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

13 March 1952
Report No. 589
(Quarterly Summary)
Copy No. 21

# EXPLOSIVES RESEARCH

*Contract N7onr-46208*

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13 March 1952

Report No. 589
(Quarterly Summary)

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# EXPLOSIVES RESEARCH

Contract N7onr-46208

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Period Covered:

1 November 1951 through 31 January 1952

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AEROJET ENGINEERING CORPORATION

Azusa, California

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## CONTENTS

CONTENTS (cont.)

## CONTRACT FULFILLMENT

This quarterly summary report is submitted in partial fulfillment of Contract N7onr-46208.

## I. SUMMARY

A. This quarterly report is submitted under Contract N7onr-46208 and covers the period 1 November 1951 through 31 January 1952.[1] The objective of this contract is divided into three phases, as follows:

1. Synthesis and physical studies of new high-energy nitro compounds as potential explosives.

2. Desensitization of RDX with materials which will not lower its oxygen balance appreciably.

3. Development of bomb and shell casings made of an explosive plastic.

B. The more important results and conclusions of the work reported are presented below.

1. Methylene bis-(trinitroethyl carbamate) has been prepared by the addition of trinitroethanol to methylene diisocyanate in the presence of boron trifluoride catalyst.

2. Methylene N-nitro-bis-(trinitroethyl carbamate) was prepared by the nitration of methylene bis-(trinitroethyl carbamate) with a mixture of 100% nitric acid and acetic anhydride at 0 to 5°C.

3. Methylene N,N'-dinitro-bis-(trinitroethyl carbamate) was prepared by the nitration of methylene bis-(trinitroethyl carbamate) with a mixture of 100% nitric acid and acetic anhydride at 40 to 45°C.

4. bis-(3,3,3-Trinitropropyl) urea was prepared by treating 3,3,3-trinitropropyl isocyanate with tert-butyl alcohol.

5. N-Nitro-bis-(3,3,3-trinitropropyl) urea was prepared by nitrating bis-(3,3,3-trinitropropyl) urea with a mixture of 100% nitric acid and acetic anhydride at 0 to 5°C.

6. The following samples were prepared and submitted to the Naval Ordnance Laboratory for testing: (1) 1,1,1,3,5,5,5-heptanitropentane, (2) 2,2,4,6,6-pentanitroheptane, and (3) N-nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate.

7. A quantity of desensitized RDX was prepared by applying a coating of polymer I-A from solution, and 20 gm (designated Sample 1) was sent to the Naval Ordnance Laboratory for evaluation.

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[1] See also Aerojet Report No. 512 (14 June 1951); No. 538 (11 September 1951); No. 562 (14 December 1951).

## 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 nitro compounds with a preferred oxygen balance on the positive side or very near zero on the negative side.

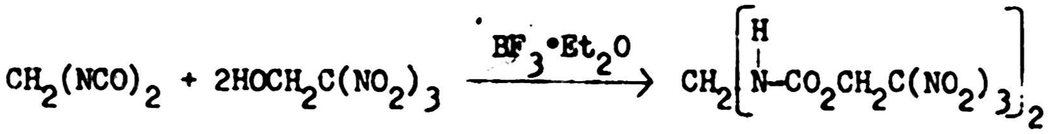
2. The present report deals with the preparation of four new potential explosives.

### B. PREPARATION OF METHYLENE bis-(TRINITROETHYL CARBAMATE)

#### 1. Discussion

a. The preparation of methylene bis-(trinitroethyl carbamate), by heating a mixture of trinitroethanol and trinitroethyl carbamate for 4 hr at 120°C, has been previously reported.[2]

b. In extending the work on the preparation of nitro carbamates as potential explosives, the utility of methylene diisocyanate was studied. It has been found that methylene diisocyanate will react in the presence of boron trifluoride etherate with two mols of trinitroethanol to form methylene bis-(trinitroethyl carbamate).



$$CH_2(NCO)_2 + 2HOCH_2C(NO_2)_3 \xrightarrow{BF_3 \cdot Et_2O} CH_2\left[\overset{\overset{H}{|}}{N}\text{-}CO_2CH_2C(NO_2)_3\right]_2$$

#### 2. Experimental

A 4.9-gm sample (0.05 mol) of methylene diisocyanate, 18.1 gm (0.1 mol) of trinitroethanol, 13.2 ml (0.1 mol) of boron trifluoride etherate, and 200 ml of dry, alcohol-free chloroform were placed in a 500-ml round-bottom flask, fitted with a condenser and drying tube. The solution was refluxed for 20 hr and concentrated in vacuo, leaving a white mushy solid. This product was dissolved in ether and the ether solution was washed with six 100-ml portions of water to remove the boron trifluoride. The ether solution, which turned yellow during the washings, was dried and concentrated in vacuo to give a yellow, viscous oil. The oil was recrystallized from chloroform to give 5.8 gm (46.6%) of a yellow crystalline solid, mp 115 to 118°C (dec.). The solid was dissolved in methylene chloride to give a yellow solution, and charcoal was added; on filtration, a colorless solution was obtained. The solution was evaporated to dryness in vacuo, giving a white solid. A sample was recrystallized three times from chloroform to give white needles, mp 125 to 126°C, I.S. = 100 cm/2 kg, and dried at 40°C and 2 mm for 4 hr.

---

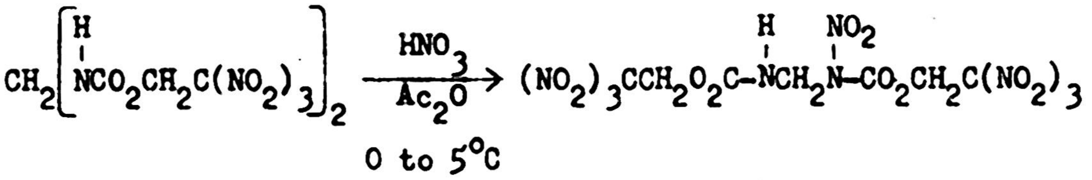
[2] Hercules Powder Co. Monthly Progress Report (4 August 1950).

Anal. Calc'd for $C_7H_8N_8O_{16}$: %C, 18.27; %H, 1.75; %N, 24.35
Found: %C, 18.40; %H, 1.64; %N, 23.73

## C. PREPARATION OF METHYLENE N-NITRO-bis-(TRINITROETHYL CARBAMATE)

### 1. Discussion

a. The nitration of methylene bis-(trinitroethyl carbamate) at 0 to 5°C with a mixture of 100% nitric acid and acetic anhydride gave methylene N-nitro bis-(trinitroethyl carbamate), a white crystalline solid, mp 103 to 104°C, with an oxygen balance of +1.6.



$$CH_2\left[\overset{H}{\overset{|}{N}}CO_2CH_2C(NO_2)_3\right]_2 \underset{0\text{ to }5^\circ C}{\xrightarrow[Ac_2O]{HNO_3}} (NO_2)_3CCH_2O_2C\text{-}\overset{H}{\overset{|}{N}}CH_2\overset{NO_2}{\overset{|}{N}}\text{-}CO_2CH_2C(NO_2)_3$$

### 2. Experimental

Ten ml of 100% nitric acid was placed in a 100-ml 3-necked flask, fitted with a mechanical stirrer, thermometer, and addition funnel. The flask was cooled in an ice-salt bath and 10 ml of acetic anhydride was added dropwise, followed by 1 gm of methylene bis-(trinitroethyl carbamate). The solution was stirred for 20 min and poured onto ice. A white solid was precipitated, which became an oil as the ice melted. The oil was dried in an evacuated desiccator over potassium hydroxide. The dried oil, wt 1.0 gm, was treated with charcoal and crystallized from chloroform to give a white solid. The product was recrystallized three more times from chloroform (mp 103 to 104°C).

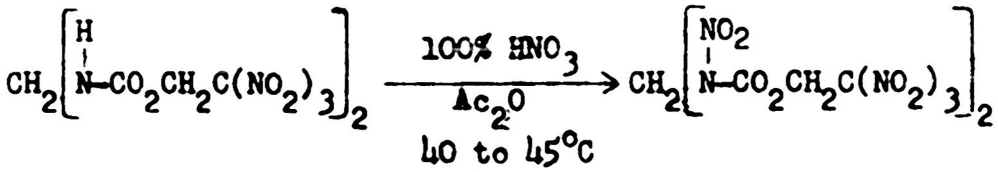
Anal. Calc'd for $C_7H_7N_9O_{18}$: %C, 16.64; %H, 1.40; %N, 24.96
Found: %C, 16.66; %H, 1.25; %N, 24.44

## D. PREPARATION OF METHYLENE-N,N'-DINITRO-bis-(TRINITROETHYL CARBAMATE)

### 1. Discussion

a. The nitration of methylene bis-(trinitroethyl carbamate) with a mixture of 100% nitric acid and acetic anhydride at 40 to 45°C gave a white crystalline solid which melted at 95 to 96°C, resolidified at about 102°C, and then melted again at 112 to 113°C. When this preparation was repeated a white crystalline solid was obtained which melted at 112 to 113°C without going through the lower melting phase. The mixed melting point was 112 to 113°C, and the analysis of both samples corresponded with that of methylene-N,N'-dinitro-bis-(trinitroethyl carbamate), indicating that the two samples are polymorphous. This compound has an oxygen balance of +8.7, I.S. = 25 to 30 cm/2 kg.

b. When a nitration mixture containing 100% nitric acid and acetic anhydride is heated, the temperature must be controlled very carefully. The solution was heated slowly to 40 to 45°C and then cooled quickly. If the temperature is permitted to rise much above 45°C a fume-off may occur.



$$CH_2\left[\overset{H}{N}\text{-}CO_2CH_2C(NO_2)_3\right]_2 \xrightarrow[\substack{Ac_2O \\ 40 \text{ to } 45^\circ C}]{100\%\ HNO_3} CH_2\left[\overset{NO_2}{N}\text{-}CO_2CH_2C(NO_2)_3\right]_2$$

2. **Experimental**

a. Fifteen ml of 100% nitric acid was placed in a 100-ml 3-necked flask, fitted with a mechanical stirrer, thermometer, and addition funnel. The flask was cooled in an ice bath and 15 ml of acetic anhydride was added dropwise, followed by 1 gm of methylene bis-(trinitroethyl carbamate). The solution was stirred for 15 min, permitted to warm to room temperature, heated at 40 to 45°C for 5 min, cooled, and poured onto ice. An oil was formed which crystallized to a white solid. The solid was collected, washed with water, and dried in vacuo. The yield of methylene N,N-dinitro bis-(trinitroethyl carbamate) was 1.2 gm, mp 92 to 93°C. Three recrystallizations from chloroform gave white needles, mp 95 to 96°C, which resolidified at 102°C and melted at 112 to 113°C; I.S. = 25 to 30 cm/2 kg.

