\ \text{CH}_2-\text{CH}_2-\text{NCO}_2-\text{CH}_2-\text{C}(\text{NO}_2)_3 \\ & & \\ \text{NO}_2 & & \text{NO}_2 \end{matrix}$	92.5	130-131 Styrene dichloride	$\text{C}_{10}\text{H}_{11}\text{N}_2\text{O}_8$	21.36 21.51 21.91	21.87 21.70 25.38
$\begin{matrix} \text{NO}_2 \\ \\ \text{CH}_2-\text{CH}_2-\text{NCO} \\ \\ \text{NO}_2 \end{matrix}$	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	$\begin{matrix} \text{NO}_2 & \text{H} & \text{NO}_2 \\ & & \\ \text{CH}_2-\text{CH}_2-\text{NCO}_2-\text{CH}_2-\text{C}(\text{NO}_2)_3 \\ & & \\ \text{NO}_2 & & \text{NO}_2 \end{matrix}$	91.3	157-159 Methanol-water	$\text{C}_{11}\text{H}_{13}\text{N}_2\text{O}_8$	21.06 21.27 21.56	21.34 21.59 25.37
CH_3NCO	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	$\begin{matrix} \text{NO}_2 & \text{H} & \text{NO}_2 \\ & & \\ \text{CH}_3-\text{NCO}_2-\text{CH}_2-\text{C}(\text{NO}_2)_3 \\ & & \\ \text{NO}_2 & & \text{NO}_2 \end{matrix}$	Quant. Oil	--	$\text{C}_4\text{H}_5\text{N}_2\text{O}_6$	--	--

CONFIDENTIAL

II Technical Progress (cont.)

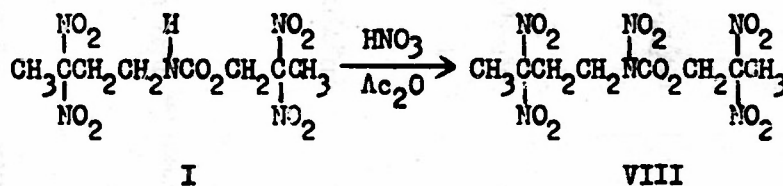
Report No. 737

C. PREPARATION OF POSTNITRATED ALIPHATIC POLYNITROCARBAMATES

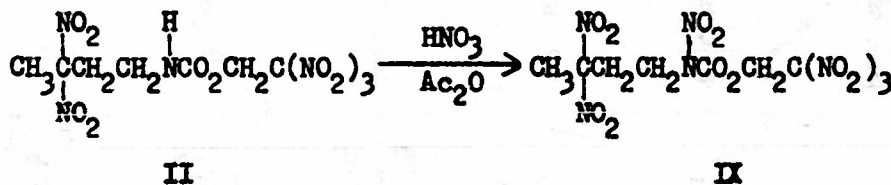
1. Discussion

a. The following postnitrated aliphatic polynitrocarbammates were prepared by the nitration of the nitrocarbammates described in Section I, B with a mixture of 100% nitric acid and acetic anhydride at 5 to 10°C.

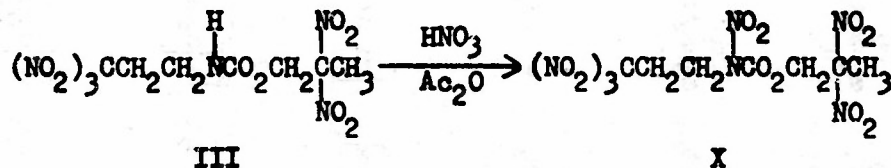
(VIII): (1) 2,2,6,9,9-Pentanitro-4-oxa-5-keto-6-aza-decane



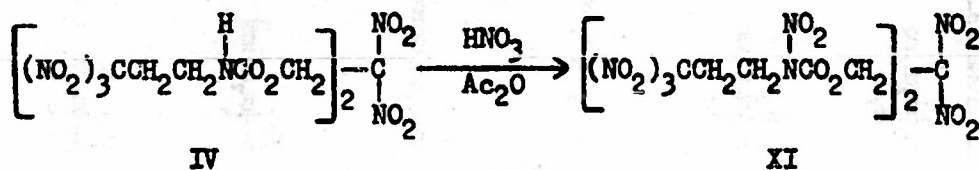
(IX): (2) 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane (IX):



(3) 2,2,6,9,9,9-Hexanitro-4-oxa-5-keto-6-aza-nonane (X):



(4) 1,1,1,4,8,8,12,15,15,15-Decanitro-4,12-diaza-5,11-diketo-6,10-dioxa-pentadecane (XI):

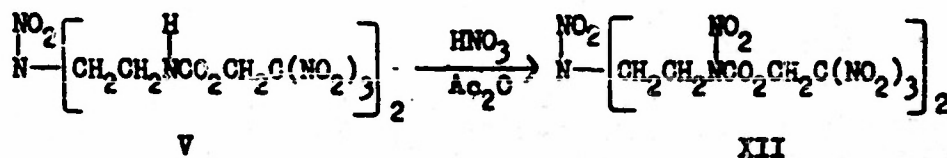


CONFIDENTIAL

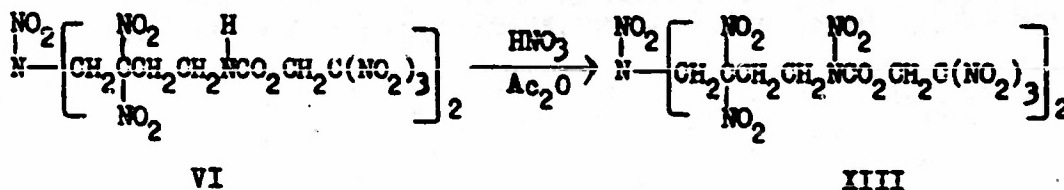
II Technical Progress, C (cont.)

Report No. 737

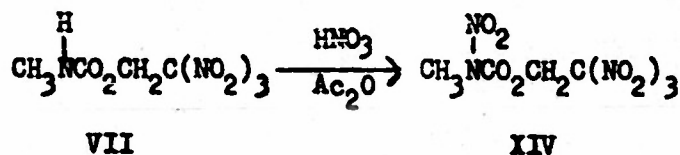
(5) 1,1,1,5,8,11,15,15-Nonanitro-3,13-dioxo-4,12-diketo-5,8,11-triaza-pentadecane (XII):



(6) 1,1,1,5,8,8,10,12,12,15,19,19,19-Tridecanitro-3,17-dioxo-4,16-diketo-5,10,15-triaza-nonadecane (XIII):



(7) 1,1,1,5-Tetranitro-3-oxo-4-keto-5-azahexane (XIV):



2. Experimental

a. The general procedure used was to cool the 100% nitric acid to 0 to 5°C and add the acetic anhydride dropwise, keeping the temperature below 10°C. The solid nitrocarbamate was added portionwise at 5 to 10°C. (Ten ml of 100% nitric acid and 10 ml of acetic anhydride was used per gram of nitrocarbamate.) The solid dissolved readily, and the solution was stirred for 20 min at 5 to 10°C and poured on ice. The white solid was collected, washed with water, and dried in vacuo over potassium hydroxide.

b. In the instances in which the nitrocarbamate was an oil (Compounds I, II, IV, and VII), the oil was dissolved in the acetic anhydride and the solution was added dropwise to the 100% nitric acid, keeping the temperature at 5 to 10°C.

CONFIDENTIAL

II Technical Progress, C (cont.)

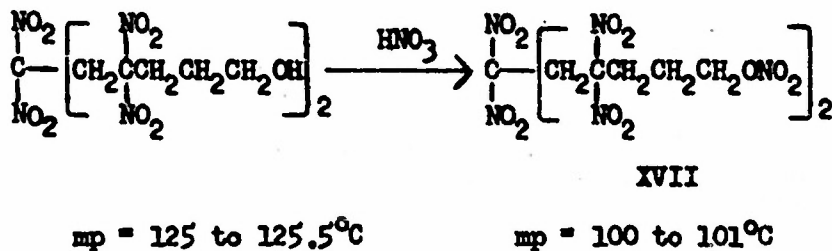
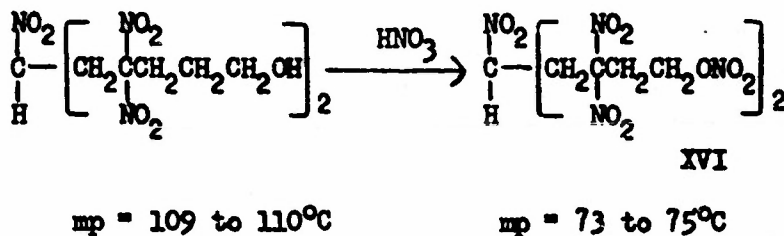
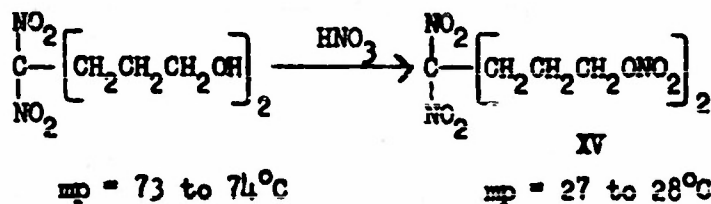
Report No. 737

c. The experimental results are summarized in Table II.

D. PREPARATION OF ALIPHATIC POLYNITRONITRATES

1. Discussion

a. A number of polynitrodiols have been synthesized on the nitropolymer program as intermediates for the preparation of condensation polymers.* It was of interest to the explosives program to convert these polynitrodiols to the corresponding polynitrodinitrates and evaluate these compounds as high explosives. Accordingly, the following nitrates were prepared: 4,4-dinitro-1,7-heptane-dinitrate (XV), 4,4,6,8,8-pentanitro-1,11-undecanedinitrate (XVI), 4,4,6,6,8,8-hexanitro-1,11-undecanedinitrate (XVII), and 4,4,6,8,8-pentanitro-6-aza-1,11-undecanedinitrate (XVIII).



*Aerojet Reports No. 563, 638.

TABLE II

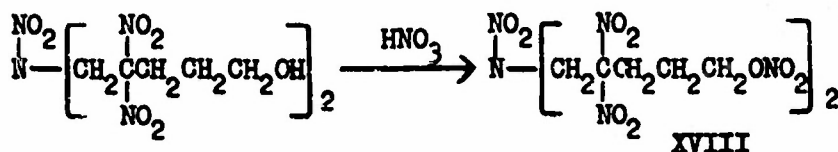
POSTNITRATED ALIPHATIC POLYESTERCARBONATES

Postnitrated Polyester Carbonate	Yield %	MP, °C	Recrystallization Solvent	Formula	Analyses				Heat of Combustion cal/g	Calculated Lead-Block Value (PDX = 157)	Calculated Ballistic Mortar Value (RDX = 150)			
					Calculated C	Calculated H	Found C	Found H						
$\text{CH}_3\text{C}(\text{NO}_2)\text{CH}_2\text{CH}_2\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)\text{CH}_3$	52.8	70-72	Chloroform	$\text{C}_8\text{H}_{12}\text{N}_2\text{O}_6$	25.01	3.15	21.88	25.96	3.36	21.73	2666	2643	117	126
$\text{CH}_3\text{C}(\text{NO}_2)\text{CH}_2\text{CH}_2\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)\text{CH}_3$	10.5	107-108	Chloroform	$\text{C}_7\text{H}_9\text{N}_2\text{O}_6$	20.25	2.19	23.62	20.26	2.21	23.67	2032	2007	153	140
$(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$	83.7	71-72	Chloroform	$\text{C}_7\text{H}_9\text{N}_2\text{O}_6$	20.25	2.19	21.62	20.51	2.22	24.05	2033	2051	153	140
$[(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{CO}_2\text{CH}_2]_2\text{C}(\text{NO}_2)_2$	89.7	94-96	Chloroform	$\text{C}_{11}\text{H}_{12}\text{N}_2\text{O}_{14}$	18.97	1.74	24.14	19.29	2.00	24.21	1704	1834	164	132
$\text{H} \left[\text{CH}_2\text{CH}_2\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)_3 \right]_2$	86.3	120-122	Ethylene dichloride	$\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_{22}$	18.41	1.86	25.77	18.98	2.23	25.87	1792	1826	162	140
$\text{H} \left[\text{CH}_2\text{CH}_2\text{CH}_2\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)_3 \right]_2$	53.3	150-151	Ethylene dichloride	$\text{C}_{11}\text{H}_{16}\text{N}_2\text{O}_{30}$	18.93	1.82	25.23	19.12	1.84	25.19	1857	1958 1946	164	142
$\text{CH}_3\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$	74.3	39-40	Diisopropyl ether	$\text{C}_4\text{H}_5\text{N}_2\text{O}_{10}$	16.97	1.78	24.74	17.29	1.86	24.91	—	—	168	137

CONFIDENTIAL

II Technical Progress, D (cont.)

Report No. 737



mp = 106 to 108°C

mp = 133 to 135°C

b. Generally nitrates have a lower melting point than the corresponding alcohols. This is confirmed by the behavior of Compounds XV, XVI, and XVII, which melt 25 to 36 degrees lower than the diols from which they were prepared. However, Compound XVIII melts 27 degrees higher than the corresponding diol, a significant factor which must be attributed to the presence of the nitramino group. This is of interest because it is believed that there is a correlation between the melting point and thermal stability of an explosive, the higher-melting compound being more stable provided that other factors are the same.

c. Nitration of 3,3-dinitro-1,5-pentanediol (mp 71 to 72°C), 2,2,4,4,-tetranitro-1,5-pentanediol (mp 97 to 99°C), and 2,2-dinitro-1,3-propanediol (mp ca. 140°C) gave oils which were not investigated further.

2. Experimental

The general procedure was to add the solid polynitrodiol by portions to a tenfold excess of 100% commercial nitric acid at ice-bath temperature. The acid solution was stirred for 15 min at 5 to 10°C and poured on ice. The white solid was collected, washed with water, and dried in vacuo over potassium hydroxide. The results are summarized in Table III.

