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FLUORINATED DESENSITIZING INGREDIENTS FOR PROPELLANTS

24 May 1980

Final Report

Covering the Period 24 July 1979 to 23 April 1980

By: John M. Guimont

Prepared for:

NAVAL SEA SYSTEMS COMMAND
Department of the Navy
Washington, DC ~~20362~~ 20362

Attn: R. F. Cassel
NAVSEA 62R22

Contract No. N00024-79-C-5653

SRI Project PYU 8742

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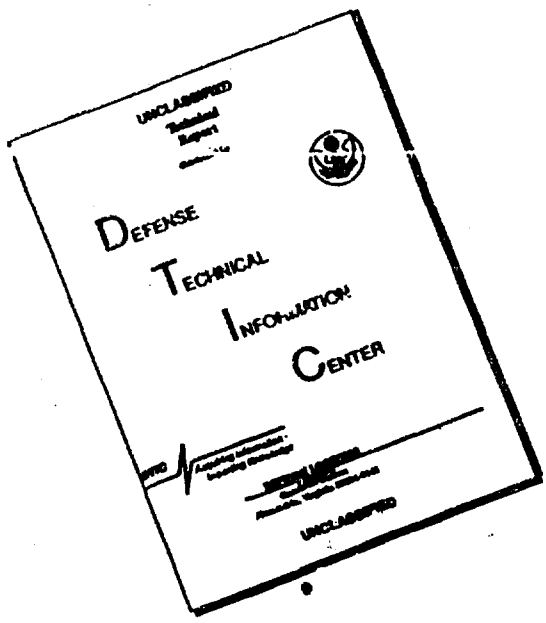

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Approved:

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19. KEY WORDS (Continued)

20 ABSTRACT (Continued)

Compound	Structure
3-Fluoro-1,2-propanediol dinitrate	$FCH_2CH(ONO_2)CH_2ONO_2$
FDNP	
3,3,3-Trifluoro-1,2-propanediol dinitrate	$F_3CCH(ONO_2)CH_2ONO_2$
TFDNP	
4,4,4-Trifluoro-1,2,3-butanetriol Trinitrate	$F_3CCH(ONO_2)CH(ONO_2)CH_2ONO_2$
TFBTTN	

Preliminary examination of the physical properties and sensitivity characteristics of FDNP and TFDNP indicates that these partially fluorinated nitrate esters are potentially useful propellant ingredients. When compared with 1,2-propanediol dinitrate (DNP), which is currently used in Otto Fuel II, FDNP has a lower vapor pressure, higher density, less sensitivity to initiation by impact, and a higher calculated detonation pressure and velocity. TFDNP also compares favorably with DNP, except that its vapor pressure is higher than DNP. A small sample of TFBTTN was prepared, but physical property measurements have not been made. Despite its structural similarity to nitroglycerin, TFBTTN was found to detonate only when subjected to a very strong hammer blow.

The results of the research conducted under this contract demonstrated that fluorinated nitrate esters can be prepared readily using known synthetic routes to fluorinated alcohols and an efficient nitration system developed under an ONR contract. The fluorinated nitrate esters are more energetic and less sensitive to initiation by impact than the nonfluorinated analogs. They also have good physical properties and thermal stability, making them desirable energetic materials for use in liquid propellants or as plasticizers for solid propellants.

CONTENTS

PREFACE

This report is submitted in partial fulfillment of the contractual obligation for Contract No. N00024-79-C-5653 titled "Fluorinated Desensitizing Ingredients for Propellants." The report contains a summary of the work performed during the period 24 July 1979 through 23 April 1980.

The research was performed by the staff of SRI's Chemistry Laboratory of the Physical Sciences Division under the supervision of Donald L. Ross. John M. Guimont was the principal investigator, and laboratory assistance was provided by D.V.H. Son and Steven J. Staats.

The project officer was R. F. Cassel of the Naval Sea Systems Command.

SUMMARY

Under the sponsorship of Naval Sea Systems Command, SRI has been investigating the use of partially fluorinated nitrate esters as desensitizing ingredients for propellants. Three nitrate esters were prepared, and preliminary characterization of the first two shown below was completed:

Compound	Structure
3-Fluoro-1,2-propanediol dinitrate FDNP	$FCH_2CH(ONO_2)CH_2ONO_2$
3,3,3-Trifluoro-1,2-propanediol dinitrate TFDNP	$F_3CCH(ONO_2)CH_2ONO_2$
4,4,4-Trifluoro-1,2,3-butanetriol Trinitrate TFBTTN	$F_3CCH(ONO_2)CH(ONO_2)CH_2ONO_2$

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The results of the research conducted under this contract demonstrated that fluorinated nitrate esters can be prepared readily using known synthetic routes to fluorinated alcohols and an efficient nitration

system developed under an earlier ONR contract. The fluorinated nitrate esters are more energetic and less sensitive to initiation by impact than the nonfluorinated analogs. They also have good physical properties and thermal stability, making them desirable energetic materials for use in liquid propellants or as plasticizers for solid propellants.