Anal. Calc'd for $C_7H_6N_{10}O_{20}$: %C, 15.28; %H, 1.10; %N, 25.46
Found: %C, 15.53; %H, 1.26; %N, 24.96

b. Using the same procedure in the second run, the higher-melting form was isolated; the mp was 112 to 113°C, the mixed melting point was 112 to 113°C, and the I.S. was 25 to 30 cm/2 kg.

Anal. Calc'd for $C_7H_6N_{10}O_{20}$: %C, 15.28; %H, 1.10; %N, 25.46
Found: %C, 15.67; %H, 1.14; %N, 23.41

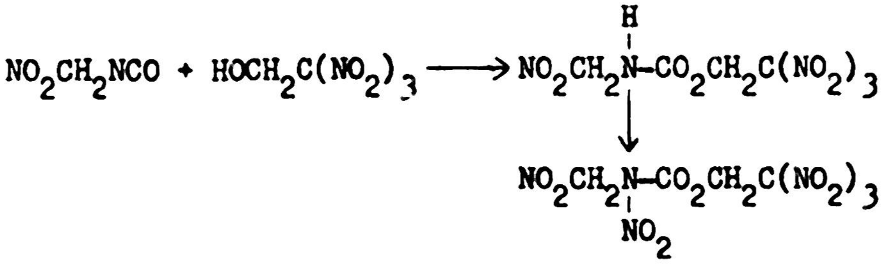
Although the nitrogen analysis showed considerable deviation from the theoretical, the identity of the compound was verified by the determination of the heat of combustion.

Heat of combustion, $\Delta H_p$, Predicted: 1251 cal/gm
Found: 1270 cal/gm

## E. ATTEMPTED PREPARATION OF NITROMETHYL ISOCYANATE

1. **Discussion**

a. Another nitro carbamate which may be of interest as a potential explosive is N-nitro-nitromethyl trinitroethyl carbamate, with an oxygen balance of +9.7; this compound could be prepared from nitromethyl isocyanate according to the following reaction:



$$NO_2CH_2NCO + HOCH_2C(NO_2)_3 \longrightarrow NO_2CH_2\overset{H}{N}-CO_2CH_2C(NO_2)_3 \downarrow NO_2CH_2\underset{NO_2}{N}-CO_2CH_2C(NO_2)_3$$

b. Ethyl nitrocyanoacetate has been converted through its hydrazide and azide to the corresponding ethyl urethane.[3] Analogously, it was expected that methyl nitroacetate could be converted through its hydrazide and azide to nitromethyl isocyanate:

$$NO_2CH_2CO_2CH_3 \xrightarrow{KOH} KO_2N{=}CHCO_2CH_3 \xrightarrow{N_2H_4} KO_2N{=}CHCONHNH_2$$

$$\xrightarrow[NaNO_2]{HCl} NO_2CH_2CON_3 \longrightarrow NO_2CH_2NCO$$

c. The procedure that was used was essentially the same as that employed by Daraksky.[4] Decomposition of the azide gave an oil which could not be purified. When the oil was refluxed with ethanol a faint yellow liquid was obtained, bp 80 to 90°C at 5 microns, $n_D^{25}$ 1.4646. The analysis did not correspond with that of ethyl nitromethyl carbamate. Found: %C, 40.77; %H, 4.66; %N, 11.22.

## F. ATTEMPTED PREPARATION OF 2,2,2-TRINITROETHYL-N-NITRO-N-2,2,2-TRINITROETHYL GLYCINE

### 1. Discussion

a. Inasmuch as 2,2,2-trinitroethyl-N-nitro-N-2,2,2-trinitroethyl glycine (I) is an isomer of N-nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate (II),[5] it was of interest to prepare this ester and compare its properties with those of the carbamate.

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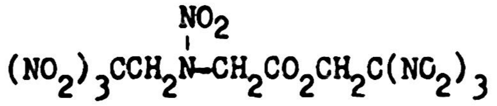
[3] Daraksky and Hillers, J. prakt. Chem. 92, 297 (1925).

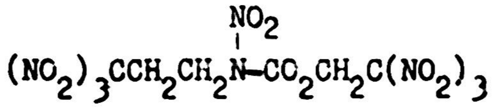
[4] Ibid.

[5] Aerojet Report No. 562 (14 December 1951).



$$(NO_2)_3CCH_2\overset{NO_2}{N}-CH_2CO_2CH_2C(NO_2)_3$$

(I)



$$(NO_2)_3CCH_2CH_2\overset{NO_2}{N}-CO_2CH_2C(NO_2)_3$$

(II)

When a chloroform solution of N-nitro-N-2,2,2-trinitroethyl glycyl chloride was refluxed with trinitroethanol, the only product isolated was ethyl N-nitro-N-2,2,2-trinitroethyl glycine. The presence of ethanol in the chloroform would account for these results. This reaction will be repeated using alcohol-free chloroform.

### 2. Experimental

a. A 7.1-gm quantity (0.026 mol) of N-nitro-N-trinitroethyl glycyl chloride, 4.76 gm (0.026 mol) of trinitroethanol, and 300 ml of chloroform, were placed in a 500-ml round-bottom flask fitted with a condenser and drying tube. The solution was refluxed for 66 hr and concentrated _in vacuo_. The gummy residue was dissolved in absolute ethanol and water was added; a white solid was precipitated. The product was collected, washed with water, and dried _in vacuo_; wt = 2.5 gm, mp 75 to 76°C. A sample was recrystallized six times from ethanol and water to give white crystalline needles, mp 77 to 78°C, which were dried at room temperature and 2 mm pressure over phosphorus pentoxide for 16 hr.

Anal. Calc'd for $C_6H_9N_5O_{10}$: %C, 23.15; %H, 2.89; %N, 22.51
Found: %C, 23.27; %H, 2.80; %N, 21.80

## G. ATTEMPTED PREPARATION OF N-NITRO-3,3,3-TRINITROPROPYL AMINE

### 1. Discussion

a. The attempts to hydrolyze the N-nitro-N-3,3,3-trinitropropyl carbamates to N-nitro-3,3,3-trinitropropyl amine have been described previously.[6]

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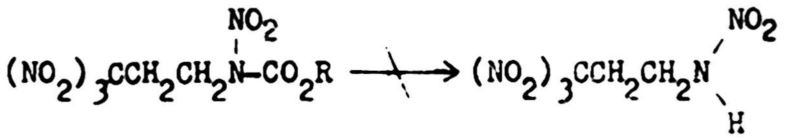
[6] Ibid.



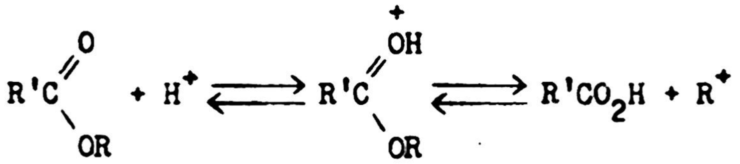
$$(NO_2)_3CCH_2CH_2N(NO_2){-}CO_2R \nrightarrow (NO_2)_3CCH_2CH_2N(NO_2)H \qquad (1)$$

where

R = ethyl, trinitroethyl, nitrophenyl, or cyclohexyl

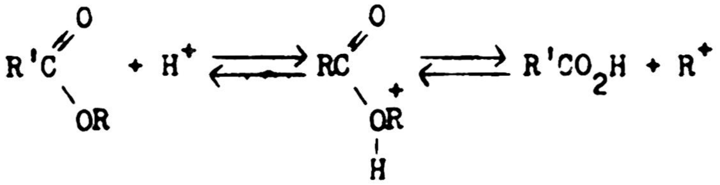
b. The hydrolysis of ethyl N-nitro-N-isopropyl carbamate to isopropyl-N-nitroamine by refluxing with 50% sulfuric acid has recently been reported.[7] When N-nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate was treated in this way, complete decomposition occurred. Newman[8] has found that certain esters which are difficult to hydrolyze can be hydrolyzed by dissolving in 100% sulfuric acid and pouring into water to give a 90% yield of the acid. The hydrolysis of N-nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate in 100% sulfuric acid unexpectedly gave N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate, indicating the high stability of the trinitroethoxy group and the preferential removal of the N-nitro group in strong acid medium. The hydrolysis of N-nitro-N-3,3,3-trinitropropyl ethyl carbamate was tried at a higher pH, with a solution of glacial acetic acid and sodium acetate; here again either starting material was recovered or complete decomposition occurred.

c. Several reasonable mechanisms for the acid-catalyzed hydrolysis of the esters of primary and secondary alcohols may be suggested *a priori*:



$$R'C(=O)OR + H^+ \rightleftharpoons R'C(=\overset{+}{O}H)OR \rightleftharpoons R'CO_2H + R^+ \qquad (2)$$

$$R^+ + H_2O \longrightarrow ROH + H^+ \qquad (2a)$$



$$R'C(=O)OR + H^+ \rightleftharpoons RC(=O)\overset{+}{O}(H)R \rightleftharpoons R'CO_2H + R^+ \qquad (3)$$

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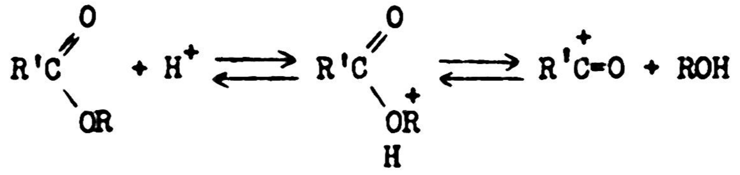
[7] Curry and Mason, J. Am. Chem. Soc. 73, 5043 (1951).