TABLE III

ALIPHATIC POLYNITRONITRATES

Compound	Yield %	mp, °C	Recrystallisation Solvent	Formula	Analyses						Heat of Combustion cal/g		Calculated Lead-Block Value (RDX = 157)	Calculated Ballistic Motor Value (RDX = 150)
					Calculated			Found			Calcd	Found		
					C	H	N	C	H	N				
$\text{N} \begin{array}{c} \text{NO}_2 \\ \\ \left[\text{CH}_2 \text{C} \begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{ONO}_2 \\ \\ \text{NO}_2 \end{array} \right]_2 \end{array}$	94.7	27-28	Methanol	$\text{C}_7\text{H}_{12}\text{N}_4\text{O}_{10}$	26.93	3.88	17.95	27.17	4.15	18.60	3103	3015	120	132
$\text{N} \begin{array}{c} \text{NO}_2 \\ \\ \left[\text{CH}_2 \text{C} \begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{ONO}_2 \\ \\ \text{NO}_2 \end{array} \right]_2 \end{array}$	95.1	73-75	Methanol	$\text{C}_{11}\text{H}_{17}\text{N}_7\text{O}_{16}$	26.25	3.41	19.48	26.79	3.64	19.18	—	—	126	137
$\text{N} \begin{array}{c} \text{NO}_2 \\ \\ \left[\text{CH}_2 \text{C} \begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{ONO}_2 \\ \\ \text{NO}_2 \end{array} \right]_2 \end{array}$	95.2	100-102	Methanol	$\text{C}_{11}\text{H}_{16}\text{N}_8\text{O}_{18}$	24.09	2.94	20.46	24.54	2.99	19.91	2643	2600	138	143
$\text{N} \begin{array}{c} \text{NO}_2 \\ \\ \left[\text{CH}_2 \text{C} \begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{ONO}_2 \\ \\ \text{NO}_2 \end{array} \right]_2 \end{array}$	95.0	133-135	Methanol	$\text{C}_{11}\text{H}_{16}\text{N}_8\text{O}_{18}$	23.82	3.20	22.22	23.88	2.96	22.15	2727	2726	135	142

CONFIDENTIAL

II Technical Progress (cont.)

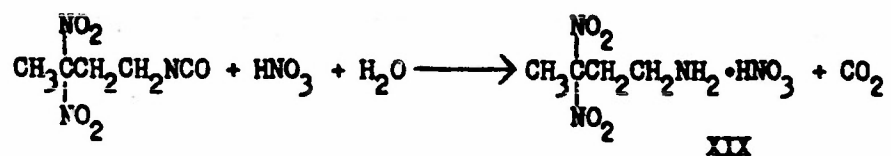
Report No. 737

E. PREPARATION OF ALIPHATIC POLYNITROAMINE SALTS

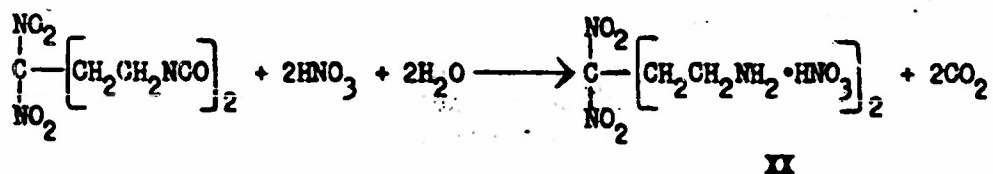
1. Discussion

a. Treatment of 3,3,3-trinitropropyl isocyanate with 35% nitric acid readily gave a quantitative yield of 3,3,3-trinitropropyl ammonium nitrate.* This nitrate salt was of interest for evaluation as a potential high explosive and also as an intermediate for the production of Mannich condensation products. This work suggested the conversion of the isocyanates prepared on the nitropolymer program to the corresponding nitrate salts. Accordingly, the following nitrate salts were prepared:

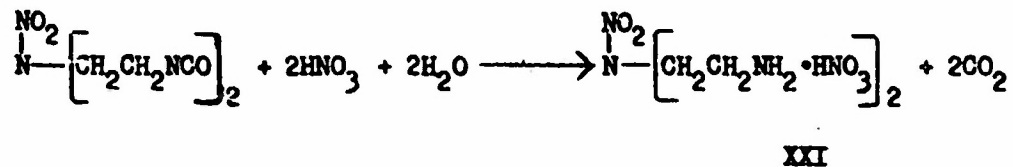
(1) 3,3-Dinitrobutyl Ammonium Nitrate (XIX):



(2) 3,3-Dinitro-1,5-Pentane Diammonium Dinitrate (XX):



(3) 3-Nitroso-1,5-Pentane Diammonium Dinitrate (XXI):



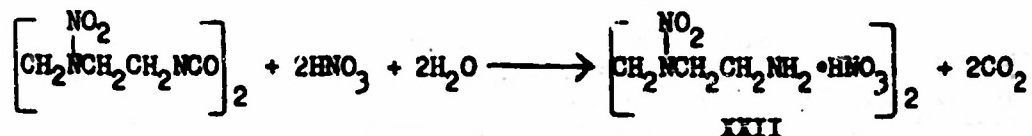
*Aerojet Report No. 682, p. 9.

CONFIDENTIAL

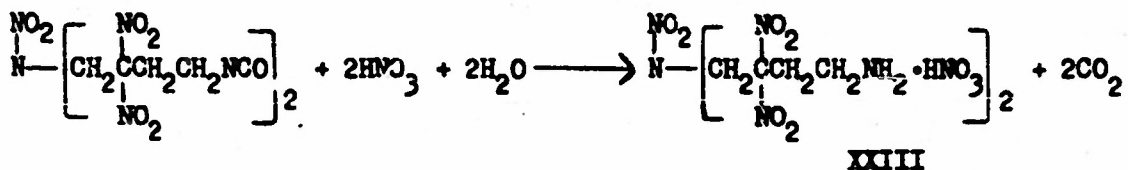
II Technical Progress, E (cont.)

Report No. 737

(4) 3,6-Dinitro-1,8-Octane Diammonium Dinitrate (XXII):



(5) 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane Diammonium Dinitrate (XXIII):



The 3,3,5,7,7-pentanitro-5-aza-1,9-nonane diammonium dihydrochloride (XXIV) was also prepared.

2. Experimental

The general procedure was to heat the isocyanate with a tenfold to fifteenfold excess of 35% nitric acid or concentrated hydrochloric acid on the steam bath for 3 hr. When the solution was cooled, or concentrated if necessary, a quantitative yield of the polynitroamine salt separated. The results are summarized in Table IV.

F. PREPARATION OF SAMPLES FOR EVALUATION BY THE NAVAL ORDNANCE LABORATORY

1. The following samples were prepared and submitted to the Naval Ordnance Laboratory for evaluation:

- a. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane
- b. 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diaza-decane
- c. 4,4,6,8,8-Pentanitro-1,11-undecane-dinitrate
- d. 4,4,6,6,8,8-Hexanitro-1,11-undecane-dinitrate
- e. 4,4,6,8,8-Pentanitro-6-aza-1,11-undecane-dinitrate
- f. 2,2,6,9,9-Pentanitro-4-oxa-5-keto-6-aza-decane

TABLE IV

ALIPHATIC POLINITROAMINE SALTS

Compound	M.P., °C	Recrystalliz. Solvent	Formula	Analyzes						Heat of Combustion cal/g	Calculated Lead-Block Value (RDP-157)	Calculated Ballistic Mortar Value (RDP-150)				
				Calculated			Found									
				C	H	N	O	C	H	N	O	Calcd Found (RDP-157)				
$\begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3 \\ \\ \text{NO}_2 \end{array}$	131-133	Methanol	$\text{C}_4\text{H}_{10}\text{N}_2\text{O}_7$	21.24	4.46	24.78	—	21.39	4.63	25.19	—	2725	2629	112	116	
$\begin{array}{c} \text{NO}_2 \\ \\ \text{C} - \text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3 \\ \\ \text{NO}_2 \end{array}$	151-153	Methanol	$\text{C}_5\text{H}_{14}\text{N}_2\text{O}_6$	18.87	4.43	26.41	—	19.07	4.70	26.22	—	2493	2390	120	130	
$\begin{array}{c} \text{NO}_2 \\ \\ \text{N} - \text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3 \\ \\ \text{NO}_2 \end{array}$	153-155	Methanol-water	$\text{C}_4\text{H}_{11}\text{N}_2\text{O}_6$	17.52	5.15	30.65	—	17.66	5.11	30.78	—	2526	2516	112	126	
$\begin{array}{c} \text{NO}_2 \\ \\ \text{N} - \text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3 \\ \\ \text{NO}_2 \end{array}$	185-190	Methanol-water	$\text{C}_6\text{H}_{16}\text{N}_2\text{O}_6$	19.89	5.01	30.93	—	20.56	5.01	30.78	—	2519	2816	109	125	
$\begin{array}{c} \text{NO}_2 \\ \\ \text{N} - \text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3 \\ \\ \text{NO}_2 \end{array}$	175-180	Concentrated nitric acid	$\text{C}_8\text{H}_{18}\text{N}_2\text{O}_{16}$	18.83	3.56	27.45	—	18.95	3.88	27.10	—	—	—	135	144	
$\begin{array}{c} \text{NO}_2 \\ \\ \text{N} - \text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3 \\ \\ \text{NO}_2 \end{array}$	173-178	Methanol-water	$\text{C}_8\text{H}_{18}\text{N}_2\text{O}_{12}$	21.02	3.97	24.51	15.51	20.51	4.47	25.40	14.97	—	—	—	—	—

CONFIDENTIAL

II Technical Progress, F (cont.)

Report No. 737

- g. 2,2,6,9,9,9-Hexanitro-4-oxa-5-keto-6-aza-nonane
- h. 1,1,1,4,8,8,12,15,15,15-Decanitro-4,12-diaza-5,11-diketo-6,10-dioxa-pentadecane
- i. 1,1,1,4,13,16,16,16-Octanitro-4,13-diaza-5,12-diketo-6,11-dioxa-8-hexadecyne
- j. 1,1,1,5,8,11,15,15,15-Nonanitro-3,13-dioxa-4,12-diketo-5,8,11-triaza-pentadecane
- k. 1,1,1,5,8,8,8,10,12,12,15,19,19,19-Tridecanitro-3,17-dioxa-4,16-diketo-5,10,15-triaza-nonadecane
- l. 3,3-Dinitrobutyl ammonium nitrate
- m. 3,3-Dinitro-1,5-pentane diammonium dinitrate
- n. 3-Nitrazo-1,5-pentane diammonium dinitrate
- o. 3,6-Dinitrazo-1,8-octane diammonium dinitrate
- p. 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane diammonium dinitrate

2. The results of the preliminary tests by the Naval Ordnance Laboratory on Compounds A53-11 to A53-21 are summarized in Table V. As with most of the polynitrocarbamates, these compounds have good thermal stability and fair to good density.

3. SPIA data sheets, which are presented in the appendix, have been completed on the following compounds:

- a. 1,3-bis(3',3',3'-Trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine
- b. 1,3,5-tris(3',3',3'-Trinitropropyl)-hexahydro-1,3,5-triazine
- c. 2,2,5,7,7,9,12,12-Octanitro-5,9-diaza-tridecane
- d. 2,2,4,7,7,10,12,12-Octanitro-4,10-diaza-tridecane
- e. N',N'-bis(3,3,3-Trinitropropyl) oxamide
- f. N,N'-Dinitro-N,N'-bis(3,3,3-trinitropropyl) oxamide
- g. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane
- h. 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diaza-decane

TABLE V
MCL PRELIMINARY TESTS ON HIGH EXPLOSIVES

Compound	mp. °C	T ₈₀ sec/2.5 hr	Cryst. Dens. g/cm ³	Ignition Temp °C	Thermal Stability (Gas Vol/g at 100°C)			Calculated Lead-Block Value (DIX = 157)	Calculated Ballistic-Mortar Value (DIX = 150)
					45 hr	72 hr	180 hr		
$\text{CH}_3\text{C}(\text{NO}_2)_2\text{CH}_2\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ A53-11	107-108	15 (Class 5)	1.733	221	0.50 (Class I)	—	—	153	140
$\text{CH}_3\text{C}(\text{NO}_2)_2\text{CH}_2\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ A53-12	140-141	18 (Class 5)	1.790	212	1.01 (Class I)	—	—	167	144
$\text{H}-\left[\text{C}(\text{NO}_2)_2\text{CH}_2\text{C}(\text{NO}_2)_2\text{CH}_2\text{CH}_2\text{ONO}_2\right]_2$ A53-13	73-75	34 (Class 4)	—	—	—	—	—	126	137
$\text{H}-\left[\text{C}(\text{NO}_2)_2\text{CH}_2\text{C}(\text{NO}_2)_2\text{CH}_2\text{CH}_2\text{ONO}_2\right]_2$ A53-14	100-102	28 (Class 4)	—	—	—	—	—	138	144
$\text{H}-\left[\text{C}(\text{NO}_2)_2\text{CH}_2\text{C}(\text{NO}_2)_2\text{CH}_2\text{CH}_2\text{ONO}_2\right]_2$ A53-15	133-135	85 (Class 2)	—	—	—	—	—	135	142
$\text{CH}_3\text{C}(\text{NO}_2)_2\text{CH}_2\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ A53-16	70-72	47 (Class 3)	1.61	293	(Class II)	—	—	117	126
$(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{CO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ A53-17	66-68	16 (Class 5)	—	229	(Class III)	—	—	153	140
$\text{H}-\left[\text{C}(\text{NO}_2)_2\text{CH}_2\text{C}(\text{NO}_2)_2\text{CH}_2\text{C}(\text{NO}_2)_3\right]_2$ A53-18	94-96	20 (Class 5)	1.74	212	5.0 (Class II)	9.2	27.2	164	132
$\left[(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{CO}_2\text{CF}_2\text{C}(\text{NO}_2)_2\right]_2$ A53-19	110-111	16 (Class 5)	1.65	192	14 (Class III)	—	—	137	134
$\text{H}-\left[\text{C}(\text{NO}_2)_2\text{CH}_2\text{C}(\text{NO}_2)_2\text{CH}_2\text{C}(\text{NO}_2)_3\right]_2$ A53-20	120-122	17 (Class 5)	1.77	220	1.1 (Class I)	1.6	3.5	162	140
$\text{H}-\left[\text{C}(\text{NO}_2)_2\text{CH}_2\text{C}(\text{NO}_2)_2\text{CH}_2\text{C}(\text{NO}_2)_3\right]_2$ A53-21	150-151	12 (Class 5)	1.76	217	1.4 (Class I)	1.9	3.3	154	142

CONFIDENTIAL

Report No. 737

III. DESENSITIZATION OF RDX

A. INVESTIGATION OF COMBINATIONS OF POLYDINITROBUTYL ACRYLATE, PLASTICIZERS, AND SURFACE-ACTIVE AGENTS

1. Discussion

a. In the investigation of new coatings for RDX by the method of preparing composition A, a systematic test of various combinations of a plasticizer and a surface active agent with polydinitrobutyl acrylate is in progress. This study has a dual purpose:

(1) Correlation of the compatibility of such combinations with their desensitizing effect.

(2) Pointing out new, promising combinations for further consideration.

