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(TITLE UNCLASSIFIED)
**DEVELOPMENT OF PROPELLANTS
CONTAINING AN ENERGETIC OXIDIZER**

T. Rudy
United Technology Center

TECHNICAL REPORT

~~AFRPL-TR-67-2~~

DECEMBER 1966

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**AIR FORCE ROCKET PROPULSION LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND,
UNITED STATES AIR FORCE
EDWARDS, CALIFORNIA**



UTC 2139-QTR3

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DIVISION OF UNITED AIRCRAFT CORPORATION

20 January 1967

ADP-134-67-F

Air Force Flight Test Center
Directorate of Procurement
Edwards Air Force Base,
California 93523

Attention: FTMKR-4

Subject: AFRPL-TR-67-2 (UTC 2139-QTR3)

Reference: Contract AF 04(611)-10786,
DD Form 1423, Line Item No. 5

Gentlemen:

United Technology Center submits one (1) copy of the subject report in accordance with the referenced contract.

This report covers the period 1 September 1966 through 30 November 1966.

Very truly yours,

UNITED TECHNOLOGY CENTER
A Division of United Aircraft Corporation



A. D. Parker, Manager
Contract Management

ADP:btg

Enclosure

cc: AFRPL, Edwards, Calif. 93523
Attn: RPMC (w/3 encl.)
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**DEVELOPMENT OF PROPELLANTS
CONTAINING AN ENERGETIC OXIDIZER**

T. Rudy

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UNCLASSIFIED FOREWORD

(U) This Third Quarterly Technical Progress Report under Contract No. AF 04(611)-10786, dated December 1966, covers work performed during the period 1 September 1966 through 30 November 1966, by United Technology Center, Sunnyvale, California. The contract was initiated under Air Force Rocket Propulsion Laboratory, Research and Technology Division, Project No. 3059. For internal purposes, this report is designated UTC 2139-QTR3.

(U) The Air Force Program Monitor is Karl W. Joffs, 1/Lt., USAF/RPMCP of the Research and Technology Division, Air Force Rocket Propulsion Laboratory, Air Force Systems Command, United States Air Force, Edwards, California, 93523.

(U) The Program Manager for United Technology Center is E. J. Walden. Principal contributors to the work reported herein are:

J. W. Allan
R. Alvarado
L. S. Bain

T. W. Nakagawa
J. T. Robinson
M. E. Steinle

(U) This report contains classified information extracted from the following documents: Final Technical Progress Report, Contract No. AF 04(611)-9894, August 1965; and Quarterly Progress Report No. 2, Contract No. AF 04(611)-11199, April 1966. Both documents are classified CONFIDENTIAL, Downgrading Group 4.

(U) Subject matter contained herein has been placed under Secrecy Order with Permit A by the United States Patent Office. Recipients of this document must take all reasonable safeguards to prevent the unauthorized disclosure of the subject matter. Failure to properly safeguard this information may be punishable by a fine of not more than \$10,000, or imprisonment for not more than 2 years, or both as provided by 35USC186.

(U) Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

Approving authority is
Lt. K. W. Joffs

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CONFIDENTIAL ABSTRACT

(C) United Technology Center is conducting a 24-month program to produce the first practical propellants exploiting the high energy and favorable density of nitronium perchlorate. Since Reta-coated nitronium perchlorate is not available, emphasis remains on definition and improvement of the stability of propellants containing uncoated, particulate nitronium perchlorate. The effectiveness of reduced storage temperature in extending propellant life has been further demonstrated. Differential thermal analysis provides a rough, but useful, indication of the tendency of deteriorated propellants to burn explosively. Increased concentrations of the binder crosslinking agent significantly improve propellant stability. The isoparaffinic plasticizer used previously impairs propellant stability. Improved plasticizers have been demonstrated. Propellant stability has thereby been increased dramatically. Oxidizer particle size has little influence on the stability of improved formulations.