[8] J. Am. Chem. Soc. 63, 2431 (1941).

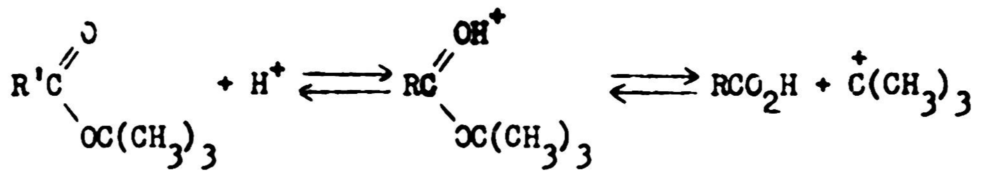
$$R^+ + H_2O \longrightarrow ROH + H^+ \qquad (3a)$$



$$R'C(=O)OR + H^+ \rightleftharpoons R'C(=O)\overset{+}{O}(R)H \rightleftharpoons R'\overset{+}{C}{=}O + ROH \qquad (4)$$

$$R'\overset{+}{C}{=}O + H_2O \longrightarrow R'CO_2H + H^+ \qquad (4a)$$

It will be noticed that mechanisms (2) and (3) postulate the transitory existence of $R^+$ ion. That such an ion is probably not produced in acid hydrolysis, and hence that mechanism (4) is the only one of the three which is possible, was demonstrated by Ingold.[9] A different mechanism operates in the hydrolysis of carboxylic esters of tert-butyl alcohol.[10]



$$R'C(=O)OC(CH_3)_3 + H^+ \rightleftharpoons RC(=\overset{+}{O}H)OC(CH_3)_3 \rightleftharpoons RCO_2H + \overset{+}{C}(CH_3)_3 \qquad (5)$$

$$\overset{+}{C}(CH_3)_3 + H_2O \longrightarrow HOC(CH_3)_3 + H^+ \qquad (5a)$$

Inasmuch as reactions (4) and (5) are equilibrium reactions, the driving force will be the stability of the carbonium ion which is formed. Since the tert-butyl carbonium ion is the more stable, the hydrolysis proceeding according to mechanism (5) should be easier.

d. The above considerations indicate that the best chance of achieving success in reaction (1) would be if R were a group such as tert-butyl. The synthesis of N-nitro-N-3,3,3-trinitropropyl tert-butyl carbamate posed a problem. It was recognized that tert-butyl alcohol reacts abnormally with some isocyanates to form ureas:

$$(CH_3)_3COH \longrightarrow H_2O + CH_2{=}C(CH_3)_2$$

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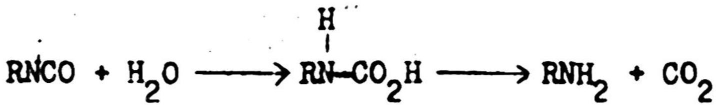
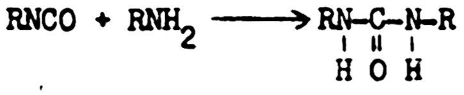
[9] J. Chem. Soc., 756 (1932).

[10] Cohen and Schneider, J. Am. Chem. Soc. 63, 3382 (1941).



$$RNCO + H_2O \longrightarrow R\overset{H}{N}\text{-}CO_2H \longrightarrow RNH_2 + CO_2$$



$$RNCO + RNH_2 \longrightarrow R\underset{H}{N}\text{-}\underset{O}{\underset{\|}{C}}\text{-}\underset{H}{N}\text{-}R$$

while it reacts normally with other isocyanates, such as phenyl isocyanate, to form the corresponding carbamate. Unfortunately, the reaction of 3,3,3-trinitropropyl isocyanate and tert-butyl alcohol proceeded abnormally, giving bis-(3,3,3-trinitropropyl) urea.

## 2. Experimental

### a. Hydrolysis of N-Nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl Carbamate with 50% Sulfuric Acid

One-half gm of N-nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate and 14 ml of 50% sulfuric acid was placed in a 50-ml round-bottom flask fitted with a condenser. After the reaction mixture was heated at 110°C for 2 hr, complete decomposition had occurred.

### b. Hydrolysis of N-Nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl Carbamate with 100% Sulfuric Acid

One gm of N-nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate and 44 ml of 100% sulfuric acid was placed in a 125-ml Erlenmeyer flask. The reaction mixture was heated on the steam bath for 5 min, at which time solution was effected. The solution was permitted to stand for ten minutes and then poured onto ice. The white solid was collected and dried, wt = 0.6 gm, mp 91 to 92°C, I.S. > 100 cm/2 kg. A sample (mp 92 to 92.5°C) was recrystallized three times from carbon tetrachloride; the mixed melting point with N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate (mp 91 to 92°C) was 90 to 92°C.

Anal. Calc'd for $C_6H_7N_7O_{14}$: %C, 17.96; %H, 1.76; %N, 24.44
Found: %C, 18.41; %H, 2.09; %N, 23.47

### c. Hydrolysis of N-Nitro-N-3,3,3-trinitropropyl Ethyl Carbamate with Glacial Acetic Acid and Sodium Acetate

(1) One-half gm of N-nitro-N-3,3,3-trinitropropyl ethyl carbamate, 0.1 gm of sodium acetate, and 20 ml of glacial acetic acid was placed in a 50-ml round-bottom flask fitted with a condenser. The solution was heated on the steam bath for 2 hr and poured onto ice. A quantitative yield of starting material was recovered.

(2) A 0.4-gm quantity of N-nitro-N-3,3,3-trinitropropyl ethyl carbamate, 0.2 gm of sodium acetate, and 20 ml of glacial acetic acid was placed in a 50-ml round-bottom flask fitted with a condenser. The solution was heated on the steam bath for 19 hr and poured onto ice. Starting material (0.1 gm) was recovered.

(3) One-half gm of N-nitro-N-3,3,3-trinitropropyl ethyl carbamate, 1.0 gm of sodium acetate, and 20 ml of glacial acetic acid was placed in a 50-ml round-bottom flask fitted with a condenser. The solution was heated on the steam bath for 4.5 hr and poured onto ice. A clear solution was obtained, indicating complete decomposition.

## H. PREPARATION OF bis-(3,3,3-TRINITROPROPYL) UREA

### 1. Discussion

The discussion of the preparation of bis-(3,3,3-trinitropropyl) urea is included in Section G, c, above.

### 2. Experimental

A 2.2-gm quantity (0.01 mol) of 3,3,3-trinitropropyl isocyanate, and 10 ml of tert-butyl alcohol was placed in a 50-ml round-bottom flask, fitted with a condenser and drying tube. On mixing, an exothermic reaction occurred. The solution was refluxed for 2 hr, and after cooling a white crystalline solid separated. The yield of bis-trinitropropyl urea was 2.0 gm (68%). A sample was recrystallized three times from chloroform and dried at 4J.°C and 2 mm for 3 hr; the mp was 171 to 173°C (dec.), I.S. = 60 to 65 cm/2 kg.

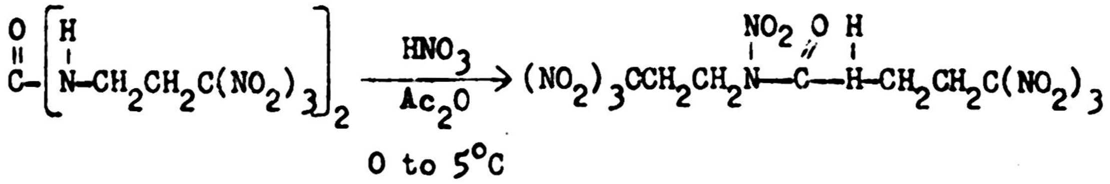
Anal. Calc'd for $C_7H_{10}N_8O_{13}$: %C, 20.30; %H, 2.43, %N, 27.05

Found: %C, 20.36; %H, 2.28; %N, 26.93

## I. PREPARATION OF N-NITRO-bis-(3,3,3-TRINITROPROPYL) UREA

### 1. Discussion

The nitration of bis-(3,3,3-trinitropropyl) urea with 100% nitric acid and acetic anhydride at 0 to 5°C gave N-nitro-bis-(3,3,3-trinitropropyl) urea, a white crystalline solid, mp 78 to [illegible]°C, I.S. = 25 to 30 cm/2 kg, with an oxygen balance of -12.2.



$$O{=}C\left[\overset{H}{\overset{|}{N}}{-}CH_2CH_2C(NO_2)_3\right]_2 \xrightarrow[Ac_2O,\ 0\ \text{to}\ 5^\circ C]{HNO_3} (NO_2)_3CCH_2CH_2\overset{NO_2}{\overset{|}{N}}{-}\overset{O}{\overset{\|}{C}}{-}\overset{H}{\overset{|}{H}}{-}CH_2CH_2C(NO_2)_3$$

### 2. Experimental

Ten ml of 100% nitric acid was placed in a 100-ml 3-necked flask, fitted with a mechanical stirrer, thermometer, and addition funnel. The flask was cooled in an ice bath and 10 ml of acetic anhydride was added dropwise, followed by 1.4 gm of bis-(3,3,3-trinitropropyl) urea. The solution was stirred for 15 min and poured onto ice. A yellow oil was formed which crystallized on standing. The cream-colored solid was collected, washed with water, and dried in vacuo. The yield of N-nitro-bis-(3,3,3-trinitropropyl) urea was 1.6 gm, mp 75 to 78°C. The product was treated with hot chloroform and charcoal and a small amount of oil separated. The solution was decanted from the oil and on cooling a white solid separated, mp 78 to 79°C, I.S. = 25 to 30 cm/2 kg. Another recrystallization from chloroform did not change the melting point.