For the plasticizers, four commercially used compounds, along with 2,2-dinitropropane, were chosen. The surface-active agents were seven ester-type materials selected as the best of the many agents tested in the past. Each combination was made up and tested for compatibility. As the next step, specimens of RDX coated with the same combinations are being prepared, and their impact stabilities determined.

b. Every attempt was made to insure uniform, reproducible conditions. Fixed proportions were used throughout. The polymer had been worked up by reprecipitating, to eliminate light fractions that can themselves provide a plasticizing action.

c. Present results are too sketchy for correlation of compatibility with desensitizing action. With respect to the second purpose, these results have already confirmed the softening action of several plasticizers in improving the efficacy of polydinitrobutyl acrylate coating. The results have also confirmed earlier observations* that some alternate surface-active agents are as good as or better than Span 85.

d. Some further experiments were performed with the aim of increasing the softness of the coating and at the same time increasing its oxygen balance (thus increasing explosive strength). Using only Atlas G-3785 surface-active agent, these experiments employed the copolymer of dinitrobutyl acrylate and mono nitrobutyl acrylate for increased softness, and also varied the amounts of the constituents. These experiments show the possibility of appreciably raising the oxygen balance with increased amounts of 2,2-dinitropropane. They also indicate that 1% of G-3785 surface-active agent performs as well as 2.5%, and has the advantage of higher oxygen balance. However, none of the coating formulations gave as good desensitization as is attained with Composition A.

*Aerojet Report No. 682, et seq.

CONFIDENTIAL

III Desensitisation of RDX, A (cont.)

Report No. 737

2. Experimental

a. Specimens composed of polydinitrobutyl acrylate, plasticizer, and surface-active agent were made up in the ratio 8:2:3.3. This ratio gives a content of surface-active agent slightly in excess of the amount ordinarily used in coating; the actual coating compositions are thus bracketed between specimens containing surface-active agents and those containing none. These specimens were dissolved in acetone and the solutions were dropped on a glass plate. The solvent was evaporated and the residual films were inspected and compared. The three qualities selected for rating the films were opacity, separation, and fragility. The results are given in Table VI.

b. A parallel experiment was begun, in which RDX is coated with the same combinations of polydinitrobutyl acrylate, plasticizer, and surface-active agent in the ratio 8:2:2.5% RDX. The procedure was the previously described adaptation of the process for making Composition A,* except that the amount of RDX used was reduced to 4.0 g, and the other amounts were reduced proportionately. The products were evaluated by measuring impact stabilities on the Bureau of Mines apparatus with the 2-kg weight, using the bare anvil and also (sometimes) 5/0 sandpaper. Results of these tests are shown in Table VII.

c. Some additional coating tests were performed using similar materials but falling outside the experimental pattern described above. Results of these tests are given in Table VIII. Oxygen balances of these formulations were calculated, and are also tabulated here.

d. All polymers used had been thoroughly cured, and then worked up by reprecipitating twice from acetone solution in methanol. This treatment removed fractions of low molecular weight.

*Aerojet Report No. 682, p. 21.

CONFIDENTIAL

III Desensitization of EDX, A (cont.)

Report No. 737

TABLE VI

COMPATIBILITIES OF SYSTEMS OF POLYDINITROBUTYL
ACRYLATE, PLASTICIZER, AND SURFACE-ACTIVE AGENT

Films contain 8 parts PDNBA
2 parts plasticizer
3.3 parts surface-active agent

Note: All samples were inspected for opacity (first column of numerals); separation (second column); and fragility (third column), and rated by a 1-2-3 system, where 1 denotes least, 2 medium, and 3 most.

Plasti- cizer	Surface-Active Agent							
	--	Span 85	Glyc. Mono O	Prosol 307	G-949	G-1077	G-2135	G-3785
--	1,1,3	2,3,3	3,2,3	3,1,3	2,3,3	3,2,3	3,2,3	3,1,3
DOS	3,1,3	3,3,3	3,3,3	3,3,3	2,3,3	3,2,3	3,3,3	3,1,3
DBS	3,1,1	3,3,3	3,2,3	3,2,3	2,3,3	3,2,3	3,2,3	3,2,1
DOP	2,1,2	2,3,3	3,2,3	3,2,3	2,3,3	3,2,3	3,2,3	3,2,3
TCP	1,1,1	2,3,2	1,1,3	1,2,3	1,3,2	2,2,3	3,2,2	3,1,2
DNP	1,1,2	2,3,2	3,2,2	3,1,2	1,3,3	3,2,2	3,2,2	3,1,3

DOS is dioctyl sebacate
DBS is dibutyl sebacate
DOP is dioctyl phthalate
TCP is tricresyl phosphate
DNP is 2,2-dinitropropane

CONFIDENTIAL

III Desensitization of RDX, A (cont.)

Report No. 737

TABLE VII

**IMPACT STABILITIES OF SYSTEMS COMPOSED OF POLYDINITROBUTYL
ACRYLATE, PLASTICIZER, AND SURFACE-ACTIVE AGENT**

Results in parentheses determined on 5/0
sandpaper; others on bare anvil.

Plasti- cizer	Impact Stability of Systems Containing The Following Surface-Active Agents, cm/2 kg							
	Span 85	Glyc. Mono O	Prosol 307	G-949	G-1077	G-2139	G-3785	
--	35-40	85	--	>100 to 45-50*	--	--	--	90-100
DOS	--	--	--	--	--	--	--	--
DBS	60-75	>100	--	--	--	--	--	>100 (15-20)
DOP	--	--	--	--	--	--	--	--
TCP	80-85	75-80	<55	--	--	--	--	>95
DNP	50-55	>100	--	55-60	--	75-80	--	>95, >100

Polydinitrobutyl acrylate, plasticizer, and surface active agent in the
ratio 8:2:2.5% RDX.

Reference: RDX = 40 (10-15)
Comp. A-3, > 100 (45-50)

*Large variations among separate preparations.

CONFIDENTIAL

III Desensitization of RDX, A (cont.)

Report No. 737

TABLE VIII

MISCELLANEOUS DESENSITIZATION EXPERIMENTS

Run	Coating	Oxygen* Balance	I.S., cm/2 kg	
			Bare	On paper
6/18/1	5% PDNBA/5% DNP/1% G-3785	-29	>100	10-15
6/18/2	5% PDNBA/5% DNP/2.5% G-3785	-32	>100	10-15
6/18/3	7.5% PDNBA/7.5% DNP/2.5% G-3785	-34	>100	10-15
6/5/1	8% Copolymer/2% DBS/2.5% G-3785	-37	>100	20-25
6/19/1	6% Copolymer/2% DBS/2% DNP/G-3785	-34	>100	25
	RDX	-22	40	10-15
	Comp. A-3	-43	>100	40-45

Copolymer made from 50% dinitrobutyl acrylate and 50% mono-nitrobutyl acrylate.

*Oxygen balances calculated by assuming a reasonable average formula for all surface-active agents.

CONFIDENTIAL

III Desensitization of RDX (cont.)

Report No. 737

B. SURFACE-ACTIVE AGENTS CONTAINING NITRO GROUPS

1. Discussion

a. Previous trials of many surface-active agents for use in coating RDX have indicated that certain agents are most efficacious.* These had the common feature of being esters of polyols and oleic acid, or other long-chain fatty acids. In fact, such agents by themselves can provide good temporary desensitization (temporary because of the drainage of liquid coatings). These results suggested the use of similar compounds containing substituted nitro groups, either as surface-active agents or as coatings per se.

b. The first compound whose synthesis was attempted was the monocarbamate of 3,3,3-trinitropropyl isocyanate and glycerol mono oleate; this product was a viscous liquid which could not be crystallized. This circumstance, along with the mixed nature of the original oleate, might well lead to a mixed product, and analysis confirmed that the product was considerably different from the simple monocarbamate. The product was subjected to the adsorption test described in Report No. 682, in which the amount adsorbed on RDX from an acetone-water system is measured under standardized conditions. Next, a coating experiment was performed, using the method of preparation of Composition A.** Application of 10% of the product alone gave a coated specimen whose impact stability was approximately as good as that of Composition A. When used as a surface active agent, in conjunction with polydinitrobutyl acrylate coating, the product was comparable to the agents now in use.

c. The preparation of chemically similar solid compounds will be attempted next.

2. Experimental

a. The reaction between glycerol mono oleate and 3,3,3-trinitropropyl isocyanate was carried out in the same manner as described in II,B,2. The product was a viscous liquid:

Anal. Calc'd for monocarbamate $C_{25}H_{43}N_4O_{11}$: %C, 52.16; %H, 7.53; %N, 9.74

Found: %C, 70.44; %H, 7.54; %N, 8.55

*Aerojet Reports No. 589, 682.

**Aerojet Report No. 682, p. 21.

CONFIDENTIAL

III Desensitization of RDX, B (cont.)

Report No. 737

b. The product was subjected to the standard adsorption test for surface-active agent. Results, compared with the values for Span 85, which is known to be a good surface-active agent, are shown below:

Adsorption of 0.15 g Agent on 3.00 g RDX from a Medium
of 5 ml Acetone and 10 ml Water

	<u>Appearance of Agent in Medium</u>	<u>% Adsorbed</u>	<u>Impact Stab.,* cm/2 kg</u>
Carbamate	Suspension and oil layer	81	65-75
Span 85	Suspension	52-62	>100**

*For carbamate alone, >100; for RDX, 30.

**Value for RDX from a different lot.

c. The new material may be too oily, from its behavior in the test; the coating on the RDX was noticed to be somewhat uneven. This degree of desensitization is fair though not as good, per unit amount adsorbed, as that obtained with the best surface-active agents described in Report No. 682.

d. Using the standard coating procedure, 10% of this product was applied to RDX. In another experiment it was used in conjunction with polydinitrobutyl acrylate, replacing both the plasticiser and the surface-active agent used in the systematic experiments described in the preceding section. Impact stabilities of the products were measured; the results are:

<u>Coating</u>	<u>Impact Stability, cm/2 kg</u>	
	<u>Bare</u>	<u>On Paper</u>
10% Carbamate	>100	40
8% PDNBA/4.5% carbamate	>100	20-25
— RDX	40	10-15
— Comp. A-3	>100	40-45

e. The RDX used in these experiments was Wabash L-119.

CONFIDENTIAL

Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8631 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,3-bis(3',3',3'-Trinitropropyl)-

Name 5,5-dinitro-hexahydro-1,3-diazine

Empirical formula (C₁₀H₁₂N₄O₁₆)

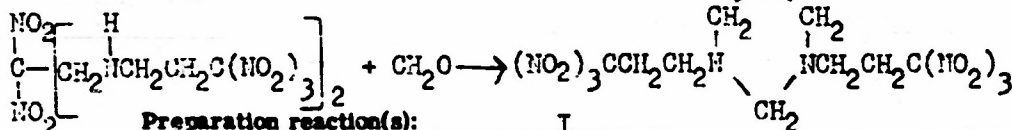
Structure: (configuration) I

Information submitted by:

Activity Aerojet-General Corporation

Person M. B. Frankel and L. T. Garleton

Date 1 August 1953



Preparation reaction(s): I

1. Quantitative analysis:(% by weight)	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	22.65	2.66		26.42		
By determination	22.95	2.65		26.72		

2. Burning properties:(compared to nitrocellulose, under nitrogen at atmospheric pressure:
(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet. Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL OSRD 3401 p.8	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL OSRD 3401 p.8	7 hr at 80°C
d. Temperature of Explosion	OSRD 3401 p.6	
e. Temperature of Ignition	NOL OSRD 3401 p.8	
f. Impact Stability	Bureau of Mines Bull. No. 346, p. 72 (2-kg weight)	
g.		
h.		

RESULTS OF ABOVE TESTS

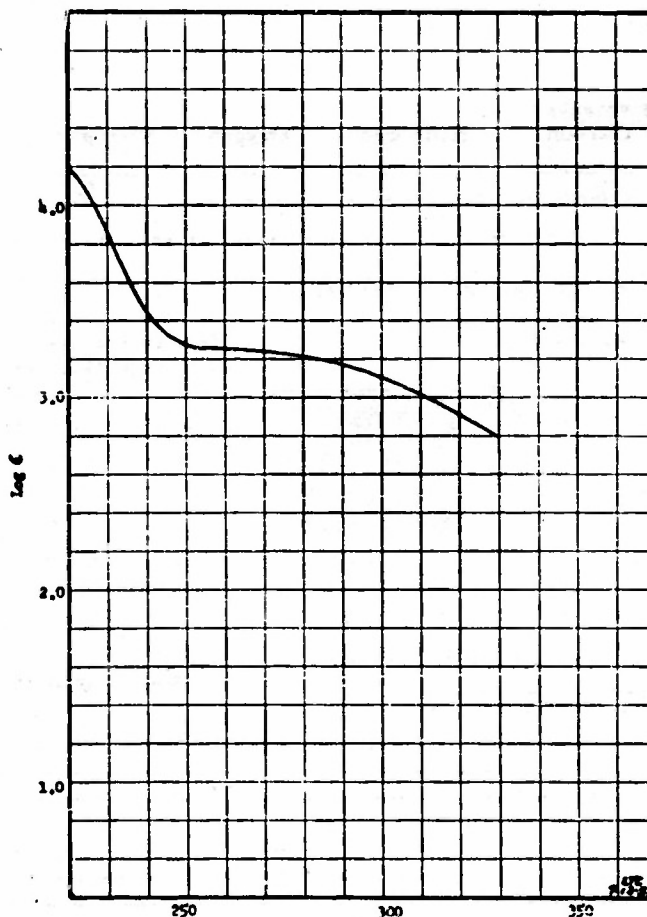
Reference compound (designation-TNT, Tetryl, H.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	20 cm/2.5 kg
b.	
c.	>30 cc/g
d.	
e.	129°C
f. BDX, 30 cm/2 kg	25-30 cm/2 kg
g.	
h.	