INTRODUCTION AND BACKGROUND

Accidental initiation of explosives and propellants is a problem that has plagued the Armed Services for many years. The standard method of alleviating the problem has been to add desensitizing ingredients, which not only reduce the sensitivity of the propellant to initiation, but also frequently reduce the total energy of the system. Among the exceptions is the use of BDNPA and BDNPF for coating RDX for use in certain PBXs. Thus, an ideal desensitizing ingredient would be energetic as well, but an increase in energy is likely to be followed by a corresponding increase in sensitivity, which leads us back to the original problem. However, research at SRI indicates that one solution may be at hand.

Results of research under ONR Contract N00014-76-C-0810 showed that the sensitivity of energetic materials to shock initiation could be reduced by substituting fluorine for some of the hydrogen in the explosive molecule. Briefly, our work has shown that partially fluorinated, high energy materials are generally more thermally stable and less sensitive to initiation by impact and shock than the corresponding materials containing no fluorine. We and other workers have found that introduction of fluorine into the molecule of an energetic material often does not result in a loss in energy. In fact, some materials show an increase in energy as measured by the calculated detonation pressure and velocity (Kamlet equation).

Replacement of hydrogen by fluorine in a given energetic compound may contribute more overall energy to a formulation for two reasons. First, fluorinated materials are generally more dense than their corresponding hydrogen analogs; therefore, a greater weight of the fluorinated compound can be placed in the same or smaller volume. Second, higher thermal stability and lower shock sensitivity of energetic fluorinated materials may effectively desensitize a formulation and thus offset the need for using a nonenergetic desensitizing ingredient.

OBJECTIVE

Using the theory of desensitization as a basis, we proposed an investigation of the synthesis and characterization of FDNP, TFDNP, and TFBTTN as candidate ingredients for Otto Fuel or other liquid propellants. The immediate objective was to determine if these fluorinated nitrate esters could be used to replace a portion of n-butyl sebacate to provide a modified Otto Fuel of higher energy than operational formulations but of equal or lower sensitivity to shock initiation.

SYNTHESIS

3-Fluoro-1,2-propanediol Dinitrate (FDNP)

In our efforts to prepare FDNP, three synthetic routes were investigated: Schemes I, II, and III. Initially we anticipated repeating the work of Olah and Pavlath[1] in which they prepared 3-fluoro-1,2-propanediol in 52% yield, equation (1).

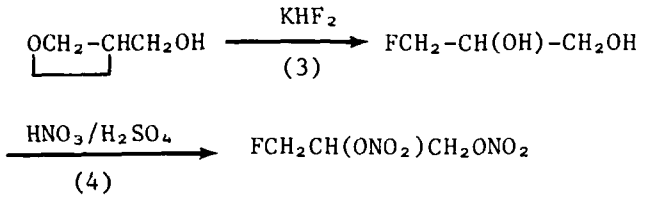
$$ClCH_2CH(OH)CH_2OH \xrightarrow[(1)]{LiF/h\nu} FCH_2CH(OH)CH_2OH$$

$$\xrightarrow[(2)]{HNO_3/H_2SO_4} \underset{FDNP}{FCH_2CH(ONO_2)CH_2ONO_2}$$

Scheme I

Five experiments were conducted in our laboratory following the conditions described by Olah and also by Gryszkiewicz-Trochimowski,[2] but none was successful. Since Olah did not specify the frequency of the UV source, we used three different UV lamps having different wavelength ranges. The NMR spectra and refractive indices of collected products indicated that they were mainly starting material. Because we could not repeat the reported work, we began work on an alternative to reaction (1).

According to Dummell and Kun[3], a more convenient method to prepare 3-fluoro-1,2-propanediol involves using glycidol as starting material, equation (3), Scheme II.

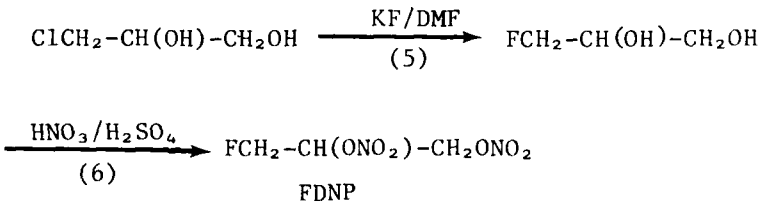


Scheme II

Several attempts to repeat the reported procedure resulted in polymerization of the glycidol; none of the desired product was detectable.

The third synthesis route to FDNP that was investigated, Scheme III, proved to be the least difficult and most successful.