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CONTENTS

<u>Section</u>		<u>Page</u>
I	INTRODUCTION	1
II	EFFECT ON STORAGE TEMPERATURE ON STABILITY OF PROPELLANT A	5
	1. Thermal Stability	5
	2. Mechanical Properties and Impact Sensitivity	6
	3. Deflagration	6
III	EFFECT OF PROPELLANT COMPOSITION ON STABILITY	15
	1. Increased Crosslinker Concentration	15
	2. Plasticizers	16
	3. Oxidizer Particle Size	17
IV	CONCLUSIONS AND FUTURE ACTIVITY	19

TABLES

<u>Table</u>		<u>Page</u>
I	(U) Propellant Formulations	3
II	(U) Storage Stability of Propellants	7
III	(U) Combustion of Propellant A	13

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ABBREVIATIONS AND SYMBOLS

DTA	differential thermal analysis
HMN	2,2,4,4,6,8,8-heptamethylnonane
MAPO	tris [1-(Z-methyl)aziridinyl] phosphine oxide
MRPX	a light, isoparaffinic oil obtained by special arrangement from Shell Oil Company
NP	nitronium perchlorate
NTEB	nitrilotriethyl- β -ethyleniminobutyrate, available from American Cyanamid Company
Reta	a poly (chlorinated xylylene) coating developed by Union Carbide Corporation
TMPD	2,6,10,14-tetramethylpentadecane
UTREZ	α,ω -dicarboxylated polyisobutylenc

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SECTION I

INTRODUCTION

(C) Under Contract No. AF 04(611)-9894, United Technology Center (UTC) conducted a 12-month program to develop a curable binder containing a saturated hydrocarbon, difunctional polymer, and to establish the compatibility of this binder with NP. A further objective of the program was ballistic characterization of the binder/NP formulation in small motors.

(C) These objectives were achieved by the development of a system based on UTREZ prepolymer, MRPX plasticizer, and NTEB crosslinker, a trifunctional aziridinyl compound. Propellants containing both coated and uncoated NP of 30-40 mesh particle size were mixed, cast, and cured at room temperature. Those containing uncoated oxidizer were stable for only a few days at room temperature in a dry environment. Propellants containing Reta-coated oxidizer were found to be stable for periods up to 6 months under the same conditions. General characteristics of the propellants were insensitivity to impact and friction, sensitivity to moisture, limited thermal stability, and reasonable mechanical properties.

(C) All of the propellants were ballistically characterized in micromotor firings, and a formulation containing Reta-coated NP was tested in nominal 1-lb motor firings. Efficient oxidizer utilization, high burning rates, and moderately high pressure exponents were indicated by these tests. The most significant relationships were believed to be those derived from the 1-lb motors:

$$r_b = 0.68 (P_c / 1,000)^{0.49}$$

$$K_n = 320 (P_c / 1,000)^{0.54}$$

(U) Under the present contract, experimental work is directed toward improvement of the basic propellant system with respect to stability, ballistic performance, and mechanical properties. Improved stability is by far the most important objective and is being emphasized accordingly.

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(C) Initial plans called for extensive work with Reta-coated NP. In February 1966, Union Carbide Corporation was in the process of preparing oxidizer for this program when 6 lb of coated and partly coated material were destroyed in an explosion. This unprecedented event is still under investigation, and until safety of both the coating operation and product is established, Reta-coated oxidizer will not be available for use under the present contract. Therefore, recent studies at UTC have been concerned with defining and improving the stability of propellants containing uncoated NP.

(U) During the quarter preceding the present report period, the feasibility of extending propellant life by storage at reduced temperatures was explored. It was found that a formulation (propellant A, UTX-9118),* which deteriorates to a useless and dangerous state in about 10 days at room temperature, is stable for several months at 35° F. Unexpectedly, a still lower storage temperature, 10° F, proved to be less favorable than 35° F. Another result of this study was the indication that increased concentrations of the binder crosslinking agent, NTEB, might improve storage stability of the propellant.

(U) During the present report period, studies of propellant A were essentially completed, and formulation variables were explored in an effort to improve storage stability.

* All propellant formulations discussed in this report are listed in table I.

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TABLE I
(U) PROPELLANT FORMULATIONS