4. Heat of formation: (ΔH) ± -20 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

CONFIDENTIAL

Report No. 737
SPIA/MS

- | | By Experiment | By Calculation | Method |
|---|--|-----------------|--|
| 5. Energy of explosion (Q)
(at 25°C. H ₂ O liquid) | _____ | cal/gm _____ | Description or reference. Separate sheet if necessary. |
| 6. Heat of combustion (H _c)
(at 25°C. H ₂ O liquid) | 2637 | cal/gm 2637 | Aerojet Report No. 417A |
| 7. Specific impulse (I _{sp}) calc: | _____ | lb-sec/lb _____ | |
| 8. Physical form of compound (viscous liquid, crystalline type, etc.) | needle-shaped crystals | | |
| 9. Simple microscope analysis data:
(crystal studies) | _____ | | |
| 10. Density (micro method) $\frac{1.683}{(NOL)}$ gm/cm ³ .
(Micro or other method) _____ gm/cm ³ .
(Indicate on separate sheet any unique methods you use.) | | | |
| 11. Index of refraction: ($n_D^{25^\circ C}$) _____ | 12. Color Yellow | 13. Odor None | |
| 14. pH at 25°C. 2.1
(Method reference OSRD 3401 v.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator used or Beckman pH meter.) | With Beckman pH meter, 0.054 M in acetone/water (5/1 volume ratio) | | |



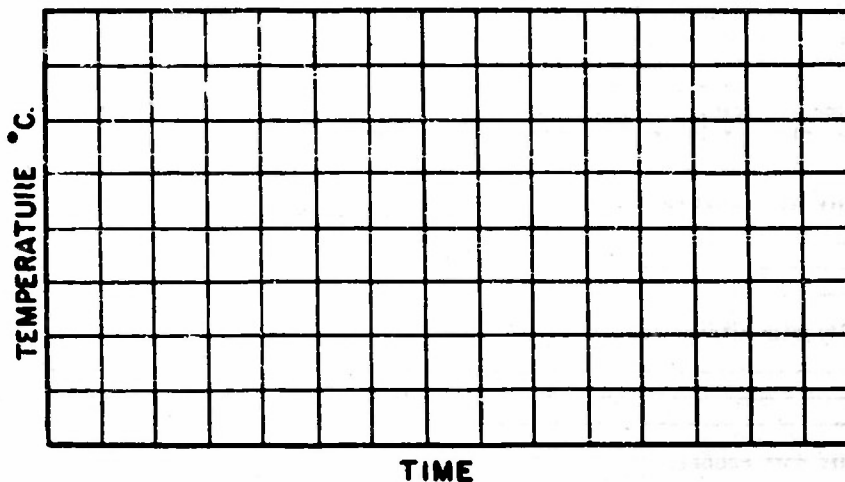
Ultraviolet Absorption Spectrum of 1,3-bis(3',3'-Trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine in Ethanol

20. Melting point: 119-121 °C. (d)

CONFIDENTIAL

Report No. 737
SPIA/M3

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)

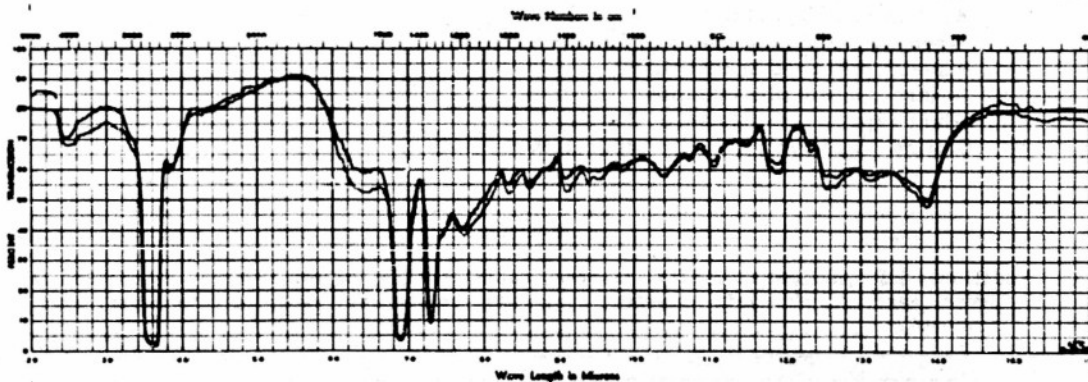


22. Solubility of new compound:

<0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
160 g/100 ml acetone at 25 °C.
(name material used as solvent)
4.3 g/100 ml toluene at 25 °C.
(name material used as solvent)

I.R. SPECTROPHOTOMETER
NACO INSTR.

NO.	DATE		PAGE	
	3-13-53		1	
NAME				
1, 2, 4, 6, 8, 11-Dinitro- propyl 2, 2'-azobis(2- methyl-1-propanol) Resubmitted to other solvent				
FORM				
THERM. CELL				
CIRCUIT				
SOLV.				
VOL. C.C.				
FA. SOLV. C. C.				



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____ : _____

29. Polymerizing properties of the new compound:
 (a) By itself _____
 (b) In mixtures (with additives) _____
 (c) Inhibiting action on polymerization of:
 Thickol _____
 Methacrylate _____
 Other compounds _____

30. Availability
 a. Amount now available? _____ research quantities
 b. When was available material first prepared? _____
 c. Amount prepared at that time? _____
 d. Is large production feasible? _____
 e. Plant capacity in existence, lbs/day? _____
 f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 153 }
 Calculated Ballistic Mortar Value = 157 } Method of Aerojet Report No. 512, p. 8

CONFIDENTIAL

Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound further. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AF/JSU, 8651 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,3,5-tris(3',3',3'-Trinitropropyl)-

Name hexahydro-1,3,5-triazine

Empirical formula C₁₂H₁₂N₆O₁₈

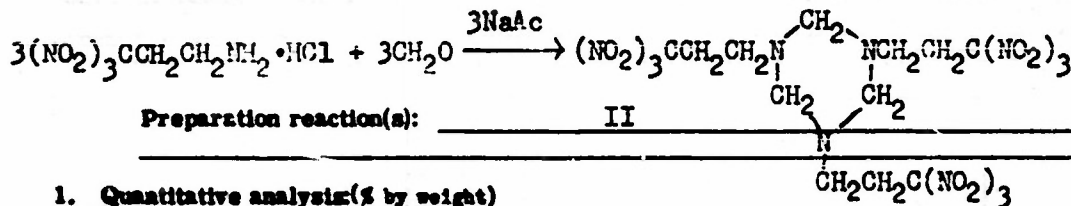
Structure: (configuration) II

Information submitted by:

Activity Aerojet-General Corporation

Person M. B. Frankel and L. T. Carleton

Date 1 August 1953



Preparation reaction(s): II

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen
Calculated from formula	23.31	2.93		27.19
By determination	23.78	2.68		27.21

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

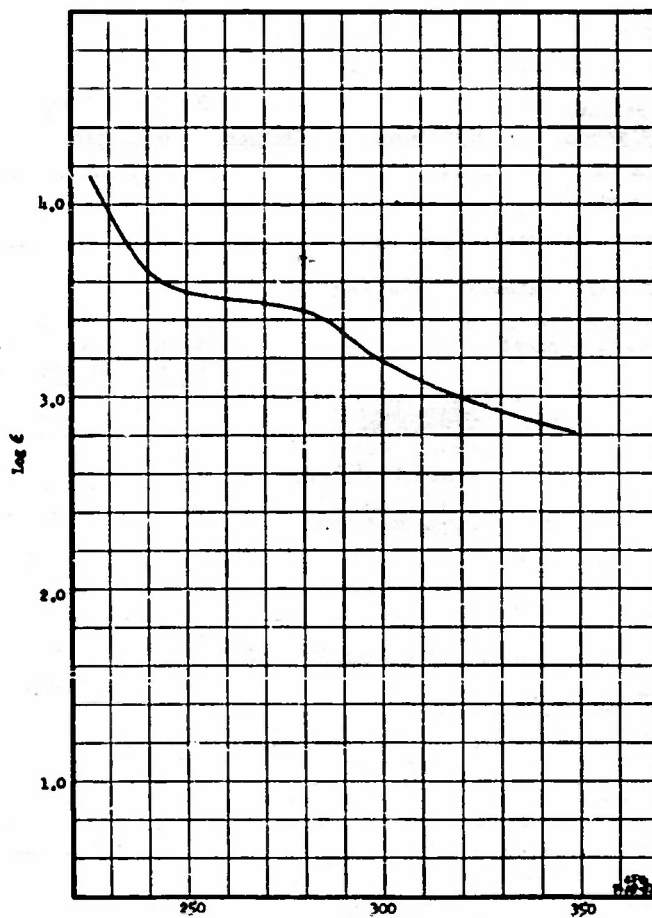
Name of test	Recommended method	
a. Impact Sensitivity	NOL XXXXXXXXXX	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL XXXXXXXXXX	at 80°C
d. Temperature of Explosion	OSRD 3401 p.8	
e. Temperature of Ignition	NOL XXXXXXXXXX	
f. Impact Stability, Bureau of Mines Bull. No. 346, p. 72 (2-kg weight)		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, H.C., etc.)	New Compound test results
a. <u>Tetryl, 32 cm/2.5 kg</u>	<u>20 cm/2.5 kg</u>
b.	
c.	<u>exploded in 1 hr</u>
d.	
e.	<u>740°C</u>
f. <u>RDX, 30 cm/2 kg</u>	<u>15-20 cm/2 kg</u>
g.	
h.	

4. Heat of formation: $(\Delta H) \pm \frac{-141}{(\text{indicate sign})}$ Kg. calories at 25°C., 1 atm. pressure

- | | By Experiment | By Calculation | Method |
|---|--------------------------|---|---|
| 5. Energy of explosion (Q)
(at 25°C. H ₂ O liquid) | _____ | cal/gm _____ | Description or reference. Separate sheet if necessary. |
| 6. Heat of combustion (H _c)
(at 25°C. H ₂ O liquid) | 2748 | cal/gm 2812 | Aerojet Report No. 417A |
| 7. Specific impulse (I _{sp}) calc: | _____ | lb-sec/lb _____ | |
| 8. Physical form of compound (viscous liquid, crystalline type, etc.) | Crystalline Solid | | |
| 9. Simple microscope analysis data: _____
(crystal studies) | | | |
| 10. Density (Macro method) | 1.657 gm/cm ³ | (Micro or other method) _____ gm/cm ³ | (NOL) (Include in separate sheet any unique methods you use.) |
| 11. Index of refraction (n _D ^{25°C.}) | _____ | 12. Color Yellow | 13. Odor None |
| 14. pH at 25°C. | 6.2 | (Method reference OESD 3401 v.4, or OESD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator used or Beckman pH meter.) In Beckman pH meter, 0.060 M in acetone/water (5/1 volume ratio) | |



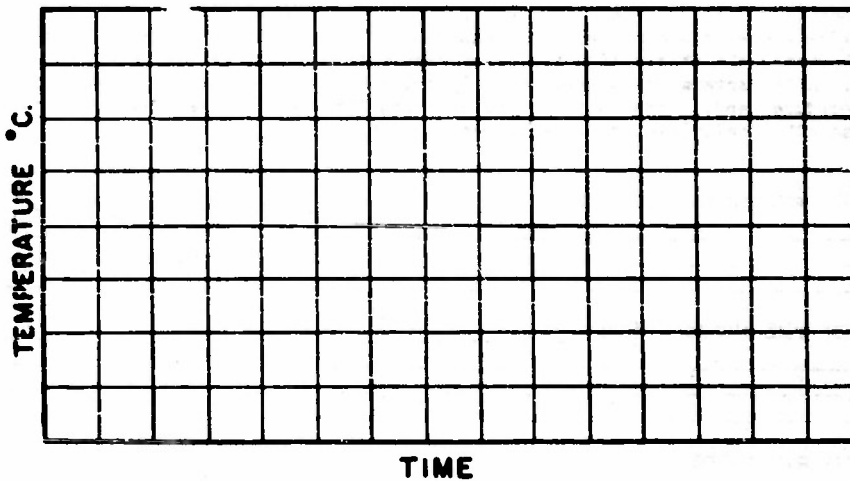
Ultraviolet Absorption Spectrum of 1,3,5-tris(3',3',3'-Trinitropropyl) hexahydro-1,3,5-triazine in Ethyl Ether

20. Melting point: 120-122 °C.(d)

CONFIDENTIAL

Report No. 737
SPLA/M3

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



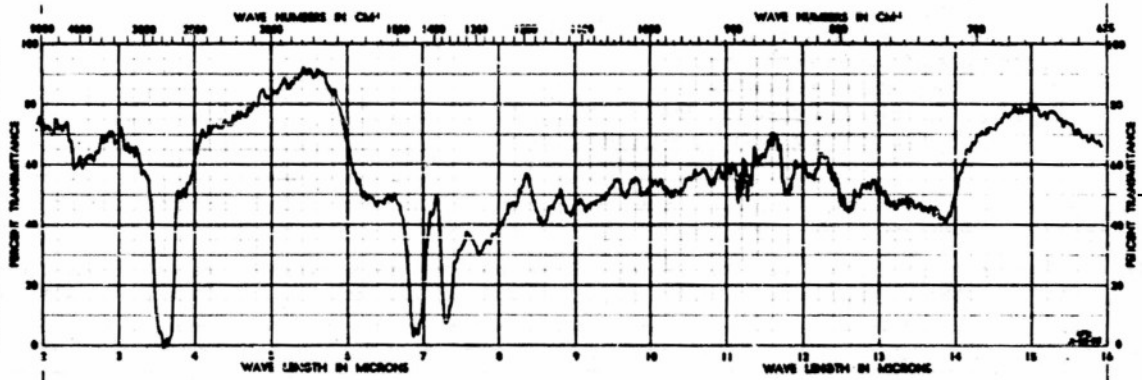
22. Solubility of new compound:

0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
55 g/100 ml acetone at 25 °C.
(name material used as solvent)
0.2 g/100 ml toluene at 25 °C.
(name material used as solvent)

I.R. SPECTROPHOTOMETRIC
NaCl PRISM

NO.	DATE	OPER.
1-2-53		
SAMPLE		
1,3,5-trimethyl-2,4,6-trinitrobenzene		
Prepared in white solvent		
41		
PREP. BY - J.M.		
SAMP. CELL	MM	CHL.
REF. CELL	MM	CHL.
CHRM.	MM	MM
SOLV.		
VOL. CC.	N	CC. N
FA. SOLV.	C	SAE

BARCO ASSOCIATES, INC.
CAMDEN, N.J., U.S.A.



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSGD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simultaneous observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strins of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:

(a) By itself _____

(b) In mixtures (with additives) _____

(c) Inhibiting action on polymerization of:

Thiokol _____

Methacrylate _____

Other compounds _____

30. Availability

a. Amount now available? research quantities

b. When was available material first prepared? _____

c. Amount prepared at that time? _____

d. Is large production feasible? _____

e. Plant capacity in existence, lbs/day? _____

f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 130

Calculated Ballistic-Mortar Value = 140

Method of Aerojet Report No. 512

CONFIDENTIAL

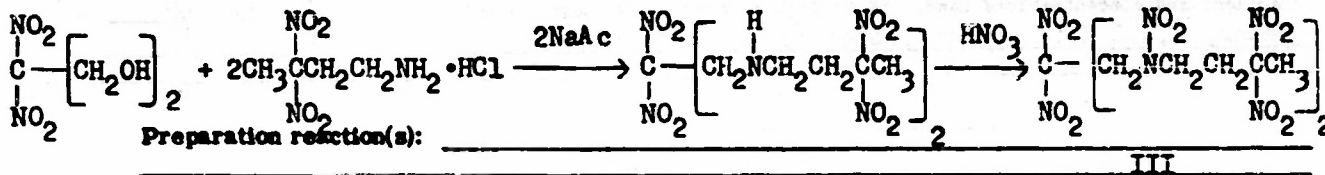
Date Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8631 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in leaflet manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 2,2,5,7,7,9,12,12-Octanitro-

Name 5,9-diaza tridecane
Empirical formula C₁₇H₁₈N₁₀O₁₆
Structure: (configuration) III

Information submitted by:
Activity Aerojet-General Corporation
Person H. E. Frankel and L. T. Carleton
Date 1 August 1953



1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen
Calculated from formula	24.18	3.32		25.64
By determination	24.76	3.11		25.52

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure)

(faster? slower? residue? etc.)