Anal. Calc'd for $C_7H_9N_9O_{15}$: %C, 18.31, %H, 1.98; %N, 27.45
Found: %C, 18.44; %H, 1.95; %N, 27.15

The explosive properties of these new polynitro compounds are listed in Table I.

## J. ATTEMPTED PREPARATION OF 2,2,4,4-TETRANITRO-1,5-PENTANEDIOL DINITRATE

### 1. Discussion

It was of interest to nitrate 2,2,4,4-tetranitro-1,5-pentanediol and characterize the properties of the dinitrate. This compound, with an oxygen balance of +4.3, might be of value to the explosives program if it were sufficiently stable and possessed the desired physical properties. Nitration of 2,2,4,4-tetranitro-1,5-pentanediol gave an oil which was so unstable that it fumed off on standing at room temperature.

### 2. Experimental

Twenty-five ml of 100% white nitric acid was placed in a 200-ml 3-necked flask, fitted with a mechanical stirrer, thermometer, and dropping funnel. The flask was cooled in an ice-salt bath and 3.0 gm of 2,2,4,4-tetranitro-1,5-pentanediol was added. The solution was stirred for 20 min and 25 ml of concentrated sulfuric acid was added. After stirring for an additional 15 min, the solution was poured onto ice, causing a white oil to separate. The oil was extracted with methylene chloride and dried over sodium sulfate; on standing overnight the solution turned yellow. The solution was concentrated in vacuo, leaving a viscous brown oil which fumed off.

## TABLE I

## EXPLOSIVE PROPERTIES OF NITRO COMPOUNDS

| Compound | Oxygen Bal. to $CO_2$ | mp °C | I.S. cm/2 kg | Calc. Heat of Explos. cal/gm | Calc. Force cal/gm | Calc. Lead-Block Value (TNT=100) | Calc. Ballistic Mortar Value (TNT=100) |
|---|---|---|---|---|---|---|---|
| RDX (reference) | -21.6 | 201 | 28 | 1221 | 345 | 157 | 150 |
| Methylene bis-(trinitroethyl carbamate) | -7.0 | 125 to 126 | 100 | 1228 | 280 | 155 | 130 |
| Methylene N-nitro-bis-(trinitroethyl carbamate) | +1.6 | 103 to 104 | -- | 1358 | 286 | 167 | 132 |
| Stoichiometric mixture with TNT (1.7 wt % TNT) | 0 | -- | -- | 1391 | 290 | 170 | 134 |
| Methylene N,N'-dinitro-bis-(trinitroethyl carbamate) | +8.7 | 95 to 96<br>112 to 113 | 25 to 30 | 1223 | 269 | 154 | 126 |
| Stoichiometric mixture with TNT (10.6 wt % TNT) | 0 | -- | -- | 1459 | 299 | 176 | 138 |
| bis-Trinitropropyl Urea | -23.2 | 171 to 173 | 60 to 65 | 1124 | 310 | 145 | 142 |
| N-Nitro-bis-trinitropropyl Urea | -12.2 | 78 to 79 | 25 to 30 | 1350 | 325 | 166 | 148 |

## K. 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. 9.4 gm of 1,1,1,3,5,5,5-heptanitropentane, mp 103 to 104°C, I.S. = 30 cm/2 kg (recrystallized from 70% nitric acid at 65°C, in the same manner as earlier samples)

b. 9.3 gm of 1,1,1,3,5,5,5-heptanitropentane, mp 103 to 104°C, I.S. = 30 cm/2 kg (recrystallized by dissolving in 100% nitric acid at room temperature and adding water; this procedure should be a means of avoiding nitration or nitrosation)

c. 28.3 gm of 2,2,4,6,6-pentanitroheptane, mp 82 to 83°C, I.S. $>$ 100 cm/2 kg

d. 7.0 gm of N-nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate, mp 96 to 97°C, I.S. = 30 cm/2 kg

2. The conclusions reached by the Naval Ordnance Laboratory with respect to these compounds are as follows:

a. 1,1,1,3,5,5,5-Heptanitropentane

(1) The sample recrystallized at room temperature from 100% nitric acid and water is of higher purity than that recrystallized from 70% nitric acid at 65°C.

(2) Heptanitropentane is definitely unstable above its melting point and could never be safely cast on a large scale. Furthermore, its stability in the solid state at 90°C appears to be fairly poor, and the possibility of improvement does not appear to be promising at this time.

b. 2,2,4,6,6-Pentanitroheptane

(1) This compound was requested because its melting point suggests that if it were stable in the liquid state it would be of some use as a castable compound similar to TNT.

(2) The thermal stability is poor in the liquid state; furthermore, because of its relatively low density and oxygen content and the need for desensitization, there is no further interest in this compound at the present time.

c. N-Nitro-N-3,3,3-Trinitropropyl-2,2,2-trinitroethyl Carbamate

This compound possesses excellent thermal stability at 90°C but poor stability at 100°C. As it melts at 96°C, it belongs in the class of explosives that exhibit good stability in the solid state but poor stability in the liquid state. Such compounds cannot be cast but can be pressed satisfactorily. The compound has a high density, which is very desirable; however, it would require elaborate desensitization, which would reduce the density and effective oxygen content somewhat. It will be evaluated as a primary explosive. The results are summarized in Table II.

## III. TECHNICAL PROGRESS: DESENSITIZATION OF RDX

### A. INTRODUCTION

It was shown in the previous report[11] that RDX could be desensitized (1) by suspending it in an aqueous medium and precipitating a nitro polymer from emulsion on the RDX crystals, and (2) by suspending it in an aqueous slurry and applying a thin coating of a nitro polymer from solution in a solvent compatible with the slurry medium. During the present quarter, the conditions for successful coating by these methods have been defined more clearly.

### B. DISCUSSION

#### 1. Desensitization by Coating from Emulsion

a. The best emulsion of poly-dinitrobutyl acrylate (N-245) was prepared by shaking a mixture of 10 gm of dinitrobutyl acrylate, 0.1 gm of partially hydrolyzed (86 to 89%) polyvinyl alcohol of medium viscosity (20 to 25 cps), 0.5 gm of Nonisol 210, 0.02 gm of methyl amyl ketone peroxide, and 40 ml of water in a 120-ml narrow-mouth bottle for 92 to 97 hr at 45°C. The polymer itself is soft and rubbery. The amount of polymer per gram of emulsion was determined by precipitating the polymer with methanol from a known weight of the emulsion.

b. The coating of RDX was carried out using the emulsions containing 2 to 10% of poly-dinitrobutyl acrylate in the presence of 2.5% Span 85 as a wetting agent. The coated samples of RDX had impact stabilities varying from 36 to >100 cm/2 kg. It was difficult to obtain reproducible results, particularly when the amount of RDX used was increased. It is believed that these difficulties are due to the nonhomogeneity of the emulsion itself, which results from the use of impure 2,2-dinitrobutyl acrylate. Work on the preparation of a purer 2,2-dinitrobutyl acrylate is now in progress.

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[11] Aerojet Report No. 562 (14 December 1951), p. 14.

## TABLE II

## PRELIMINARY TESTS ON EXPERIMENTAL EXPLOSIVES

| Compound | | 1,1,1,3,5,5,5-Heptanitropentane | | 2,2,4,6,6-Pentanitroheptane | N-Nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl Carbamate |
|---|---|---|---|---|---|
| NOL Number | | A-51-2c(1) | A-51-2c(2) | A-51-3 | A-51-4 |
| Melting Point, °C | | 100 to 103 | 102 to 103 | 80 to 83 | 95 to 97 |
| Crystal Density, $gm/cm^3$ | | not redetermined (previous value 1.898) | | 1.622 | 1.816 |
| Impact Sensitivity, cm/2.5 kg | | 10 | 12 | 56 | 8 |
| Hot-Bar Ignition Temperature, °C | | 189 | 195 | 386 | 214 |
| Thermal Stability (cc gas/gm in 48 hr) | 100°C | — | — | decomposed completely | 18.2 |
| | 90°C | 14 | 6.09 | 3.15 | 0.5 |

(1) Crystallized from hot 70% nitric acid

(2) Crystallized from cold 100% nitric acid and water

### 2. Desensitization by Coating from Solution

a. In the best preparation made to date by this process of coating from solution, the RDX is coated with 5% polymer I-A (polyurethane of 2,2-dinitro-1,3-propanediol and 3,3-dinitro-1,5-pentane diisocyanate), using 4% Atlas Span 85 as a surface-active agent. Using this procedure, 20 gm of coated RDX was prepared and submitted to the Naval Ordnance Laboratory for evaluation. On the NOL impact-test machine, the 2.5-kg weight caused explosion in 50% of the trials, when dropped from a height of 49 cm. The explosive was screened and the part passing through the 16-mesh and retained on the 30-mesh screen was mixed with the part passing through the 30-mesh and retained on the 50-mesh screen. This mixture exploded 50% of the time under the impact of a 2.5-kg weight dropped from a height of 47 cm. These results may be compared with the following sensitivities of common explosives: PETN = 12, RDX = 24, Comp. B = 60, and Comp. A-3 = 59.

b. This preparation has the following defects:

(1) Too much surface-active agent is needed.

(2) Its stability falls short of the values which appear to be attainable by this process. This defect is attributed to the known hardness and brittleness of polymer I-A. The polymer is slightly softened by the presence of Span 85, which thus acts as a modifier in the polymeric coating as well as a surface-active agent. However, the compatibility of polymer I-A with Span 85 is not good. A polymer which is intrinsically soft and pliable should give better desensitization, and at the same time require less surface-active agent in its application.