Formulation UTX-	NP		Al		Binder			Plasticizer	
	% wt	Mesh Size	40 μ , % wt	% wt	UTREZ Equivalent wt	NTEB Equivalent wt	Type	% wt	
9118	63	30-40	2	35	1.0	1.2	MRPX	15	
9143	65	30-40	---	35	1.0	1.5	---	---	
9144	65	30-40	---	35	1.0	2.0	---	---	
9145	65	30-40	---	35	1.0	2.5	---	---	
9146	65	30-40	---	35	1.0	1.2	---	---	
9147	63	8-20	2	35	1.0	1.2	MRPX	15	
9163	63	30-40	2	35	1.0	2.0	---	---	
9166	63	8-20	2	35	1.0	2.0	---	---	
(109-1)	65	8-20	---	35	1.0	2.0	---	---	
(109-2)	65	30-40	---	35	1.0	2.0	---	---	
(109-3)	63	8-20	2	35	1.0	2.0	---	---	
(109-4)	63	30-40	2	35	1.0	2.0	---	---	
(109-5)	65	8-20	---	35	1.0	2.5	---	---	
(109-6)	65	30-40	---	35	1.0	2.5	---	---	
(109-7)	63	8-20	2	35	1.0	2.5	---	---	
(109-8)	63	30-40	2	35	1.0	2.5	---	---	
(I-A)	60	30-40	---	40	1.0	2.0	MAPO	5	
(I-B)	60	8-20	---	40	1.0	2.0	MAPO	5	
(I-C)	60	8-20	---	40	1.0	2.0	MAPO	5	
		Crushed							
(II-A)	60	30-40	---	40	1.0	2.5	MAPO	5	
(II-B)	60	8-20	---	40	1.0	2.5	MAPO	5	
(II-C)	60	8-20	---	40	1.0	2.5	MAPO	5	
		Crushed							

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SECTION II

EFFECT ON STORAGE TEMPERATURE ON STABILITY OF PROPELLANT A

(U) In the last quarterly report the effect of storage temperature on thermal stability of propellant A was discussed in some detail. Thermal stability was judged primarily on the basis of differential thermal analysis (DTA) and, to a lesser extent, on the basis of combustion characteristics and mechanical properties of the stored propellant. Continuation of the studies of propellant A during the present quarter included additional evaluations of mechanical properties and deflagration characteristics. To correlate these properties with thermal stability, control samples of stored propellants were subjected to periodic DTA.

1. THERMAL STABILITY

(U) Results of DTA are shown in table II. Samples A3T and A4T are controls for the tensile specimens, while samples A1D, A2D, A3D, and A4D are controls for deflagration specimens. Conditions of storage were essentially the same as those reported previously. That is, moisture content of the environment was maintained below 5 ppm of water vapor and refrigerated samples were maintained at 60°, 35°, or 10° F. Unrefrigerated samples were subjected to unavoidable variations in the ambient temperature of the dry box. This temperature normally averaged 70° to 75° F, but on occasion it fell below 65° F. Cases in which low ambient temperature is believed to influence test results are noted in table II.

(U) Results of DTA of the tensile and deflagration control samples are generally consistent with those of earlier studies in that the order of increasing stability as a function of storage temperature is ambient, 60°, 10°, and 35° F. However, overall stability is greater than that reported for earlier studies with propellant A. This is attributed to lower average ambient temperature during the cure period immediately after mixing.

(U) Another perturbing factor during storage of many of the samples shown in table II was the accidental loss of moisture discipline during a 5-day period. During this period, moisture content of the dry box increased from a level below 5 to 90 ppm before corrective action was effective. Fortunately, the exposure to moisture does not appear to have invalidated any important storage program. As a matter of fact, additional useful understanding has resulted. The latter is discussed in section III.

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2. MECHANICAL PROPERTIES AND IMPACT SENSITIVITY

(C) The mechanical properties of microtensile specimens of propellant A were determined after storage for 7 days at ambient temperature followed by 17 days at 35° F (cf sample A3T). The results, 21.8% at maximum stress (10.2 psi), compare unfavorably with properties of a comparable propellant formulated with Reta-coated oxidizer (43% at 36 psi). Impact sensitivity of propellant A after curing 7 days at ambient temperature was found to be 31 kg cm (2 kg wt). This value is consistent with previous results.

3. DEFLAGRATION

(C) While poor storage stability and low ignition temperature of NP propellants are well recognized, there is limited appreciation of their potentially dangerous combustion characteristics. Many attempted motor firings of NP propellants have resulted in what is euphemistically termed "over-pressurization." When similar explosions occur without benefit of a confining motor case, a different term, such as explosive combustion, seems appropriate. This phenomenon has been observed many times during UTC's studies of NP propellants and is not yet understood in detail. However, certain general factors are known which appear equally applicable to the results reported by other investigators.

(C) In most cases the temperature of the propellant under test is usually only a little (50° to 80° C) below the ignition temperature. In addition, deterioration leading to gas formation, hence porosity, is in progress. Resultant debonding of binder from the oxidizer may provide paths for extremely rapid propagation of the ignition front. Finally, it appears likely that the interaction of NP with organic substrates would produce explosive species such as organic nitrates and perchlorates as well as free perchloric acid.