3. Stability and Sensitivity. Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

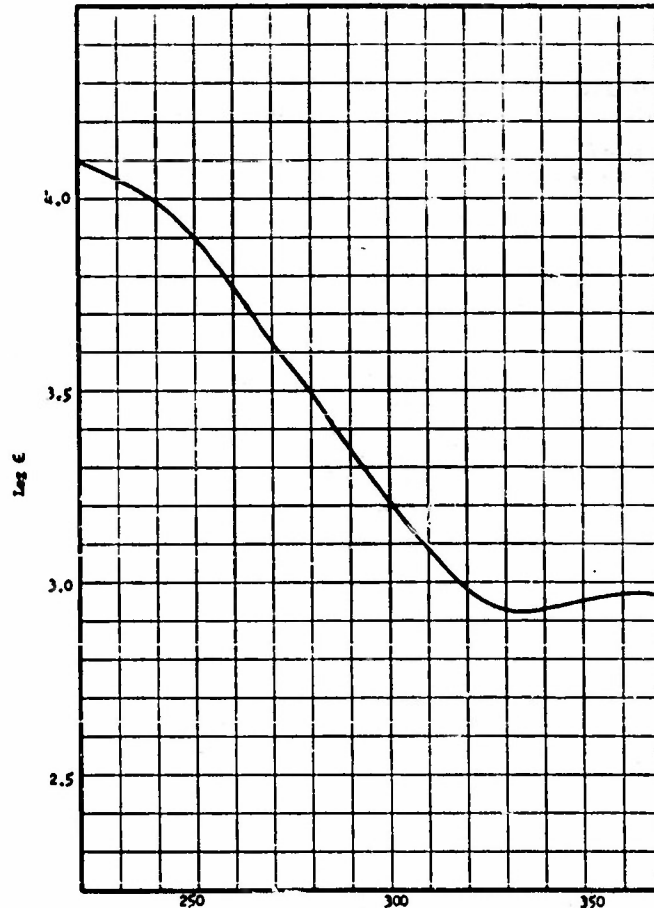
Name of test	Recommended method	
a. Impact Sensitivity	NOL <u>OSRD 3401 p.8</u>	<u>2.5-kg weight</u>
b. Thermal Stability	NOL <u>OSRD 3401 p.8</u>	
c. Vacuum Stability	NOL <u>OSRD 3401 p.8</u>	<u>48 hr at 80°C</u>
d. Temperature of Explosion	NOL <u>OSRD 3401 p.8</u>	
e. Temperature of Ignition	NOL <u>OSRD 3401 p.8</u>	
f. Impact Stability	Bureau of Mines, Bull. No. 346, p. 72 (2-kg weight)	
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound (designation Tetryl, Tetryl, H.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	<u>37 cm/2.5 kg</u>
b.	
c.	<u>2.2 cc/g</u>
d.	
e.	<u>180°C</u>
f. RDX, 30 cm/2 kg	<u>10-15 cm/2 kg</u>
g.	
h.	

4. Heat of formation: $(\Delta H) \pm \frac{-62}{\text{(Indicate sign)}} \text{ Kg. calories at } 25^\circ\text{C., 1 atm. pressure}$

- | | By Experiment | By Calculation | Method |
|---|-----------------------------------|---|--|
| 5. Energy of explosion (G)
(at 25°C. H ₂ O liquid) | _____ | cal/gm _____ | Description or reference. Separate sheet if necessary. |
| 6. Heat of combustion (H _c)
(at 25°C. H ₂ O liquid) | 2897, 2913 | cal/gm 2879 | Aercjet Report No. 417A |
| 7. Specific impulse (I _{sp}) calc: | _____ | lb-sec/lb _____ | |
| 8. Physical form of compound (viscous liquid, crystalline type, etc.) | Fine crystals | | |
| 9. Simple microscope analysis data: _____
(crystal studies) | | | |
| 10. Density (Macro method) | 1.676 gm/cm ³
(NOL) | (Micro or other method) _____ gm/cm ³
(Indicate on separate sheet any unique methods you use.) | |
| 11. Index of refraction (n _D ^{25°C.}) | _____ | 12. Color White | 13. Odor None |
| 14. pH at 25°C. | 5.0 | (Method reference OSRD 3401 v.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator used or Beckman pH meter.) With Beckman pH meter, 0.030 M in acetone/water (5/1 volume ratio) | |

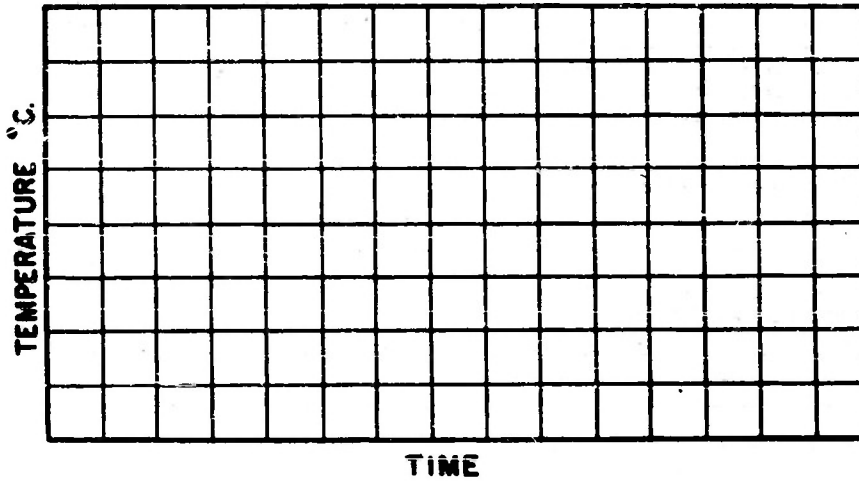


Ultraviolet Absorption Spectrum of 2,2,5,7,7,9,12,12-Octanitro 5,9-Di-aza tridecane in Acetonitrile

20. Melting point: 137-143 °C. (d)

CONFIDENTIAL

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)

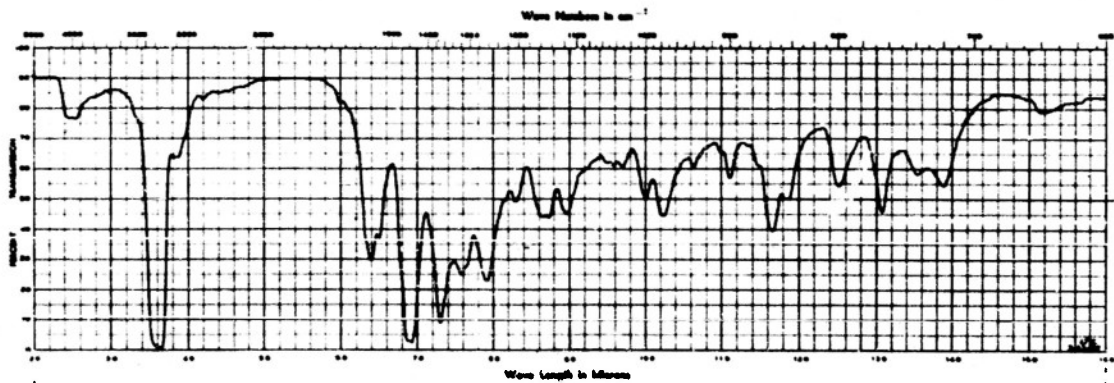


22. Solubility of new compound

0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
31 g/100 ml acetone at 25 °C.
(same material used as solvent)
0.1 g/100 ml toluene at 25 °C.
(same material used as solvent)

I.R. SPECTROPHOTOMETER
NACI PRISM

NO.	DATE	INDEX
1-21-51	10/21/51	
3,7,5,7,9,11,13-hydroxystro- 5,7-dione lactone		
Sampled in 10% solvent 0.1		
WGM	File # 12	cm
SLAMP CELL	mm	CAL
REF. CELL	mm	CAL
CHINA	mm	mm
SOLV		
VOL. C.C.	ml	C.C.
PA. SOLV	C	mm
BARR ASSOCIATES, INC. CAMBRIDGE, MASS., U.S.A.		



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thickol _____
 Methacrylate _____
 Other compounds _____

30. Availability
a. Amount now available? Research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 128 }
Calculated Ballistic-Mortar value = 140 } Method of Aerojet Report No. 512, p. 8

CONFIDENTIAL

Tests Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFJ/JHU, 8631 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 2,2,4,7,7,10,12,12-Octanitro-

Name 4,10-diaza tridecane

Empirical formula C₁₁H₁₈N₂O₁₆

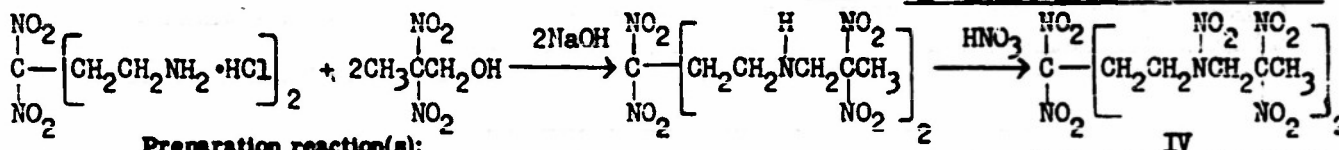
Structure: (configuration) IV

Information submitted by:

Activity Aerojet-General Corporation

Person M.B. Frankel and L.T. Carleton

Date 1 August 1953



1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	<u>24.78</u>	<u>3.32</u>		<u>25.64</u>		
By determination	<u>24.76</u>	<u>3.35</u>		<u>24.98</u>		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL <u>OSRD 3401 p.8</u>	<u>2.5-kg weight</u>
b. Thermal Stability	<u>OSRD 3401 p.8</u>	
c. Vacuum Stability	NOL <u>OSRD 3401 p.8</u>	<u>48 hr at 100°C</u>
d. Temperature of Explosion	<u>OSRD 3401 p.8</u>	
e. Temperature of Ignition	NOL <u>OSRD 3401 p.8</u>	
f. Impact Stability	Bureau of Mines Bull. No. <u>346</u> , p. <u>72</u> (2-kg weight)	
g.		
h.		

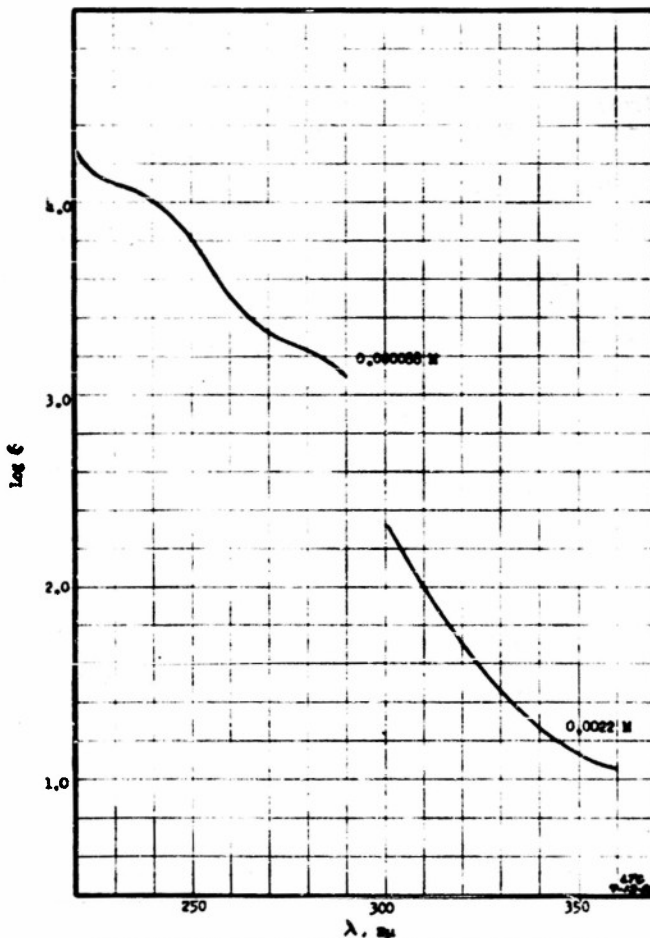
RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, H.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	<u>44 cm/2.5 kg</u>
b.	
c.	<u>0.7 cc/g</u>
d.	
e.	<u>244°C</u>
f. RDX, 30 cm/2 kg	<u>40-45 cm/2 kg</u>
g.	
h.	

4. Heat of formation: (ΔH_f°) -104 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

CONFIDENTIAL

- | | By Experiment | By Calculation | Method |
|---|--------------------------|--|--|
| 5. Energy of explosion (Q)
(at 2500. H ₂ O liquid) | _____ cal/gm | _____ cal/gm | Description or reference. Separate sheet if necessary. |
| 6. Heat of combustion (H _c)
(at 2500. H ₂ O liquid) | 2830, 2825 | 2878 | Aerojet Report No. 417A |
| 7. Specific impulse (I _{sp}) calc: | _____ lb-sec/lb | _____ lb-sec/lb | |
| 8. Physical form of compound (viscous liquid, crystalline type, etc.) | Fine crystals | | |
| 9. Simple microscope analysis data:
(crystal studies) | | | |
| 10. Density (Micro method) | 1.708 gm/cm ³ | (Micro or other method) _____ gm/cm ³ | (Retain on separate sheet any unique methods you use.) |
| 11. Index of refraction (n _D ^{25°C.}) | (NOL) | 12. Color | White |
| | | 13. Odor | None |
| 14. pH at 25°C. | 6.8 | (Method reference OGD 3401 v.4, or OGD 5966. Indicate method used, i.e. solvent and concentrations used. pH indicator used or Beckman pH meter.) | |
| | | With Beckman pH meter, saturated solution in acetone/water (5/1 volume ratio) | |

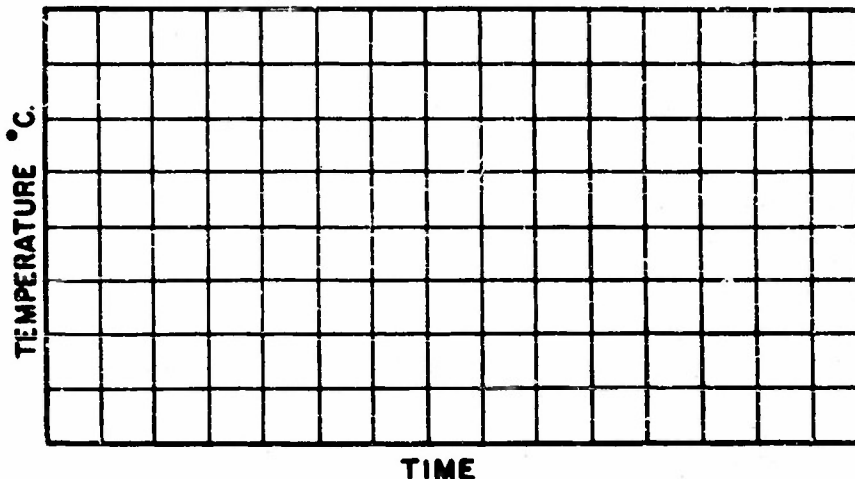


Ultraviolet Absorption Spectra of 2,2,4,7,7,10,12,12-Octanitro-4,10-diaza tridecane in Acetonitrile