### 3. Testing Procedures

a. Impact stabilities were measured on the modified impact machine of the Bureau of Mines type with a 2-kg weight, using samples weighing approximately 0.02 gm. The values reported are the heights of fall that resulted in explosion in 50% of the tries.

b. Accelerated aging tests were conducted by storing samples at 50°C and 65°C, removing after various intervals of time, and testing for impact stability.

c. Certain samples were molded without heating in a 3/4-in.-dia. mold in a Carver laboratory press at pressures of 11,000 and 13,900 psi.

### 4. Relation of Impact Stability and Physical Form

a. The coated RDX obtained by the emulsion technique was a uniform fine powder, while there were large differences in the states of aggregation of the RDX obtained from the various preparations made by the solution procedure. These latter products were isolated as large amorphous masses, as small globules, as friable crusts, or as powders.

b. Impact stability, as determined on the Bureau of Mines apparatus, depends to some extent on the form of the sample which is tested. In general, when large particles are ground the impact stability of the resulting powder is less than that of the original particles. Because of the possibility of dislodging the protective coating in grinding, it is not known what part of this decrease, if any, is due to smaller particle size as such. In this investigation, samples were often tested as originally obtained from the drying operation. In addition, other test samples were prepared by crushing the original particles to a fine powder on a polyethylene sheet with a Teflon roller. This procedure was followed because the more lumpy samples must be prepared in some way for testing, either by crushing or slicing or powdering, and it was hoped that crushing all preparations in this manner would make the results more nearly comparable. This crushing is not, of course, a reproducible operation, and the lack of standardization is recognized.

c. The effect of crushing was investigated quantitatively with some of the desensitized RDX prepared by the solution procedure for NOL evaluation. This material was crushed by the method described, and the resulting powder was separated into fractions according to particle size by sieving. The impact stabilities of these fractions are:

| | |
|---|---|
| Original | >100 cm/2 kg |
| Crushed powder before sieving | 90 to >100 |
| >80 mesh (ca. 40%) | 80 to >100 |
| 80 to 100 mesh (ca. 10%) | <75 to 85 |
| <100 mesh (ca. 50%) | 65 to 70 |
| <200 mesh (small) | -- |
| Uncoated RDX | 28 to 32 |

## 5. Function of the Span 85 in Coating from Solution

a. The amount of Span 85 required, 4% by weight of the RDX, is obviously much greater than the amount needed only to provide an active layer covering the RDX crystals. What happens to the remainder is of great interest. In some preliminary experiments, portions of liquid that separated from the coated RDX were evaporated to dryness and the residue was weighed; after correcting for the small quantity of RDX normally present in solution, the amount of residue remaining was undetectable. To test further this indication that all of the Span 85 is present in the solid phase, two portions of globules of desensitized RDX (from which the finest particles had been removed by screening) were crushed lightly and thoroughly extracted in a Soxhlet apparatus with carbon tetrachloride. (The solubilities of RDX and polymer I-A are negligible in carbon tetrachloride.) These extracts were evaporated to dryness, giving residues whose refractive indices closely checked the values for Span 85. The yields were:

| Wt Coated RDX gm | Wt Residue gm | Wt Span in Preparation gm | Recovery of Span 85 % |
|---|---|---|---|
| 1.955 | 0.062 | 0.072 | 86 |
| 2.851 | 0.081 | 1.050 | 77 |

This experiment proves that most of the Span 85 is taken up by the solid during the preparation. It may be presumed that the large excess over the amount needed to form an active layer over the RDX crystals enters into the polymeric coating, modifying its properties.

b. The compatibility of polymer I-A and Span 85, in the ratio used in the basic preparation, was investigated by dissolving the proper amounts together in a small quantity of acetone, evaporating the solution on a glass plate, and inspecting the residual film. The film was softer than the unmodified polymer, but it was also mechanically weak, and showed a marked separation of the two components (Figure 1). This result is considered encouraging, because it implies that much improvement is possible by selection of a better coating material.

### 6. Investigation of Variables in Coating from Solution

#### a. The Basic Preparation

(1) In the basic preparations used in coating from solution, 1.00 gm of RDX (impact stability, 28 to 32 cm/2 kg) was suspended in 15 ml water and 5 ml acetone containing 0.04 gm Atlas Span 85 as a surface-active agent. This suspension was stirred rapidly in a small flask, and a solution of 0.050 gm of polymer I-A (the polyurethane of 2,2-dinitro-1,3-propanediol and 3,3-dinitro-1,5-pentane diisocyanate) was introduced slowly through a fine tube below the surface. The RDX crystals were coated and agglomerated into small globules. The dried product had an impact stability of $> 100$ cm/2 kg; when the material was sliced thin or powdered the impact stability was from 85 to 90 to $> 100$ cm/2 kg.

(2) A photomicrograph of typical material prepared in this way, compared with uncoated RDX, is shown in Figure 2.

(3) A number of preparations were made using double quantities of the above-mentioned materials, and the products were composited; 20 gm of this composite (designated Sample 1) was sent to the Naval Ordnance Laboratory for further evaluation. The results of these tests are reported in Section III B 2, above.

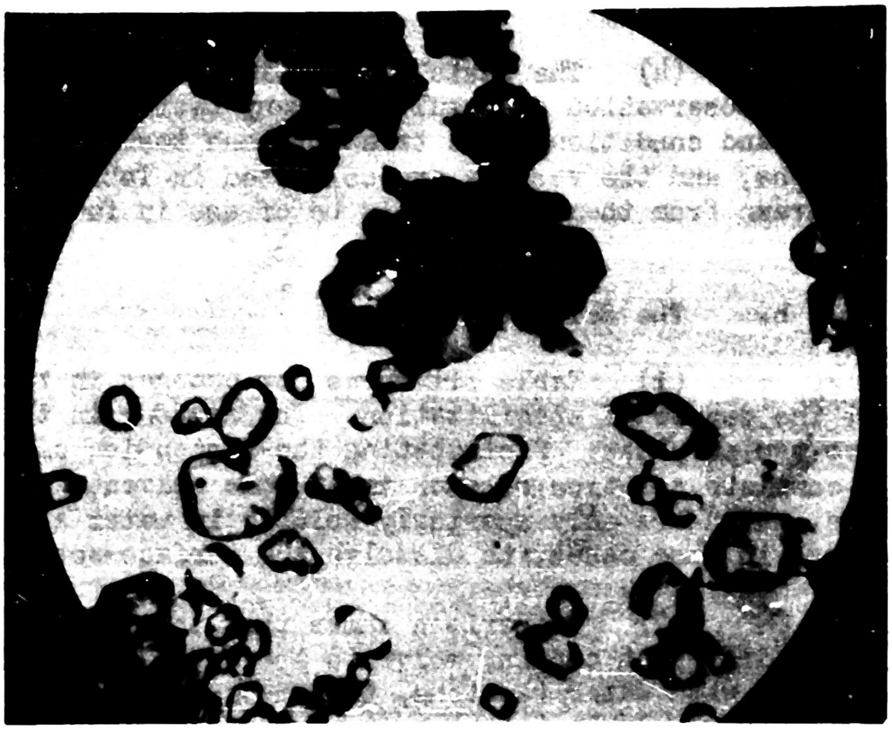

Figure 1. Compatibility of 5 Parts of Polymer I-A and 4 Parts of Span 85

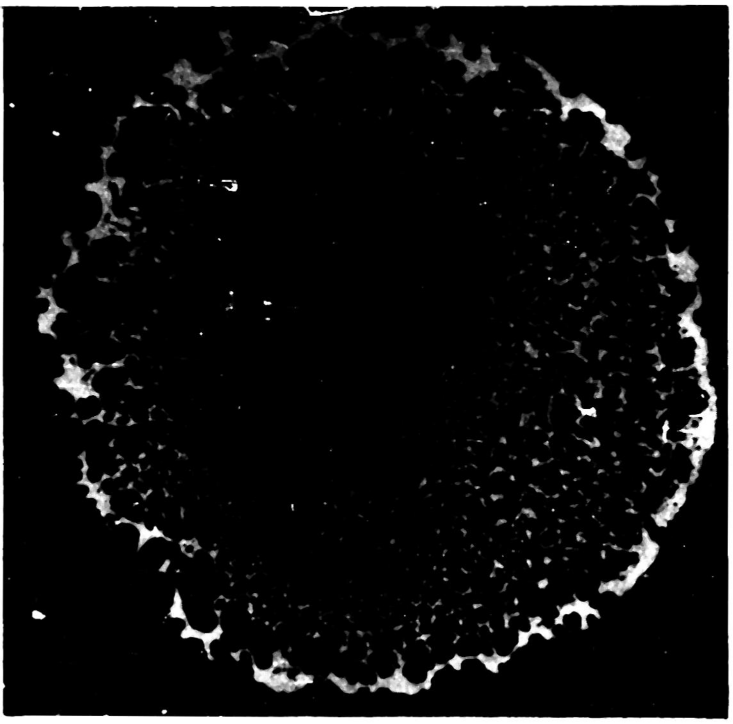

Figure 2. Contrast Between Desensitized (Upper) and Original (Lower) RDX (Desensitization with 5% polymer I-A, using 4% Span 85; magnification, 210X)

(4) The basic preparation described above was selected as best from observation of results of many tests in which different materials, amounts, and conditions were tried. These tests are described in the following paragraphs, and the results are collected in Table III. Certain conclusions can be drawn from them that should be of use in future improvements of the process.

b. The Solvent System

(1) Table III shows how changes in the system of solvents affect the degree of stabilization. The acetone in the aqueous medium appears to serve two purposes: facilitating the suspension of the RDX, and permitting the complete solution of such immiscible solvents as ethyl acetate and nitromethane, which are only sparingly soluble in water alone. (It may also be necessary, in some cases, to dissolve the surface-active agent.)