(C) It appears significant that in studies at UTC, explosive combustion of unconfined propellant has not occurred with freshly prepared formulations or with any propellant, however deteriorated, in which all of the NP is Reta-coated.

(U) One-g, cylindrical samples of propellant A were burned without confinement after storage in the dry box at ambient temperature and at 60°F. The results of these tests are shown in table III. Apparently the occurrence of explosive combustion cannot be predicted accurately on the basis of age or DTA of the sample. However, DTA can serve as a rough guide. In the case of propellant A, explosive combustion can be expected after the exotherm inception temperature under DTA has dropped to ca 55° C. Frequently, this temperature begins to increase before explosive combustion is observed.

(U) STORAGE

<u>Sample- UTX</u>	<u>A3T 9118-5</u>	<u>A4T 9118-5</u>	<u>A1D 9118-6</u>	<u>A2D 9118-6</u>	
Storage history- days/temperature	7/ambient* -/-35° F	7/ambient* -10° F	-/ambient* ---	10/ambient* -/-60° F	10/ -3!
Age during moisture exposure, days	93 to 97	93 to 97	---	---	
Thermal stability by DTA					
Age, days-	16-59/78	16-57/77	6-68/82	17-56/71	20.
exotherm, ° C/	20-61/75	20-55/76	8-64/77	20-60/76	29.
ignition, ° C	23-58/78	23-58/75	11-59/76	24-62/75	34.
(X = explosion)	29-63/77	29-57/77	13-52/74X	27-54/74	41.
	36-59/79	36-56/75	15-62/73	29-56/74	48.
	41-58/76	41-57/78	17-48/72	31-58/74	55.
	44-60/79	44-51/77	21-58/76X	34-60/72	62.
	49-60/76	49-58/75	22-55/79	38-60/73	70.
	51-64/75	51-60/75	24-62/76	41-57/72	72.
	56-61/72	56-60/76	---	45-56/75	77.
	58-57/76	58-57/73	---	48-60/78	79.
	60-60/72	60-54/72	---	---	84.
	78-55/79	78-60/78	---	---	
	85-54/72	85-57/74	---	---	
	92-59/76	92-57/74	---	---	
	99-56/72	99-55/71	---	---	
	106-57/74	106-54/72X	---	---	
	113-56/70	113-56/70	---	---	
	143-60/73	143-60/74	---	---	

* Ambient temperature was lower than the normal level of 70° to 75° F.

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TABLE II

(U) STORAGE STABILITY OF PROPELLANTS

<u>A2D</u> <u>9118-6</u>	<u>A3D</u> <u>9118-6</u>	<u>A4D</u> <u>9118-6</u>	<u>L60</u> <u>9146-1</u>	<u>L35</u> <u>9146-1</u>	<u>L10</u> <u>9146-1</u>	<u>H60</u> <u>9143-1</u>	<u>H35</u> <u>9143-1</u>	<u>H10</u> <u>9143-1</u>
0/ambient* /60° F	10/ambient* -35° F	10/ambient* -/10° F	-/60° F ---	26/35° F -/ambient	27/10° F -/ambient	-/60° F ---	39/35° F -/ambient	39/10° F -/ambient
---	---	---	50 to 54	50 to 54	50 to 54	62 to 66	62 to 66	62 to 66
7-56/71	20-62/76	17-57/72	4-69/81	4-81/92	4-79/87	4-77/86	6-81/87	6-75/88
0-60/76	29-60/76	24-60/74	8-72/81	8-78/81	8-78/88	11-76/88	11-79/89	11-79/91
4-62/75	34-66/78	27-55/74	12-76/87	12-72/81	12-72/83	14/75/88	14-74/88	14-78/89
7-54/74	41-60/76	31-57/72	15-72/83	15-68/78	15-64/72	17-76/86	17-74/88	17-75/87
9-56/74	48-62/76	38-62/76	18-60/77	18-68/80	18-67/78	21-66/82	21-74/85	21-78/89
1-58/74	55-60/76	45-55/70	22-71/83	22-71/86	27-73/82	24-70/82	24-76/85	24-76/85
4-60/72	62-52/72	52-59/72	33-65/80	26-76/88	29-66/80	28-67/78	28-66/80	28-73/84
8-60/73	70-59/76	70-56/72	35-66/78	33-69/82	32-73/86	31-68/82	31-76/86	31-71/83
1-57/72	72-60/74	72-59/72	40-66/80	35-69/81	36-74/82	35-64/81	35-68/84	40-69/82
5-56/75	77-60/74	77-58/74	42-64/78	40-64/78	39-66/80	46/64/81	39-74/83	42-66/78
8-60/78	79-62/76	79-58/72	47-70/82	42-66/78	43-64/78	48-66/82	46-66/81	45-73/84
---	84-62/75	84-59/73	49-66/78	47-66/76	46-66/81	53-71/84	48-67/84	49-74/84
---	---	---	54-62/80	49-62/74	50-60/75	55-66/80	53-62/77	52-66/84
---	---	---	56-67/80	54-60/77X	53-66/78	60-64/83	53-62/78	56-64/88
---	---	---	61-60/76	56-62/74X	---	62-62/78	60-64/79	59-66/80
---	---	---	63-62/76	---	---	67-64/80	62-60/74	63-62/74X
---	---	---	68-66/78	---	---	69-66-80	67-62/80X	66-66/81
---	---	---	70-61/78	---	---	74-62/80	69-67/84	---
---	---	---	75-58/78X	---	---	76-62/76	---	---
---	---	---	82-58/76	---	---	81-62/78	---	---
---	---	---	84-60/76X	---	---	83-64/74	---	---
---	---	---	89-62/74X	---	---	88-60/78	---	---
---	---	---	---	---	---	95-60/76	---	---
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---	---	---	---	---	---	---	---	---