20. Melting point: 206-208 °C.(d)

CONFIDENTIAL

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)

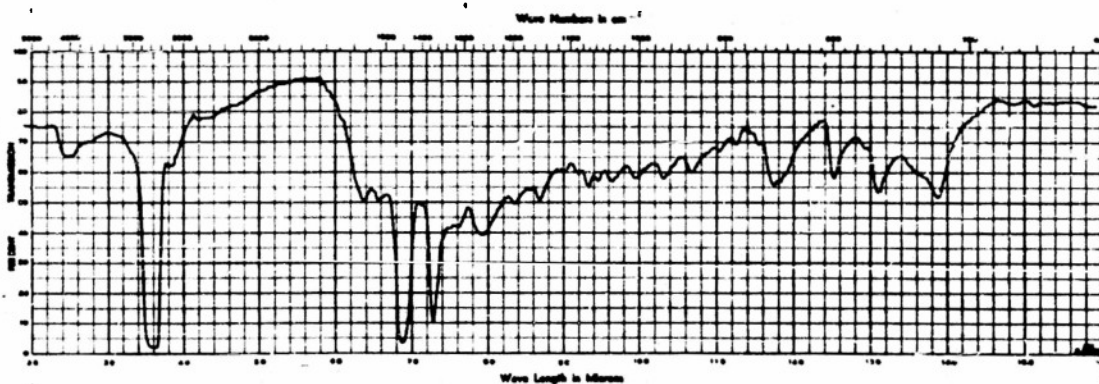


22. Solubility of new compound:

<0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
1.4 g/100 ml acetone at 25 °C.
(name material used as solvent)
<0.1 g/100 ml toluene at 25 °C.
(name material used as solvent)

I.R. SPECTROMETER
MFC Model

NO.	DATE	INDEX
	3-11-62	
SAMPLE		
2,2,4,7,7,10,10,12-octachloro-1,10-dioxane BT 2000		
Prepared in white mineral oil		
REF: Time = 32 min		
SAMP CELL	NO.	CHK.
REF. CELL	NO.	CHK.
CHGR	NO.	NO.
SOLV.		
VOL. U.C.	%	C.C. %
P.A. SOLID	C.	GAS
KAPPA ASSOCIATES, INC. CAMBRIDGE, MASS., U.S.A.		



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the followings: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a C clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____ : _____

29. Polymerizing properties of the new compound:
 (a) By itself _____
 (b) In mixtures (with additives) _____
 (c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____

30. Availability
 a. Amount now available? Research quantities
 b. When was available material first prepared? _____
 c. Amount prepared at that time? _____
 d. Is large production feasible? _____
 e. Plant capacity in existence, lbs/day? _____
 f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 128

Calculated Ballistic Mortar Value = 149 } Method of Aerojet Report No. 512, p. 6

CONFIDENTIAL

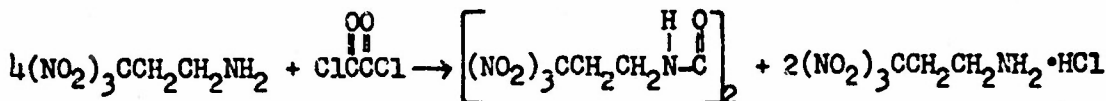
Tests Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFL/JBU, 8651 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: N,N'-bis(3,3,3-Trinitropropyl)-

Name oxamide
Empirical formula C₈H₁₀N₈O₇
Structure: (configuration) V

Information submitted by:
Activity Aerojet-General Corporation
Person M.B. Frankel and L.T. Carleton
Date 1 August 1953



Preparation reaction(s): V

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	21.73	2.28		25.34		
By determination	22.06	2.32		25.31		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure)

faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

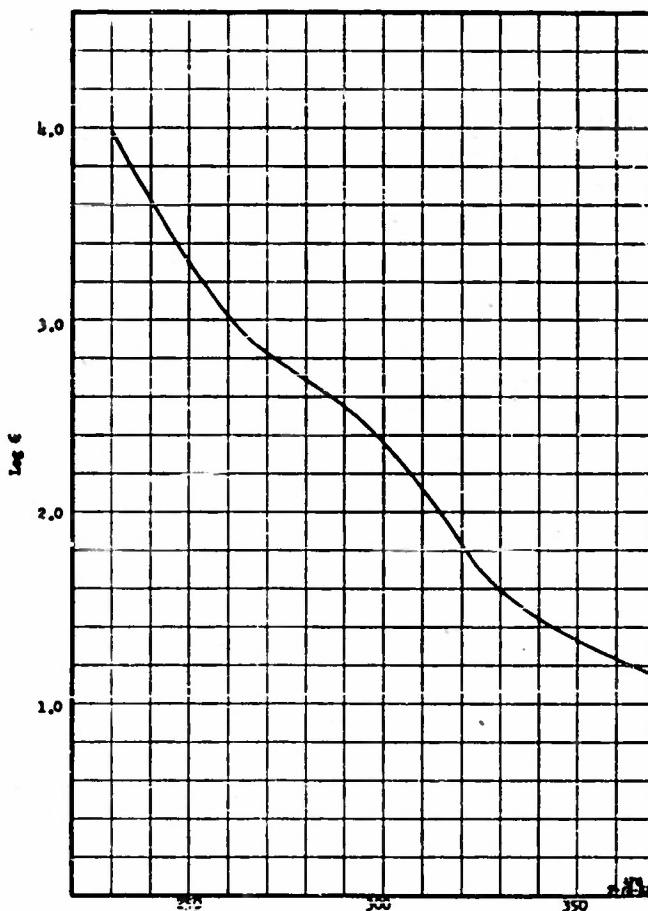
Name of test	Recommended method	
a. Impact Sensitivity	NOL OSRD 3401 p.8	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL OSRD 3401 p.8	48 hr at 100°C
d. Temperature of Explosion	OSRD 3401 p.8	
e. Temperature of Ignition	NOL OSRD 3401 p.8	
f. Impact Stability	Bureau of Mines, Bull. No. 346, p. 72 (2-kg weight)	
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	45 cm/2.5 kg
b.	
c.	0.31 cc/g
d.	
e.	215°C
f. RDX, 30 cm/2 kg	15-20 cm/2 kg
g.	
h.	

4. Heat of formation: $(\Delta H) + \frac{-145}{(\text{indicate sign})}$ Kg. calories at 25°C., 1 atm. pressure

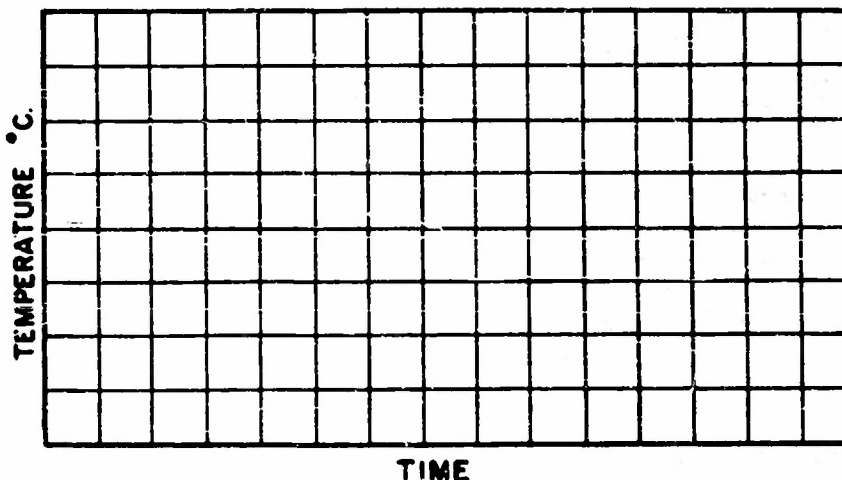
- | | By Experiment | By Calculation | Method |
|---|-----------------------------------|--|--|
| 5. Energy of explosion (Q)
(at 25°C. H ₂ O liquid) | _____ | cal/gm _____ | Description or reference. Separate sheet if necessary. |
| 6. Heat of combustion (H _c)
(at 25°C. H ₂ O liquid) | 2146 | cal/gm 2175 | Aerojet Report No. 417A |
| 7. Specific impulse (I _{sp}) calc: | _____ | lb-sec/lb _____ | |
| 8. Physical form of compound (viscous liquid, crystalline type, etc.) | Fine, needle-shaped crystals | | |
| 9. Simple microscope analysis data:
(crystal studies) | _____ | | |
| 10. Density (Macro method) | 1.692 gm/cm ³
(NOL) | (Micro or other method) _____ gm/cm ³
(Include on separate sheet any unique methods you use.) | |
| 11. Index of refraction (n _D ^{25°C.}) | _____ | 12. Color White | 13. Odor None |
| 14. pH at 25°C. | 5.6 | (Method reference OGD 1401 v.4, or OGD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator water or Beckman pH meter.)
pH meter, 0.087 M in acetone/water (5/1 volume ratio) | |



Ultraviolet Absorption Spectrum of N,N'-bis(3,3-Trinitropropyl) Oxamide in Ethanol

20. Melting point 195-197 °C. (d)

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



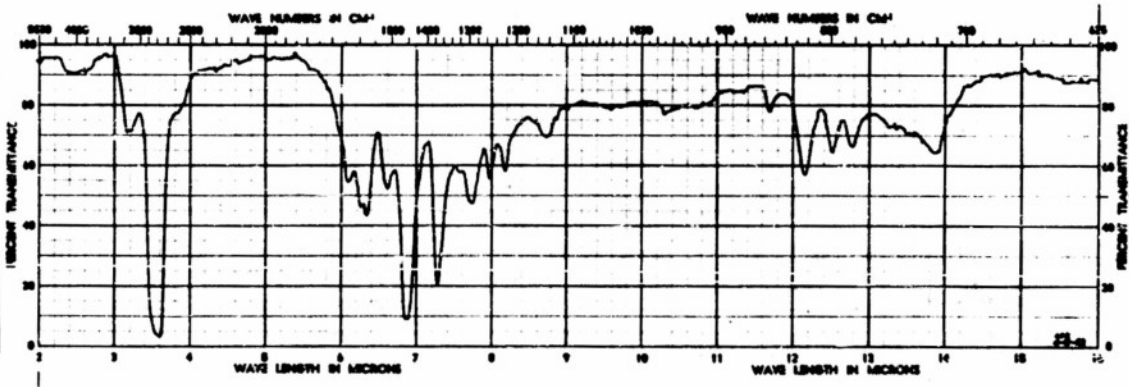
22. Solubility of new compound:

0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
17 g/100 ml acetone at 25 °C.
 (name material used as solvent)
0.3 g/100 ml toluene at 25 °C.
 (name material used as solvent)

L.S. SPECTROMETER
DUO PRISM

NO.	DATE	INDEX
SASD		
SAMPLE		
N, N'-di(1,1,3-trichloropropyl) malonamide		
Dissolved in water at 25°C		
PREP. Date = 12-14-54		
SAMP. CELL	MM	CM.
REF. CELL	MM	CM.
CHEM.	MS.	MS.
SOLV.		
VOL. C.C.	% C.C.	%
VS. SOLV.	C	SAS

PERKIN-ELMER INSTRUMENT CO.
CAMBRIDGE, MASS., U.S.A.



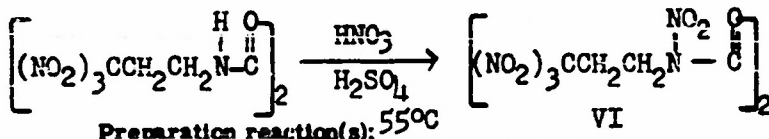
CONFIDENTIAL

Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: N,N'-Dinitro-N,N'-bis(3,3,3-trinitro-
Name propyl) oxamide
Empirical formula C₈H₈N₇O₁₈
Structure: (configuration) VI

Information submitted by:
Activity Aerojet-General Corporation
Person M.B. Frankel and L.T. Carleton
Date 7 August 1953



1. Quantitative analysis (% by weight)	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	18.05	1.52		26.32		
By determination	18.42	1.63		26.49		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot dry graphs on separate sheet Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

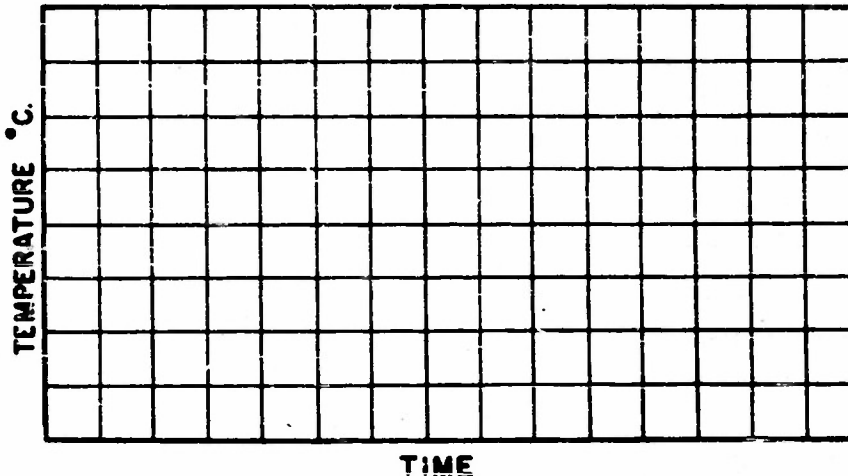
Name of test	Recommended method	
a. Impact Sensitivity	NOL	<u>2.5-kg weight</u>
b. Thermal Stability		<u>OSRD 3401 p.8</u>
c. Vacuum Stability	NOL	<u>48 hr at 100°C</u>
d. Temperature of Explosion		<u>OSRD 3401 p.6</u>
e. Temperature of Ignition	NOL	<u>OSRD 3401 p.6</u>
f. Impact Stability	Bureau of Mines, Bull. No. 346, p. 72 (2-kg weight)	
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a. <u>Tetryl, 32 cm/2.5 kg</u>	<u>9 cm/2.5 kg</u>
b.	
c.	<u>0.40 cc/g</u>
d.	
e.	<u>207</u>
f. <u>RDX, 30 cm/2 kg</u>	<u>10 cm/2 kg</u>
g.	
h.	