(2) Comparison shows that the degrees of desensitization achieved with different coating solvents are inferior with solvents which are readily miscible with water (acetone, tetrahydrofuran) and superior with solvents only sparingly soluble (ethyl acetate, nitromethane). It is believed that best desensitization is attained with a high solvent activity, just short of that needed to give a phase separation (2.3 ml ethyl acetate clouds 15 ml water + 5 ml acetone). This belief is supported by further experiments using both less ethyl acetate and enough more to give an appreciable amount of a separate liquid phase. The optimum stability attained when the ethyl acetate is nearly, but not quite, separated from the main liquid phase is very striking.

(3) One experiment was conducted in which all liquid volumes were reduced to one-half. The data are tabulated in the section on the effect of changes in the surface-active agent (Paragraph 6 c), since the amount of Span 85 was also reduced to 0.02 gm. Comparison there with the impact stability for the preparation with the normal volumes of liquid and 0.02 gm of Span shows that the reduction of liquid volumes results in decreased stability.

c. Surface-Active Agents

(1) There is no doubt of the specific effect of the surface-active agent on success or failure of the coating, as illustrated by Table III. The agent should have an affinity for RDX and, on the other hand, should assist the uniform precipitation of the coating from its organic medium. Lipophilic agents should be most successful.

(2) An application of 10% polymer I-A was made using the typical liquid system without surface-active agent. The impact stability of the pulverized product was equal to or slightly below 65 cm/2 kg, appreciably less than that of samples prepared using the best surface-active agents.

### TABLE III

### EFFECTS OF CHANGING CONDITIONS OF COATING ON DESENSITIZATION OF RDX

(Amounts in Each Experiment Based on 1.00 gm RDX)

| Reference Paragraph in Text | Slurry Medium | Coating Solution | Impact Stability, cm/2 kg* | |
|---|---|---|---|---|
| | | | Original | Crushed |
| 6 b | 15 ml water + 3 ml acetone + 0.04 gm Span 85 | 0.050 gm I-A in 2 ml acetone | — | 40 to 45 |
| | + 2 ml acetone + 0.04 gm Span 85 | 2 ml tetrahydrofuran | — | 60 to 65 |
| | 15 ml water + 5 ml acetone + 0.04 gm Span 85 | 2 ml nitromethane | — | 75 to 80 |
| | ↓ | 2 ml ethyl acetate | >100 | 85 to 90 to >100 |
| | ↓ | 1 ml EtOAc | >90 | <20 to 25,<br>35 to 40 |
| | ↓ | 3 ml EtOAc | — | 30 |
| 6 c | 15 ml water + 5 ml acetone | 0.100 gm I-A in 2 ml EtOAc | — | >65 |
| | + 0.04 gm lecithin | 0.100 gm I-A in 2 ml EtOAc | — | 20 to 30 |
| | + 0.04 gm glyceryl mono oleate | 0.050 gm I-A in 2 ml EtOAc | — | >95 |
| | + 0.04 gm R & H Triton X-100 | ↓ | — | 30 to 40 |
| | + 0.04 gm R & H Triton X-155 | ↓ | — | 40 to 45 |
| | + 0.04 gm Marasperse C | ↓ | — | 30 to 35 |
| | + 0.04 gm Atlantic Ultrawet 35-KX | ↓ | <55 to>65 | 30 |
| | 15 ml water + 5 ml acetone + 0.01 gm Span 85 | ↓ | 90 | 25 to 30 |
| | + 0.02 gm Span 85 | ↓ | 85 to 90 | 40 to 55 |
| | + 0.04 gm Span 85 | ↓ | — | 85 to 90 to >100 |
| | 7.5 ml water + 2.5 ml acetone + 0.02 gm Span 85 | ↓ | 75 to 80 | 35 to 40 |
| | + 0.02 gm glyceryl mono oleate | ↓ | 85 to 90 | 30 to 50 |
| | 15 ml water + 5 ml acetone + 0.04 gm Prosol 421 | ↓ | 40 | 20 to 25 |
| | + 0.04 gm Prosol 428 | ↓ | 55 | 45 to 55 |
| | + 0.04 gm Prosol 431 | ↓ | 55 to 60 | 35 to 40 |
| | + 0.04 gm Prosol 315 | ↓ | 40 to 50 | 30 to 50 |
| | + 0.04 gm Prosol 305 | ↓ | 50 to 70 | 30 to 50 |
| | + 0.04 gm Prosol 307 | ↓ | 70 to >100 | >100 |
| 6 d | 15 ml water + 5 ml acetone + 0.04 gm Span 85 | 0.050 gm Poly TNEMA in 2 ml EtOAc | 40 to 45 | — |
| | ↓ | 0.050 gm PEATN in 2 ml EtOAc | — | 65 to 70 |
| | ↓ | 0.050 gm Poly PEATN in 2 ml EtOAc | — | 15 to 20 |
| | 15 ml water + 5 ml acetone + 0.01 gm Span 85 | 0.050 gm Poly PEATN in 2 ml EtOAc | — | 15 to 20 |
| | 15 ml water + 5 ml acetone + 0.01 gm Span 85 | 0.050 gm I-J in 2 ml EtOAc | 60 | 25 to 40 |
| | 15 ml water + 5 ml acetone + 0.04 gm Span 85 | 0.020 gm I-D in 2 ml EtOAc | 65 to 70 | — |
| | ↓ | 0.100 gm I-D in 2 ml EtOAc | — | >100 |
| | ↓ | 0.020 gm I-A in 2 ml EtOAc | — | 40 to 45 |
| | ↓ | 0.050 gm I-A in 2 ml EtOAc | — | 85 to 90 to >100 |
| | ↓ | 0.100 gm I-A in 2 ml EtOAc | — | 85 to >100 |
| | 15 ml water + 5 ml acetone + 0.02 Span 85 + 0.02 gm lanolin | 0.050 gm I-A in 2 ml EtOAc | — | 60 to 65 |
| | 15 ml water + 5 ml acetone + 0.02 gm Span 85 | 0.050 gm I-A + 0.02 gm lanolin in 2 ml EtOAc | — | 40 to 55 |
| | ↓ | 0.050 gm I-A + 0.02 gm Emery 955 dimer acid in 2 ml EtOAc | — | 75 to 100 |
| | ↓ | 0.050 gm I-A + 0.02 gm polyvinyl acetate in 2 ml EtOAc | — | 25 to 30 |
| | ↓ | 0.050 gm I-A + 0.02 gm hexadecanol in 2 ml EtOAc | — | 40 to 55 |
| | 15 ml water + 5 ml acetone + 0.01 gm Span 85 | 0.050 gm I-A + 0.01 gm gum rubber in 2 ml cyclohexanone | 50 to 55 | 35 to 40 |

*For untreated RDX, 28 to 32 cm/2 kg

(3) A number of agents of supposedly lipophilic tendencies were tried. At first these were selected almost entirely on the basis of availability. Of the diverse materials tried, two were by far the best, namely Atlas Span 85 and a glyceryl mono-oleate from the Kessler Chemical Co.[12] Proceeding from this observation, a more systematic approach to the search for the best surface-active agent is now possible. Both compounds are long-chain esters of polyhydric alcohols. Starting with similar compounds, the effects of small, systematic alterations in structure - as in molecular weight, degree of esterification, number of functional groups, etc.—should be studied to determine the optimum values for all of these factors.

(4) In order to start this systematic study, a group of chemically related esters were obtained from the Process Chemicals Co., Los Angeles. These differed in the molecular weight of the polyhydric alcohol and in the degree of esterification. The following compounds have been tested:

Prosol 421: mono-esterified dihydric alcohol, M.W. = 400
428: further esterified
431: completely esterified

315: mono-esterified dihydric alcohol, M.W. = 200
305: further esterified
307: completely esterified

The results of the tests, given in Table III, indicate that, except for Prosol 307, these compounds are not as good as Span 85 and glyceryl mono-oleate. The Prosol 307 appears very promising and merits further study. It produces a material of unusual softness, whose apparently inverted impact stabilities may be related to the softness.

(5) Pending the possible discovery of a better agent, Span 85 was adopted as the standard, and was used in most of the other tests summarized in the table. Investigation shows that changes in the amount used have pronounced effects on impact stability. Though it would be desirable to use less than 4% Span, reduction can now be accomplished only at the expense of impact stability.

d. The Coating

(1) The ideal coating material should possess intrinsic explosive power, be stable and relatively dense, and show the special solubility

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[12] Believed to contain contaminating quantities of the di- and tri-oleates and glycerol.

behavior demanded by the process. Not many available materials satisfy all of these requirements; perhaps the most numerous class is that of the condensation nitro polymers. Several of these were investigated, along with a few other materials, and the results are encouraging. However, the hard, brittle nature of this class of polymers suggests that the development of softer nitro polymers may make possible a further improvement.

(2) The results of the tests with various coating materials in various quantities are presented in Table III. Polytrinitroethyl methacrylate (Poly TNEMA) and pentaerythritol acrylate trinitrate polymer (Poly PEATN) are very poor. Monomeric pentaerythritol acrylate trinitrate is considerably better, while the condensation polymers I-A, I-D (the polyurethane of 3,3-dinitro-1,5-pentane diisocyanate and 5,7,9-trinitro-5,7,9-triaza-3,11-dioxa-1,13-tridecanediol) and possibly I-J (the polyurethane of 3,3-dinitro-1,5-pentane diisocyanate and 2-nitro-2-methyl-1,3-propanediol) are the best. On the basis of impact stability, there is little or no choice among the three polymers. Polymer I-A was selected for more extensive study because I-D is more difficult to prepare and I-J has less explosive power.