° F.

STORAGE STABILITY

Sample UTX-	J60 9144-1	J35 9144-1	J10 9144-1	K60 9145-1	K35 9145
Storage history- days/temperature	-/60° F ---	39/35° F -/ambient	40/10° F -/ambient	-/60° F ---	39/35° F -/ambient
Age during moisture, exposure days	62 to 66	62 to 66	62 to 66	62 to 66	62 to 66
Thermal stability by DTA					
Age, days-	4-81/94	6-79/92	6-78/88	4-77/88	6-86/88
exotherm, ° C	11-81/93	11-82/94	11-82/93	11-87/97	11-88/97
Ignition, ° C	14-76/88	14-78/93	14-78/90	14-80/94	14-87/94
(X = explosion)	17-82/94	17-83/95	17-82/92	16-80/94	18-85/94
	21-78/91	21-80/94	21-80/92	20-85/96	20-84/96
	24-74/84	24-78/90	24-77/88	25-84/92	25-83/92
	26-76/80	28-66/79	28-78/87	27-73/86	27-80/86
	31-68/82	31-71/83	31-70/83	29-74/86	29-80/86
	35-68/85	35-73/93	40-74/84	32-78/90	32-81/90
	46-67/82	39-78/89	42-67/83	34-74/86	39-71/86
	48-70/86	46-69/83	45-78/92	46-71/85	46-71/85
	53-66/80	48-66/82	49-74/86	48-72/89	48-80/89
	55-72/85	53-66/81	52-66/84	53-68/81	53-71/81
	60-64/80	55-72/85	56-67/82	55-66/86	55-71/86
	62-68/82	60-67/83	59-66/82	60-69/84	60-71/84
	67-66/83	62-67/82	63-68/80	62-69/84	62-69/84
	69-67/80	67-60/76X	66-62/79	67-67/82	67-67/82
	74-64/78	69-64/78X	70-62/88	69-68/83	69-68/83
	76-66/82	74-66/82	73-66/78	74-66/80	74-66/80
	81-66/80	76-60/76X	77-67/81	76-62/80	76-62/80
	83-64/78	81-64/78	80-65/78X	81-66/78	81-66/78
	88-62/80	---	---	83-67/78	83-71/78
	95-62/80	---	---	88-64/84	88-64/84
	97-60/80	---	---	95-60/80	95-60/80
	102-68/84	---	---	97-66/82X	97-66/82X
	104-66/80	---	---	102-67/81	102-67/81
	109-64/79	---	---	104-68/84	104-68/84
	111-66/79	---	---	109-68/83	109-68/83
				111-68/84	111-68/84

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TABLE II

E STABILITY OF PROPELLANTS (Continued)