4. Heat of formation: $(\Delta H) + \frac{-90}{\text{(Indicate sign)}}$ Kg. calories at 25°C., 1 atm. pressure

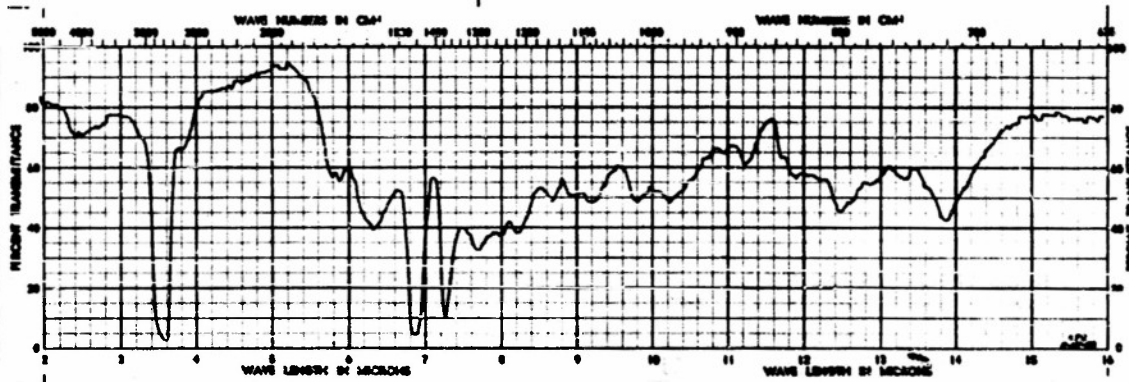
21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

< 0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
89 g/100 ml acetone at 25 °C.
(name material used as solvent)
7.0 g/100 ml toluene at 25 °C.
(name material used as solvent)

NO.		DATE		FORM	
SOLVENT		SOLVENT		SOLVENT	
Prepared in this solvent at _____ °C.					
PREP. Size - 32 mm					
SAMP. CELL	MM	CHL			
REF. CELL	MM	CHL			
CHL	MM	MM			
SOLV.					
VOL. CC.	CC.	CC.	CC.	CC.	CC.
FL. SOLV.	°C.	°C.	°C.	°C.	°C.
EASTMAN KODAK CO. CAMBRIDGE, MASS., U.S.A.					



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a C-clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____ : _____

29. Polymerizing properties of the new compound:
 (a) By itself _____
 (b) In mixtures (with additives) _____
 (c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____

30. Availability
 a. Amount now available? _____ Research quantities
 b. When was available material first prepared? _____
 c. Amount prepared at that time? _____
 d. Is large production feasible? _____
 e. Plant capacity in existence, lbs/day? _____
 f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 176
 Method of Aerojet Report No. 512, p. 8
 Calculated Ballistic-Mortar Value = 177

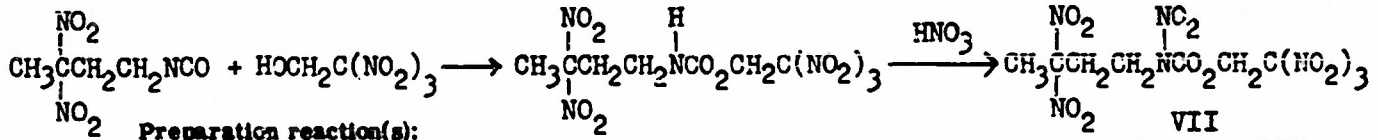
CONFIDENTIAL

Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8631 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-
Name 5-aza-nonane
Empirical formula C₇H₅N₆O₁₁
Structure (configuration) VII

Information submitted by:
Activity Aerojet-General Corporation
Person M. B. Frankel and L. T. Carleton
Date 1 August 1953



1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	20.25	2.19		23.62		
By determination	20.25	2.21		23.67		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure):

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

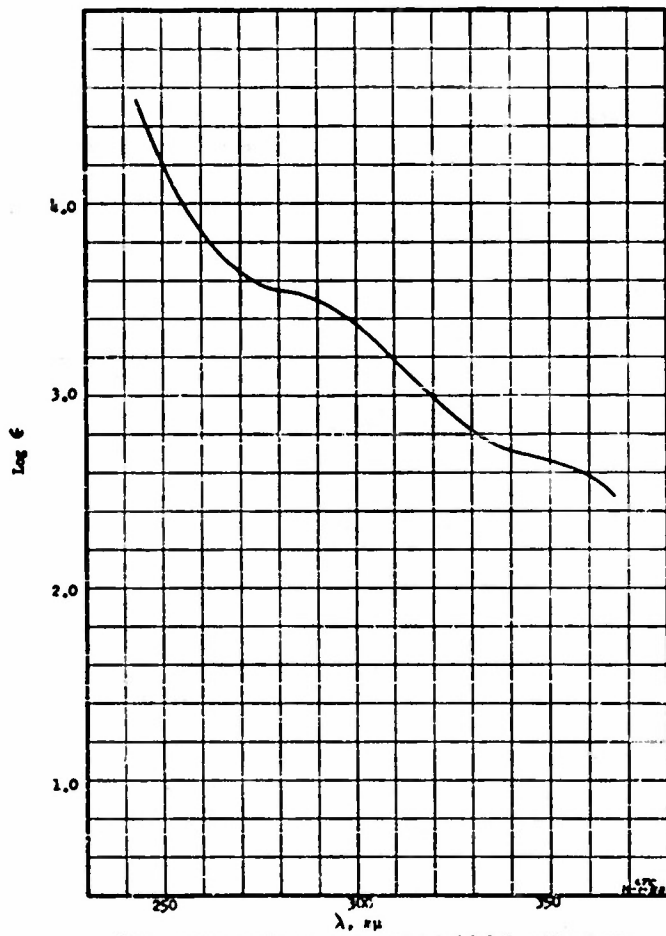
Name of test	Recommended method	
a. Impact Sensitivity	NOL	OSRD 3401 p.8
b. Thermal Stability	NOL	OSRD 3401 p.8
c. Vacuum Stability	NOL	OSRD 3401 p.8
d. Temperature of Explosion	NOL	OSRD 3401 p.8
e. Temperature of Ignition	NOL	OSRD 3401 p.8
f. _____		
g. _____		
h. _____		

RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, H.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	15 cm/2.5 kg
b. _____	
c. _____	0.50 cc/g
d. _____	
e. _____	223°C
f. _____	
g. _____	
h. _____	

4. Heat of formation: $(\Delta H) + \frac{-132}{(\text{Indicate sign})}$ Kcal. calories at 25°C., 1 atm. pressure

- | | By Experiment | By Calculation | Method |
|---|--------------------------|---|--|
| 5. Energy of explosion (Q)
(at 25°C. H ₂ O liquid) | _____ | cal/gm _____ | Description or reference. Separate sheet if necessary. |
| 6. Heat of combustion (H _c)
(at 25°C. H ₂ O liquid) | 2007 | cal/gm 2032 | Aerojet Report No. 417A |
| 7. Specific impulse (I _{sp}) calc: | _____ | lb-sec/lb _____ | |
| 8. Physical form of compound (viscous liquid, crystalline type, etc.) | Crystalline solid | | |
| 9. Simple microscope analysis data: _____
(crystal studies) | | | |
| 10. Density (micro method) | 1.733 gm/cm ³ | (Micro or other method) _____ gm/cm ³ | (NOL) (Krusin on separate sheet any unique methods you use.) |
| 11. Index of refraction (n _D ²⁰) | _____ | 12. Color White | 13. Odor None |
| 14. pH at 25°C. | 4.5 | (Method reference OHRD 3401 v.4, or OHRD 5966. Indicate method used, i.e. solvent and concentrations used. pH indicator used or Beckman pH meter.) (decreasing rapidly) | |
| With Beckman pH meter. 0.0091 M in acetone/water (5/1 volume ratio) | | | |

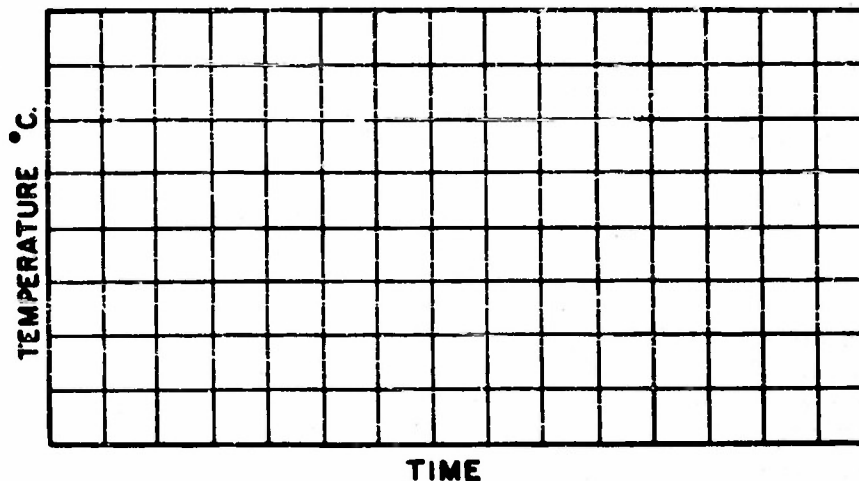


Ultraviolet Absorption Spectrum of 1,1,1,5,8,8-Hexanitro-3-oxa-4-azabicyclo-octane in Ethanol

20. Melting point: 107-108 °C.

CONFIDENTIAL

31. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



32. Solubility of new compound:

< 0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
1.0 g/100 ml acetone at 25 °C.
(same material used as solvent)
1.4 g/100 ml toluene at 25 °C.
(same material used as solvent)

I.R. SPECTROSCOPY

1000-1000

NO. _____

DATE _____

TIME _____

SAMPLE _____

1,1,1,5,5,5-hexafluoro-2,2,4,4-tetrahydro-3,4-dioxane

Prepared in white mineral oil

NO. _____

TIME - 17 min

SAMP. CELL: 1000 _____

NO. _____

CHGR: Ball, 1000 _____

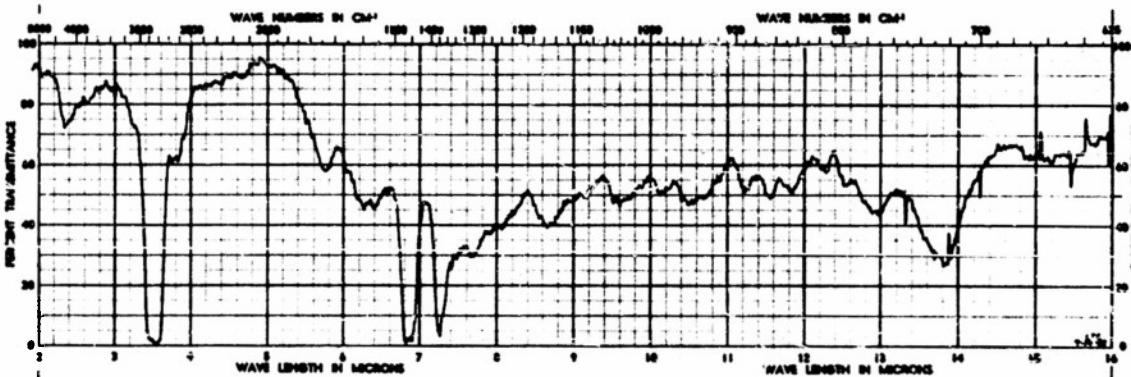
SOLV. _____

VOL. C.C. _____

PREPARED BY: _____

LABORATORY: _____

CHICAGO, ILL., U.S.A.



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OQRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of oxidation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerising properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____

30. Availability
a. Amount now available? Research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 153
Calculated Ballistic-Mortar Value = 140
Method of Aerojet Report No. 512, p. 8

CONFIDENTIAL

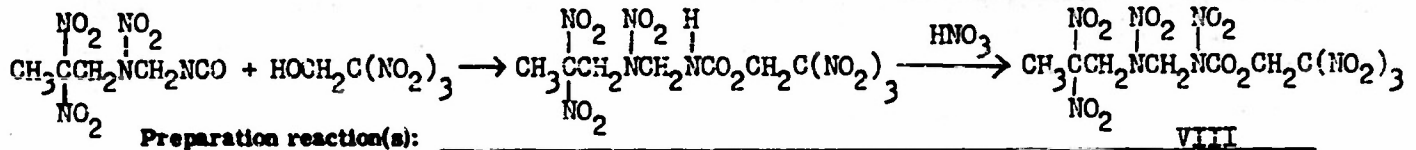
Tests Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFPL/JHU, 8631 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-

Name 5,7-diaza-decane
Empirical formula C₇H₅N₇O₇
Structure: (configuration) VIII

Information submitted by:
Activity Aerojet-General Corporation
Person M.R. Frankel and L.T. Carleton
Date 1 August 1953



Preparation reaction(s):

1. Quantitative analysis: (5 by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen
Calculated from formula	17.69	1.91		26.53
By determination	18.08	1.95		26.58

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL	OSRD 3401 p.8 2.5-kg weight
b. Thermal Stability	NOL	OSRD 3401 p.8
c. Vacuum Stability	NOL	OSRD 3401 p.8 18 hr at 100°C
d. Temperature of Explosion	NOL	OSRD 3401 p.8
e. Temperature of Ignition	NOL	OSRD 3401 p.8
f.		
g.		
h.		

RESULTS OF ABOVE TESTS

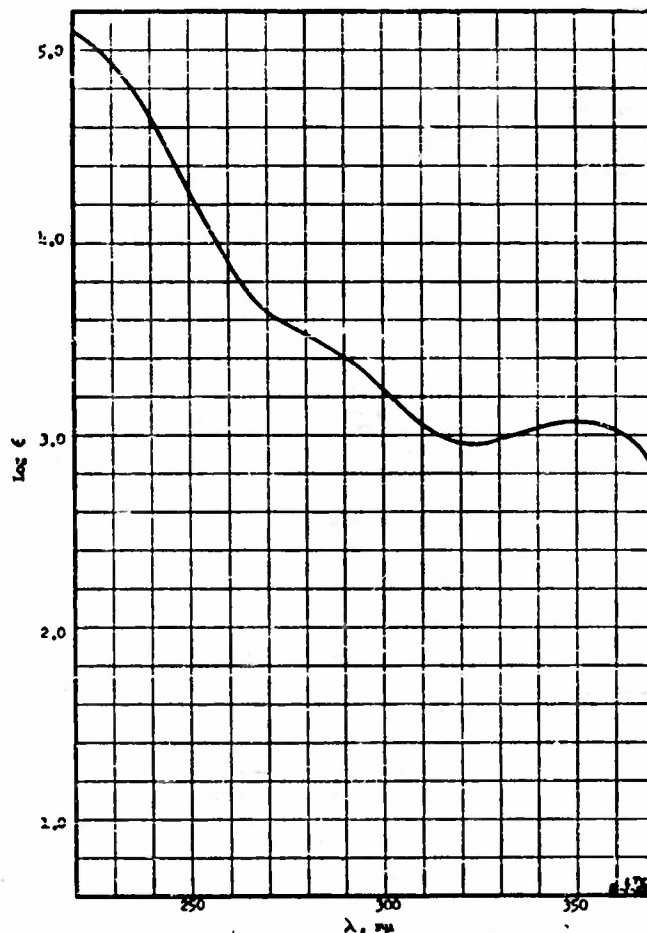
Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	18 cm/2.5 kg
b.	
c.	1.01 cc/g
d.	
e.	219°C
f.	
g.	
h.	