The reaction shown in equation (5) was conducted under reflux for three days. After vacuum evaporation of the DMF, the product was collected by distillation. The nitration, equation (6), was performed using a mixture of 100% HNO_3 and 96% H_2SO_4 at 5-10°C and gave a nearly quantitative yield of FDNP.



Scheme III

3,3,3-Trifluoro-1,2-propanediol Dinitrate (TFDNP)

The synthesis route used for preparing TFDNP, equations (7) through (10), is based on a known procedure.[4] The reactions shown in equations (7) and (8) were conducted exactly as reported, but the hydrolysis step, equation (9), was modified. Treatment of 3-bromo-1,1,1-trifluoro-2-propanol with base produces both the desired diol and 3,3,3-trifluoro-1,2-epoxypropane. After attempting the reaction several times with various bases, we found that treatment with potassium carbonate at 25°C gave a near quantitative yield of the diol.

$$CF_3\text{-}C(O)\text{-}CH_3 \xrightarrow[(7)]{Br_2/H_2SO_4} CF_3\text{-}C(O)\text{-}CH_2Br \xrightarrow[(8)]{NaBH_4} CF_3\text{-}CH(OH)\text{-}CH_2Br \xrightarrow[(9)]{K_2CO_3}$$

$$CF_3\text{-}CH(OH)\text{-}CH_2OH \xrightarrow[(10)]{HNO_3/H_2SO_4} CF_3\text{-}CH(ONO_2)\text{-}CH_2(ONO_2).$$

TFDNP

Nitration of the diol, equation (10), with 100% HNO_3 and 96% H_2SO_4 at 5-10°C produced TFDNP in high yield.

3,3,3-Trifluoro-1,2,3-butanetriol Trinitrate (TFBTTN)

Preparation of TFBTTN is based on nitration of the known triol,[5] and synthesis of triol, equations (13) through (17), was predicated on the commercial availability of ethyl ethoxyacetate. After waiting several months for delivery, we were told by the vendor that it was no longer available and were forced to prepare it as shown in Equations (11) and (12).

$$CH_3CH_2ONa + ClCH_2COOH \xrightarrow[(11)]{} CH_3CH_2OCH_2COOH$$

$$\xrightarrow[(12)]{CH_3CH_2OH} CH_3CH_2OCH_2COOCH_2CH_3$$

$$CH_3CH_2OCH_2COOCH_2CH_3 + CF_3COOCH_2CH_3 \xrightarrow[(13)]{NaOCH_2CH_3}$$

$$CF_3C(O)CH(OCH_2CH_3)COOCH_2CH_3 \xrightarrow[(14)]{[H]} CF_3CH(OH)CH(OCH_2CH_3)COOCH_2CH_3$$

$$\xrightarrow[(15)]{LAH} CF_3CH(OH)CH(OCH_2CH_3)CH_2OH \xrightarrow[(16)]{HBr}$$

$$CF_3CH(OH)CH(OH)CH_2OH \xrightarrow[(17)]{HNO_3/H_2SO_4} CF_3CH(ONO_2)CH(ONO_2)CH_2ONO_2$$

Although the procedure was very time consuming, ethyl ethoxyacetate was prepared following the reported procedure.[6] The reactions shown in equations (13) through (16) were performed exactly as reported[5] and resulted in an overall yield of 20%. Nitration with nitric and sulfuric acids proceeded as anticipated.

CHARACTERIZATION

The subject nitrate esters were characterized by physical property measurements, thermal stability, impact sensitivity, and theoretical detonation properties. All of the data for FDNP and TFDNP are given in Table 1 along with the properties of DNP for comparison. TFBTTN was not characterized because only a small amount could be prepared during the contract period.

Densities were determined with a Fisher-Davidson gravitometer, and vapor pressures were determined by extrapolation after measuring the boiling point at various pressures.

FDNP appears to be a potentially useful propellant ingredient. Compared with DNP, FDNP has a higher density, lower vapor pressure, higher oxygen balance, reduced sensitivity to initiation by impact, and improved detonation characteristics. FDNP should be studied further as a possible Otto Fuel ingredient. TFDNP exhibits all of the desired improvements over DNP with the exception of TFDNP's higher vapor pressure. The vapor pressure of TFDNP precludes its use as an Otto Fuel ingredient, but the large reduction in sensitivity to impact may make it a desirable ingredient for an application where vapor pressure is not a significant problem.