(3) Perhaps some of the Span 85 can be replaced by another substance to give improved stability. Section 6 d of Table III also shows the results of replacing 0.02 gm of Span 85 by the same amount of various other substances. Some of these were suggested by chemical similarity, others because of possible "softening" effects on the coating. In addition, one experiment was performed using reduced amounts of both Span 85 and the substitute compound, gum rubber. Of these substitute materials, the only one which appears promising enough for further investigation is Emery 955 dimer acid. This may prove to be more effective (as lanolin is) when added initially to the slurry medium rather than with the coating solution.

## C. EXPERIMENTAL

### 1. Coating from Emulsion

a. The emulsion of poly dinitrobutyl acrylate, weighed by difference, was placed in a test tube fitted with a mechanical stirrer. The calculated amounts of Span 85 and water were added and stirring was started while the RDX was being weighed. The stirring was stopped and the RDX was added by means of a funnel. A slurry was made by mixing with a spatula, and the slurry was then stirred at high speed for 5 min. Stirring was stopped and a quantity of methanol, equal in volume to the amount of water used, was added; the stirring was then continued for an additional 2 min. The RDX was collected on a filter, washed with methanol, and dried for a minimum of 6 hr in an evacuated desiccator before testing. The experimental results are summarized in Table IV.

### 2. Coating from Solution

a. Results obtained in studying the effect of the variables in the solution coating process are given in Table III.

## TABLE IV

## PROPERTIES OF RDX DESENSITIZED BY COATING WITH POLY-2,2-DINITROBUTYL ACRYLATE

| Sample No. | DNBA % | Source* DNBA | Span 85 % | RDX gm | I.S.[a] cm/2 kg | I.S.[b] Crushed Mat'l cm/2 kg | I.S.[c] Mat'l Molded at 13880 psi cm/2 kg | I.S. After Accelerated Aging for Following Number of Hours, 65°C, cm/2 kg | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | 24 | 48 | 72 | 120 | 144 | 168 | 216 | 288 | 336 | 456 |
| S-21 | 5 | N-245 | 1.2 | 1.0 | 36 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-22 | 6 | N-245 | 1.2 | 1.0 | 53 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-23 | 7 | N-245 | 1.2 | 1.0 | 66 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-24 | 8 | N-245 | 1.2 | 1.0 | 92 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-25 | 9 | N-245 | 1.2 | 1.0 | >100 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-26 | 10 | N-245 | 1.2 | 1.0 | >100 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-27 | 1 | N-245 | 2.4 | 1.0 | >100 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-39 | 1 | N-245 | 2.4 | 10.0 | >100 | 96 | 94 | — | — | — | — | — | — | — | — | — | — |
| S-28 | 2 | N-245 | 2.4 | 1.0 | >100 | — | 68 | — | — | — | — | — | — | — | — | — | — |
| S-57 | 2 | N-245 | 2.5 | 6.4 | 100 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-58 | 2 | N-245 | 2.5 | 11.7 | 100 | 72 | 87 | 100 | 100 | 100 | 100 | — | 100 | — | — | — | — |
| S-44 | 2 | N-248 | 2.5 | 15 | >100 | >100 | >100 | 71 | — | — | — | 78 | — | >100 | 100 | 100 | 85 |
| S-49 | 2 | N-248 | 2.7 | 8 | 100 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-70 | 2 | N-248 | 2.5 | 20 | 75 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-43 | 3 | N-247 | 2.5 | 50 | >100 | >100 | >100 | 87 | — | — | — | 81 | — | >100 | 90 | 98 | 93 |
| S-45 | 3 | N-248 | 3.1 | 9 | >100 | >100 | 95 | 45 | — | — | — | 80 | — | 97 | 100 | 100 | 100 |
| S-50 | 3 | N-248 | 2.5 | 12 | 71 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-54 | 3 | N-248 | 2.8 | 2 | 76 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-55 | 3 | N-248 | 2.7 | 2 | 75 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-59 | 3 | N-245 | 2.5 | 8.7 | >100 | >100 | >100 | 100 | >100 | >100 | 100 | — | 100 | — | — | — | — |
| S-64 | 3 | N-245 | 2.5 | 47 | >100 | 63 | >100 | — | 40 | — | 47 | — | — | — | — | — | — |
| S-68 | 3 | N-245 | 2.5 | 17 | 100 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-46 | 4 | N-248 | 2.5 | 11 | 98 | — | — | 33 | — | — | — | 80 | 49 | 100 | 93 | 70 | — |
| S-51 | 4 | N-248 | 2.6 | 7 | 40 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-56 | 4 | N-248 | 2.5 | 2.7 | 42 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-60 | 4 | N-245 | 2.5 | 6.9 | >100 | >100 | >100 | >100 | >100 | 100 | 100 | — | 98 | — | — | — | — |
| S-69 | 4 | N-245 | 2.5 | 13 | 98 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-65 | 4 | N-245 | 2.5 | 43 | 75 | 70 | >100 | — | 40 | — | 35 | — | — | — | — | — | — |
| S-47 | 5 | N-248 | 2.5 | 10 | 47 | — | — | 30 | — | — | — | 65 | — | 71 | 95 | 85 | 65 |
| S-52 | 5 | N-248 | 2.6 | 10 | 45 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-61 | 5 | N-245 | 2.5 | 6.6 | 100 | 100 | >100 | >100 | 100 | >100 | >100 | — | 100 | — | — | — | — |
| S-66 | 5 | N-245 | 2.5 | 40 | 79 | 60 | 91 | — | 40 | — | 35 | — | — | — | — | — | — |
| S-48 | 6 | N-248 | 2.6 | 10 | 41 | — | — | 63 | — | — | — | 54 | — | 68 | 95 | 85 | 80 |
| S-53 | 6 | N-248 | 2.5 | 10 | 51 | — | — | — | — | — | — | — | — | — | — | — | — |
| S-62 | 6 | N-245 | 2.5 | 6.2 | >100 | >100 | 100 | >100 | >100 | >100 | >100 | — | 100 | — | — | — | — |
| S-67 | 6 | N-245 | 2.5 | 3.7 | 78 | 63 | >100 | — | >100 | — | 47 | — | — | — | — | — | — |
| S-63 | 10 | N-245 | 2.5 | 5.4 | >100 | 100 | >100 | >100 | >100 | >100 | >100 | — | >100 | — | — | — | — |

I.S.[a] - impact stability of coated RDX

I.S.[b] - impact stability of coated RDX after rolling with a glass rod encased in Tygon tubing

I.S.[c] - impact stability of coated RDX after being molded at 13880 psi in a Carver Laboratory Press

*See table below.

| | DNBA gm | PVA gm | Nonisol 210 gm | MAKP gm | $H_2O$ gm | Temp. °C | Time hr | Analysis | Remarks |
|---|---|---|---|---|---|---|---|---|---|
| N-245 | 10 | 0.1 | 0.5 | 0.02 | 40 | 45 | 92 | 0.10 polymer/gm emulsion | Polymer is soft |
| N-248 | 15 | 0.15 | 0.75 | 0.03 | 60 | 45 | 95 | 0.14 gm emulsion | Polymer is soft |
| N-247 | 12 | 0.36 | 0.9<br>(Aerosol) | 0.12<br>$(NH_4)_2S_2O_8$ | 9 | 45 | 92 | 0.25 gm emulsion | Polymer is hard<br>0.24 gm tert-dodecyl mercaptan added |

b. In order to obtain more information than that provided by the simple impact stabilities, a more extended study was begun on desensitized RDX made by the basic preparation. One portion was subjected to an accelerated aging test at 50°C; the results are given in Table V. Another portion was molded at 11,000 psi pressure and a pellet of cheese-like consistency was obtained. The impact stability of the crumbled pellet was 60 to 65 cm/2 kg.

c. The density of this coated RDX was determined roughly by suspending the powdered material in a series of liquids of different densities. Values of about 1.80 gm/ml were obtained for this powder, and 1.76 gm/ml for the compressed pellet.

TABLE V

EFFECTS OF ACCELERATED AGING AT 50°C

ON BASIC PREPARATION OF RDX COATED FROM SOLUTION

| Time hr | I.S., Not Crushed cm/2 kg | I.S. Crushed cm/2 kg |
|---|---|---|
| 0 | >100 | 85 to 90 |
| 5 | — | 75 to 80 |
| 24 | — | 75 to 80 |
| 150 | 70 | 60 to 65 |

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Report No. 589
SPIA/M3

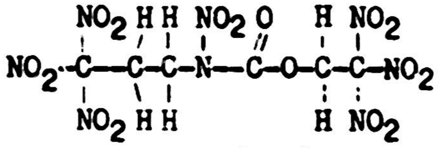
Data Questionnaire on

## 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 Ave., 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:**
**Name** N-nitro-N-3,3,3-trinitropropyl-2,2,2-trinitroethyl carbamate
**Empirical formula** $C_6H_6N_8O_{16}$
**Structure:** (configuration)

**Information submitted by:**
**Activity** Aerojet Engineering Corporation
**Person** M. Frankel, R. Parrette
**Date** 28 February 1952



**Preparation reaction(s):** $(NO_2)_3CCH_2CH_2NCO + HOCH_2C(NO_2)_3 \longrightarrow$

$(NO_2)_3CCH_2CH_2NHCO_2CH_2C(NO_2)_3 \xrightarrow[Ac_2O]{HNO_3} (NO_2)_3CCH_2CH_2N(NO_2)CO_2CH_2C(NO_2)_3$

**1. Quantitative analysis:** (% by weight)

| | Carbon | Hydrogen | Oxygen | Nitrogen |
|---|---|---|---|---|
| Calculated from formula | 16.15 | 1.36 | 57.37 | 25.12 |
| By determination | 16.35 | 1.36 | | 25.34 |

Impurity, determined from melting-point curves = 0.3 mol %

**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 | OSRD 3185 | |
| b. Thermal Stability | OSRD 3401 p.8 | 134.5°C, methyl violet paper |
| c. Vacuum Stability | OSRD 3401 p.10 | |
| d. Temperature of Explosion | OSRD 3401 p.6 | |
| e. Temperature of Ignition | OSRD 3401 p.6 | |
| f. Thermal Stability, 65.5°C | Picatinny Arsenal | Report No. 1401, 1.3 gm sample, KI-starch paper |
| g. | | |
| h. Impact Stability | Bureau of Mines | Bull. No. 346, 2 kg wt, 50% shots |