4. Heat of formation: $(\Delta H) + \frac{-118}{(\text{indicate sign})}$ Kg. calories at 25°C., 1 atm. pressure

CONFIDENTIAL

Report No. 737
SPIA/MS

- | | By Experiment | By Calculation | Method
<small>Description or reference. Separate sheet if necessary.</small> |
|--|---|------------------------|---|
| 5. Energy of explosion (Q)
<small>(at 2500, H₂O liquid)</small> | _____ | cal/gm _____ | _____ |
| 6. Heat of combustion (H _c)
<small>(at 2500, H₂O liquid)</small> | 1785 | cal/gm 1798 | Aerojet Report No. 417A |
| 7. Specific impulse (I _{sp}) calc: | _____ | lb-sec/lb _____ | _____ |
| 8. Physical form of compound (viscous liquid, crystalline type, etc.) | Flaky crystals | | |
| 9. Simple microscope analysis data:
<small>(crystal studies)</small> | _____ | | |
| 10. Density (Macro method) <u>1.790</u> gm/cm ³ .
<small>(Micro or other method) _____ gm/cm³.
<small>(Rule in on separate sheet any unique methods you use.)</small></small> | | | |
| 11. Index of refraction (n _D ²⁰) <u>(NOL)</u> | _____ | 12. Color <u>White</u> | 13. Odor <u>None</u> |
| 14. pH at 25°C. <u>3.5</u>
<small>(Method reference OIRD 3401 v.4, or OIRD 5966. Indicate method used, i.e. solvent and concentrations used. pH indicator used or Beckman pH meter.)</small> | <u>(decreasing rapidly)</u>
<u>With Beckman pH meter, 0.0091 M in acetone/water (5/1 volume ratio)</u> | | |

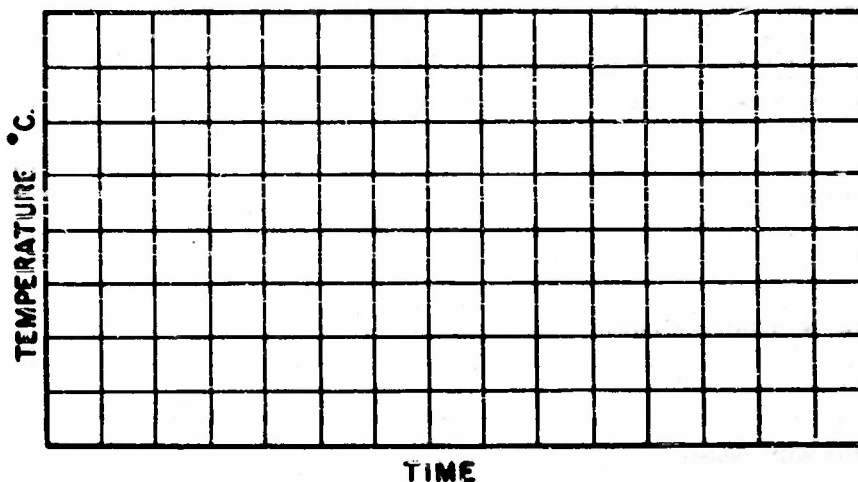


Ultraviolet Absorption Spectrum of 1,1,1,5,7,9-Heptanitro-
-buto-5,7-diene-decane in Methylene

20. Melting point: 140-141 °C.

CONFIDENTIAL

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



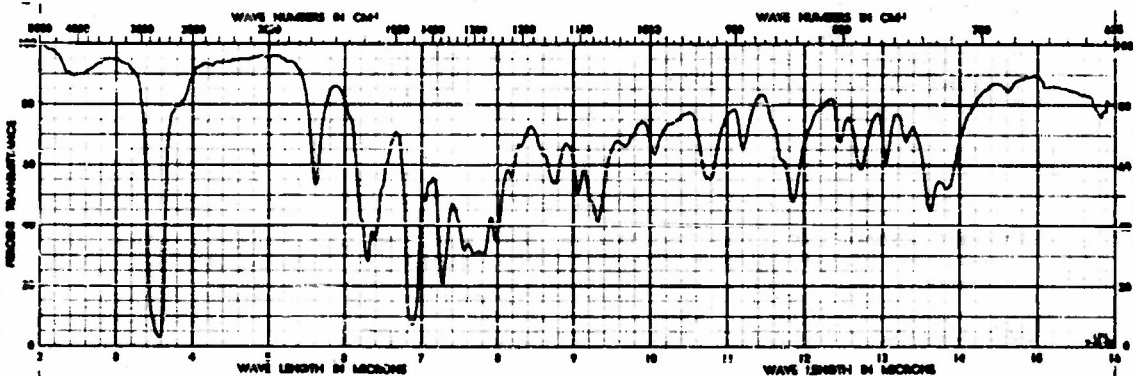
22. Solubility of new compound:

< 0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
76 g/100 ml acetone at 25 °C.
(name material used as solvent)
0.1 g/100 ml toluene at 25 °C.
(name material used as solvent)

I.R. SPECTROPHOTOMETER
MAG 7500

NO.	DATE	INDEX
J. M. L. [unclear]		
[unclear] [unclear] [unclear]		
[unclear] [unclear] [unclear]		
[unclear] [unclear] [unclear]		
PREP.	[unclear] [unclear]	
REP. CELL	NO.	CON.
OTHER	ML	ML
SOLV.	[unclear] [unclear]	
VOL. C.C.	% C.C.	%
FS. SOLV.	°C	ML

LABORATORY, INC.
CAMBRIDGE, MASS., U.S.A.



CONFIDENTIAL

Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____ : _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____

30. Availability
a. Amount now available? Research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 167 }
Calculated Ballistic-Mortar Value = 114 } Method of Aerojet Report No. 512, p. 8

CONFIDENTIAL

Report No. 737

N7onn-462, Task Order I, NOaz 53-618-c

DISTRIBUTION LIST

<u>ADDRESSEE</u>	<u>NO. OF COPIES</u>
Commanding General Aberdeen Proving Ground Maryland Attn: Ballistic Research Laboratories ORDBG-BLI	2
Department of the Army Office, Chief of Ordnance Washington 25, D. C. Attn: ORDTU	2
Department of the Army Office, Chief of Ordnance Washington 25, D. C. Attn: ORDTX-AR	2
Officer in Charge Office, Ordnance Research 2127 Myrtle Drive Duke University Durham, North Carolina	3
Commanding Officer Picatinny Arsenal Dover, New Jersey Attn: Library	2
Commanding Officer Redstone Arsenal Huntsville, Alabama Attn: ORC Technical Library	2
Department of the Air Force Hq. USAF, DCS/D Washington 25, D. C. Attn: AFDRD-AR	1
Department of the Air Force Hq. USAF, DCS/D Washington 25, D. C. Attn: AFDRD-AC-3 Col. Paul F. Nay	1

Page 1a

CONFIDENTIAL

CONFIDENTIAL

Report No. 737

<u>ADDRESSEE</u>	<u>NO. OF COPIES</u>
Commanding General Wright-Patterson Air Force Base Dayton, Ohio Attn: WCEGH-2	1
Commanding General Wright-Patterson Air Force Base Dayton, Ohio Attn: WCNEH-3	1
Allegany Ballistics Laboratory P. O. Box 210 Cumberland, Maryland Attn: Dr. L. G. Bomer	2
Armour Research Foundation of Illinois Institute of Technology Technology Center Chicago 16, Illinois Attn: Dr. L. V. Griffis	1
Atlantic Research Corporation 812 North Fairfax Street Alexandria, Virginia Attn: Dr. A. C. Scurlock	1
U. S. Bureau of Mines 4800 Forbes Street Pittsburgh 13, Pa. Attn: Dr. Bernard Lewis	1
Reaction Motors, Inc. Rockaway, New Jersey Attn: P. F. Wintermitz	1
Experiment, Incorporated P. O. Box 1-T Richmond 2, Virginia Attn: J. W. Mullen II	1
Dr. A. M. Ball Hercules Experiment Station Wilmington, Delaware	1
Jet Propulsion Laboratory 4800 Oak Grove Drive Pasadena 3, California Attn: Dr. Louis G. Dunn	2

CONFIDENTIAL

Report No. 737

ADDRESSEE

NO. OF COPIES

B. F. Goodrich Company
Research Center
Brecksville, Ohio
Attn: Vice President/Research

1

Arthur D. Little, Inc.
30 Memorial Drive
Cambridge 42, Mass.
Attn: Dr. C. S. Keevil

1

Arthur D. Little, Inc.
30 Memorial Drive
Cambridge 42, Mass.
Attn: Dr. W. C. Lothrop

1

Shell Development Company
Emeryville, California
Attn: F. G. Bollo

1

Thiokol Corporation
Elkton Laboratories
Elkton, Maryland
Attn: D. W. Kershner

1

University of Minnesota
Oak Street Laboratories
2013 University Avenue
Minneapolis, Minnesota
Attn: Prof. B. L. Crawford, Jr.

1

National Fireworks Ordnance Corp.
West Hanover, Massachusetts
Attn: Mr. S. J. Porter

1

Dr. Harold Shechter
Department of Chemistry
Ohio State University
Columbus, Ohio

1

Ohio State University
Research Foundation
Columbus 10, Ohio
Attn: Prof. M. L. Wolfson

1

Phillips Petroleum Company
Bartlesville, Oklahoma
Attn: Mr. J. P. Alden

1

Page 3a

CONFIDENTIAL

CONFIDENTIAL

Report No. 737

<u>ADDRESSEE</u>	<u>NO. OF COPIES</u>
Project Squid Princeton University Princeton, New Jersey Attn: Librarian	1
Purdue University Dept. of Chemistry Lafayette, Indiana Attn: Dr. Henry Feuer	3
Rohm and Haas Company 5000 Richmond Street Philadelphia 37, Pa. Attn: Dr. J. F. Kincaid	1
Rohm and Haas Company Redstone Arsenal Research Div. Huntsville, Alabama Attn: Dr. Clayton Huggett	2
Solid Propellant Information Agency Applied Physics Laboratory The Johns Hopkins University Silver Spring, Maryland Attn: Mr. Ross H. Petty	6
Standard Oil Company Research Department P. O. Box 431 Whiting, Indiana Attn: Dr. W. H. Bahlke	1
Office of the Chief of Ordnance Department of the Army Washington 25, D. C. Attn: CRDTA Explosives and Demolition Section	1
University of Louisville Department of Chemistry Louisville 3, Kentucky Attn: Dr. R. H. Wiley	1
U. S. Rubber Company General Laboratories Market and South Streets Passaic, New Jersey Attn: Dr. P. O. Tawney	2

Page 4a

CONFIDENTIAL

CONFIDENTIAL

Report No. 737

ADDRESSEE

NO. OF COPIES

Western Cartridge Company East Alton, Illinois Attn: Mr. R. L. Womer	1
British Joint Services Mission Technical Services 1800 K Street, N. W. Washington, D. C. Attn: Dr. L. Phillips	4
Department of the Navy Bureau of Aeronautics Washington 25, D. C. Attn: SI-5	3
Department of the Navy Bureau of Ordnance Washington 25, D. C. Attn: Ad3, Technical Library	1
Department of the Navy Bureau of Ordnance Washington 25, D. C. Attn: Section Re2d	1
Commander U. S. Naval Air Missile Test Center Point Mugu, California Attn: Technical Library	2
Commanding Officer U. S. Naval Air Rocket Test Station Lake Denmark Dover, New Jersey Attn: Technical Library	1
Commanding Officer U. S. Naval Powder Factory Indian Head, Maryland Attn: Research and Development Dept.	2
Commander U. S. Naval Proving Ground Dahlgren, Virginia Attn: M. I. Division	1

CONFIDENTIAL

Report No. 737

<u>ADDRESSEE</u>	<u>NO. OF COPIES</u>
Commandier U. S. Naval Ordnance Laboratory White Oak Silver Spring, Maryland Attn: Library	2
Commandier U. S. Naval Ordnance Test Station Inyokern, China Lake, California Attn: Technical Library Branch	3
Director Naval Research Laboratory Washington 20, D. C. Attn: Chemistry Division, Code 3230	1
Department of the Navy Office of Naval Research Washington 25, D. C. Attn: Code 429	1
Commanding Officer Office of Naval Research Branch Office 844 North Rush Street Chicago 11, Illinois Attn: LtJG. M. C. Laug	2
Dr. Elizabeth F. Riley Department of Chemistry Ohio State University Columbus 10, Ohio	1
Dr. E. R. Buchman California Institute of Technology Pasadena, California	2
Dr. Joseph W. Lang Director of Research General Aniline and Film Corporation Central Research Laboratory Lincoln and Coal Street Easton, Pennsylvania	1
Dr. Jerome Martin Commercial Solvents Corporation Terre Haute, Indiana	1

CONFIDENTIAL

Report No. 737

<u>ADDRESSEE</u>	<u>NO. OF COPIES</u>
Dr. G. B. Bachman Department of Chemistry Purdue University Lafayette, Indiana	1
Dr. T. L. Brownyard Bureau of Ordnance, Rs2c Department of the Navy Washington 25, D. C.	1
Dr. D. V. Sickman Explosives Division Naval Ordnance Laboratory White Oak Silver Spring 19, Maryland	1
Dr. William F. Sager Department of Chemistry George Washington University Washington, D. C.	
Dr. N. L. Drake Department of Chemistry University of Maryland College Park, Maryland	1
Dr. A. V. Tobolsky Frick Chemical Laboratory Princeton University Princeton, New Jersey	1
B. F. Goodrich Chemical Company Engineering Experimental Station Avon Lake, Ohio Attn: Dr. Wolfe	1
Commanding Officer Chemical Corps Chemical and Radiological Laboratories Army Chemical Center, Maryland Attn: Technical Library	1
Bureau of Aeronautics Department of the Navy Washington 25, D. C. Attn: TD-4	2

CONFIDENTIAL

Report No. 737

ADDRESSEE

NO. OF COPIES

Evans Research and Development Corp.
250 East 43rd Street
New York 17, New York
Attn: Mr. E. J. Hewitt

1

Commanding General
White Sands Proving Ground
Las Cruces, New Mexico
Attn: Technical Librarian

3

National Bureau of Standards
Ordnance Development Division
Washington 25, D. C.
Attn: Mr. J. Rabinow

1

Los Alamos Scientific Laboratory
Los Alamos, New Mexico
Attn: Technical Library

2