The differential scanning calorimetry (DSC) data that we obtained on DNP, FDNP, and TFDNP and Otto Fuel mixtures prepared from each have been omitted from the table of properties because we feel that the data are misleading. The DSC data, which were presented in earlier reports, showed that DNP was stable up to 270°C and that Otto Fuel II was stable to 235°C while FDNP and TFDNP decomposed at 160-180°C.[7] In addition, when samples of Otto Fuel prepared from each nitrate were heated in melting-point capillaries, all three decomposed with gassing at 160-180°C. There are several alternative explanations for the anomalous behavior of DNP, but the cause of the problem has not been determined. Additional

Table 1

PROPERTIES OF DNP, FDNP, AND TFDNP

	DNP	FDNP	TFDNP
Structure	$CH_3\underset{ONO_2}{CH} - \underset{ONO_2}{CH_2}$	$FCH_2\underset{ONO_2}{CH} - \underset{ONO_2}{CH_2}$	$F_3C\underset{ONO_2}{CH} - \underset{ONO_2}{CH_2}$
Molecular formula	$C_3H_6N_2O_6$	$C_3H_5N_2O_6F$	$C_3H_3N_2O_6F_3$
Formula weight	166.1	184.1	220.07
Density (g/cm^3)	1.374	1.555	1.614
Vapor pressure (mm/25°C)	0.065	0.011	0.32
Oxygen balance (wt % to CO/CO_2)	0/-29	8.7/-17	22/0
Detonation pressure[a] (kbar)	201	238	246
Detonation valocity[a] (m/sec)	7350	7660	7700
Impact[b] (kg-cm)	4-6	7-8	10-15

[a]Calculated using the Kamlet equation assuming that fluorine forms HF during detonation.

[b]Determined with a Technoproducts drop-weight tester.

characterization of FDNP should include a careful study of its thermal behavior.

Although TFBTTN has not been fully characterized, we recommend further investigation. We found that TFBTTN is not very sensitive to impact, at least qualitatively in that it requires a very strong hammer blow to cause detonation. This is quite surprising in view of its structural similarity to nitroglycerin. TFBTTN is a highly oxidized material with a possibility of being a good plasticizer for double-base systems. If our qualitative observation of its sensitivity is verified, a more complete study including synthesis improvement may be recommended.

EXPERIMENTAL DETAILS

The experimental procedures reported below are new synthesis reactions that are not reported in the literature or are significantly modified versions of known reactions. All other experiments reported in the text are a duplication of published procedures.

3-Fluoro-1,2-propanediol

A mixture of 62.8 g (0.57 mol) of 3-chloro-1,2-propanediol, 46.4 g (0.80 ml) potassium fluoride, and 80 ml of dimethyl formamide was heated to reflux for 72 hours. The reaction mixture was then cooled to ambient temperature, filtered, and distilled under reduced pressure. The desired product (40 g, 75% yield) was collected at 65-75°C/0.01 torr.

3-Fluoro-1,2-propanediol Dinitrate

In this procedure, 8.8 g (0.14 mol) of 100% nitric acid was added dropwise to 5.0 g (0.05 mol) of 3-fluoro-1,2-propanediol at 5-10°C with ice/water cooling. When the addition was complete, 29.3 g of 96% sulfuric acid was added dropwise at 5-10°C. The reaction mixture was stirred for 30 min at 5-10°C and then poured onto 80 g of ice. The aqueous solution was extracted 4 times with 25 ml portions of methylene chloride. The methylene chloride solution was dried over magnesium sulfate, filtered, and evaporated, leaving 7 g of brown liquid that was distilled at 48-49°C/0.1 torr to give 6.0 g FDNP (61% yield).

3,3,3-Trifluoro-1,2-propanediol

A mixture of 42.3 g (0.22 mol) of 1-bromo-3,3,3-trifluoro-2-propanol, 40.7 g (0.30 mol) potassium carbonate, and 230 ml water was stirred at 25°C for 1.5 hr, then at 40-50°C for 2 hr. The reaction mixture was extracted with ether, which was then dried over magnesium sulfate, filtered, and evaporated, leaving 13.3 g of 3,3,3-trifluoro-1,2-propanediol. (47% yield). The material thus obtained was used in the subsequent nitration without further purification.

3,3,3-Trifluoro-1,2-propanediol Dinitrate

To 13.3 g (0.1 mol) of 3,3,3-trifluoro-1,2-propanediol, was added dropwise 27 g of 100% nitric acid at 5-10°C over a period of 10 min. When the addition was complete, 85 g of 96% sulfuric acid was added dropwise at 5-10°C. The reaction mixture was stirred at 5-10°C for 1 hr and then poured onto 160 g of ice. The aqueous solution was extracted with methylene chloride, which was then dried over magnesium sulfate and evaporated, leaving 14.3 g crude product (65% yield). The desired product was distilled at 65°C/20 torr.

4,4,4-Trifluoro-1,2,3-butanetriol Trinitrate

In this procedure, 1 g (6.3 mmol) of 4,4,4-trifluoro-1,2,3-butanetriol was nitrated with 6 g (95 mmols) of nitric acid and 20 g of 96% sulfuric acid, and the reaction was worked up in the same manner as described for the propanediols. (Yield: 0.87 g, 50%).

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