### RESULTS OF ABOVE TESTS

| | Reference compound (designation-TNT, Tetryl, N.C., etc.) | New Compound test results |
|---|---|---|
| a. | | |
| b. | Nitrocellulose, 30 min, no color. | Colored, 17 min, no change in appearance, 5 hr |
| c. | | |
| d. | | |
| e. | | |
| f. | Nitrocellulose, 10 min, no color. | Colored, 28 min, no change in appearance, 5 hr |
| g. | | |
| h. | RDX 28 cm, PETN 17 cm | 30 cm |

**4. Heat of formation:** ($\Delta H$) ± -192 (indicate sign) Kg. calories at 25°C., 1 atm. pressure

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| | By Experiment | | By Calculation | Method<br>Description or reference. Separate sheet if necessary. |
|---|---|---|---|---|
| 5. Energy of explosion (Q) (at 25°C. $H_2O$ liquid) | | cal/gm | 1492 | Aerojet Report No. 512 |
| 6. Heat of combustion ($H_c$) (at 25°C. $H_2O$ liquid) | 1487 | cal/gm | 1495 | Aerojet Report No. 417A |

7. Specific impulse ($I_{sp}$) calc: ______ lb-sec/lb ______

8. Physical form of compound (viscous liquid, crystalline type, etc.) Fine, white, plate crystals

9. Simple microscope analysis data: ______
(crystal studies)

10. Density (Macro method) 1.805 gm/cm³. (Micro or other method) ______ gm/cm³.
(Explain 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. 6.2 (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) Water, saturated solution, Beckman meter.

15. Hygroscopicity:

| | New Compound | Reference Compound ______ ( designation ) |
|---|---|---|
| Visible change on exposure to ambient air. | | |
| % wt Increase by* (a) or (b) (Strike out method (a) or (b) if not used.) | | (If other than below methods are used, explain on separate sheet.) |

*Method:

(a) The sample (approx. 5-10 g. of whole grain small powder or 1 grain of large powder) is placed in a glass weighing bottle. The weighing bottle (cover removed) is placed in a vacuum drying oven for 5 hrs. @ 55°C. Remove weighing bottle from oven, cover with glass stopper, cool in a desiccator and weigh accurately. This is taken as original dry weight of sample. Then place weighing bottle (cover removed) in a humidor (a 10-in. desiccator is a satisfactory vessel) containing 1 liter of 18.6 ± .5% $H_2SO_4$. (This gives relative humidity 90 ± .25%). Place in an oven maintained at 30 ± 2°C. On the fourth day remove weighing bottle from the humidor, cover with glass stopper, cool in a desiccator and weigh. Then return to humidor for 24 hours, cool and reweigh. Continue daily weighings until constant weight indicates sample has reached equilibrium with 90% R.H. The % weight increase is then reported as hygroscopicity of the sample.

(b) An alternate method is in OSRD 3401 p.3.

16. Volatility: Report as rate of loss in wt % per unit area (for liquid samples measure the surface area; for solids, the sample should be screened between 100-120 mesh U.S. Standard certified sieve) per 4 hrs., after constant rate of loss is obtained during three consecutive 4-hr. periods at (a) 25°C in a vessel through which a stream of dry air is forced, (b) in an oven at 65.5°C or (c) under other test method or conditions. ______

(Description or reference to other test or conditions used. Use separate sheet if necessary.)

| Volatility results on: New Compound | Reference Compound ______ (designation) |
|---|---|
| a. | a. |
| b. | b. |
| c. | c. |

17. Boiling point, or decomposition temperature: ______ °C.
(underline which temperature is reported)

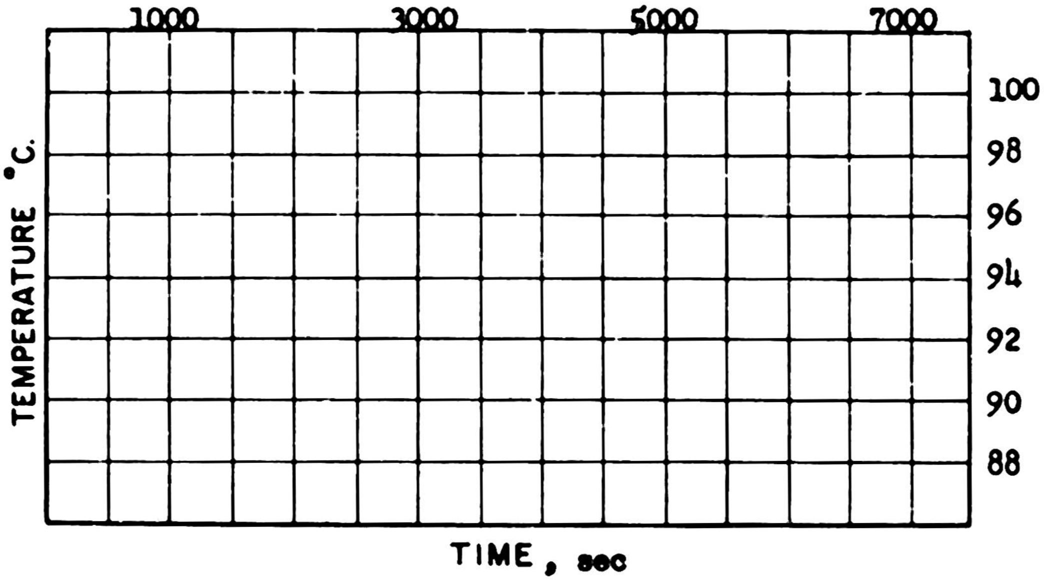
18. Heat of Vaporization: ______ g-cal./gm ______ BTU/lb.

19. Heat of Fusion: ______ g-cal./gm ______ BTU/lb.

20. Melting point 96.0 °C.

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

Apparatus of Smit, W.M.; Dissertation, Amsterdam (1946)



22. **Solubility of new compound:**

< 0.01 g/100 ml $H_2O$ at 25°C. ______ g/100 ml $H_2O$ at ______ °C.

235 g/100 ml Acetone (name material used as solvent) at 25 °C.

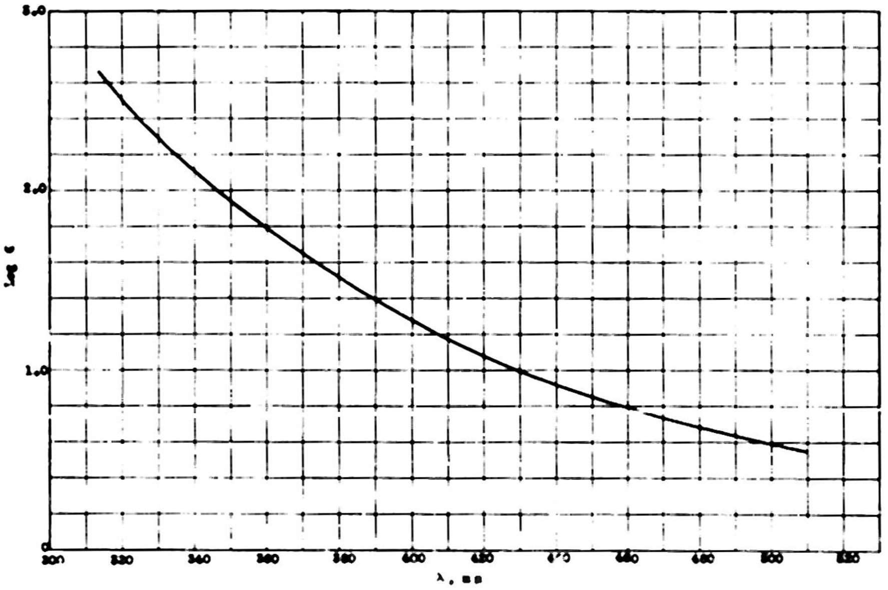
9.5 g/100 ml Toluene (name material used as solvent) at 25 °C.

23. **Viscosity of the new compound and its solutions:**

| | NEW COMPOUND wt. % in solution | SOLVENT NAME | SOLVENT Wt. % in solution | VISCOSITY at Centipoises | TEMP. °C. | METHOD USED reference |
|---|---|---|---|---|---|---|
| a. | 100 | | NONE | | 25 | |
| b. | 100 | | NONE | | | |
| c. | 95 | N.C. (13.15% N.) | 5 | | 25 | |
| d. | | | | | | |
| e. | | | | | | |
| f. | | | | | | |

Ultraviolet Absorption Spectrum of

N-Nitro-N-Trinitropropyl-Trinitroethyl Carbamate, in Ether



Under compatability 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. Sample and NC or rubber is dissolved in a mutual solvent, the solvent evaporated, and the resulting film examined for clarity and homogeneity.

25. Compatability with ethyl cellulose:

26. Compatability with nitrocellulose: Ratio, wt sample / wt NC : :

| | |
|---|---|
| 20/80 | Compatible |
| 50/50 | Compatible |
| 80/20 | Compatible |

27. Compatability with rubber: Ratio, wt sample / wt rubber : :

| | |
|---|---|
| 20/80 | Compatible |
| 50/50 | Compatible |
| 80/20 | Compatible |

28. Compatability 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?
   - b. When was available material first prepared?
   - c. Amount prepared at that time?
   - d. Is large production feasible?
   - e. Plant capacity in existance, 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.)

Oxygen balance +3.6. Gives zero oxygen balance with 4.8% admixture of TNT. Calculated explosive properties are:

| | Compound | Compound + 4.8% TNT |
|---|---|---|
| Lead block | 178 | 187 |
| Ballistic mortar | 142 | 144 |

## N7onr-46208

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