	K35 9145-1	K10 9145-1	M 9147-1	N 9163-1	O 9166-1	109-1 ---	109-2 ---	109-3 ---
F	39/35° F -/ambient	40/10° F -/ambient	-/ambient ---	-/ambient ---	-/ambient ---	-/ambient ---	-/ambient ---	-/ambient ---
6	62 to 66	62 to 66	---	7 to 11	7 to 11	---	---	---
/88	6-86/95	6-78/92	6-72/84	3-87/98	3-76/88	1-76/86	1-83/97	1-74/86
/97	11-88/98	11-83/93	8-70/84	5-78/92	5-74/87	5-73/92	5-74/87	5-80/90
/94	14-87/96	14-72/85	10-62/78	7-73/86	7-74/87	7-72/84	7-75/90	7-76/88
/94	18-85/96	18-80/94	13-66/82	10-66/78	10-66/80	9-74/90	9-69/84	9-74/90
/96	20-84/96	20-74/84	15-62/79	12-64/86	12-66/86	12-74/88	12-74/87	12-76/90
/92	25-83/95	25-80/90	17-55/74	14-64/87	14-76/90	14-72/90	14-74/89	14-71/86
/86	27-80/91	27-70/80	21-60/76	17-62/79	17-62/79	19-70/84	19-71/84	19-72/84
/86	29-80/91	29-77/89	22-56/74X	19-68/83	19-66/80X	21-66/84	21-69/87	21-71/86
/90	32-82/94	32-75/89	24-60/78	21-69/84	21-64/80	23-62/81	23-62/82	23-64/85
/86	39-78/90	34-77/89	---	24-66/79	24-68/82	26-69/87	26-67/83	26-66/84
/85	46-74/91	40-72/84	---	26-72/82	26-71/84	28-71/87	28-66/83	28-69/85
/89	48-80/94	42-72/86	---	28-73/84	28-69/82	---	---	30-66/84
/81	53-72/86	45-74/85	---	31-64/80	31-64/78	---	---	33-70/86
/86	55-70/84	49-80/94	---	---	---	---	---	35-69/86
/84	60-74/88	52-70/87	---	---	---	---	---	37-68/84
/84	62-69/80	56-68/82	---	---	---	---	---	---
/82	67-64/82	59-69/84	---	---	---	---	---	---
/83	69-64/80	63-70/84	---	---	---	---	---	---
/80	74-62/81	66-66/78	---	---	---	---	---	---
/80	76-66/81	70-62/88	---	---	---	---	---	---
/78	81-66/82	73-62/80	---	---	---	---	---	---
/78	83-75/84	77-64/80	---	---	---	---	---	---
/84	---	80-67/84	---	---	---	---	---	---
/80	---	---	---	---	---	---	---	---
/82X	---	---	---	---	---	---	---	---
/81	---	---	---	---	---	---	---	---
/84	---	---	---	---	---	---	---	---
/83	---	---	---	---	---	---	---	---
/84	---	---	---	---	---	---	---	---

(U) STORAGE STABILITY

Sample <u>UTX-</u>	<u>109-4</u> ---	<u>109-5</u> ---	<u>109-6</u> ---	<u>109-7</u> ---
Storage history- days/temperature	-/ambient	-/ambient	-/ambient	-/ambient
Age during moisture, exposure, days	---	---	---	---
Thermal stability by DTA				
Age, days- exotherm, ° C/ ignition, ° C (X = explosion)	1-80/94	1-80/92	1-88/100	1-70/80
	5-82/92	5-74/88	5-76/91	5-82/96
	7-76/88	7-76/89	7-76/88	7-78/88
	9-72/88	9-76/89	9-76/92	9-78/97
	12-73/86	12-74/88	12-76/88	12-76/88
	14-74/90	14-76/92	14-74/92	14-73/91
	19-66/80	19-72/86	19-71/86	19-73/86
	21-67/82	21-67/84	---	21-71/88
	23-64/84	23-70/86	23-66/81	23-68/83
	26-64/82	26-68/86	26-70/86	26-72/87
	28-66/82	28-69/83	28-73/86	28-74/88
	30-66/82	---	---	30-72/86
	33-69/83	---	---	33-73/89
	35-63/79	---	---	35-72/85
	37-66/82	---	---	37-66/80

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TABLE II

GE STABILITY OF PROPELLANTS (Continued)

<u>109-7</u>	<u>109-8</u>	<u>I-A</u>	<u>I-B</u>	<u>I-C</u>	<u>II-A</u>	<u>II-B</u>	<u>II-C</u>
---	---	---	---	---	---	---	---
-/ambient	-/ambient	-/75° F	-/75° F	-/75° F	-/75° F	-/75° F	-/75° F
---	---	---	---	---	---	---	---
1-70/80	1-84/98	1-85/93	1-87/96	1-86/94	1-95/100	1-86/96	1-89/100
5-82/96	5-74/85	3-83/96	3-81/93	3-79/91	3-83/90	3-95/102	3-83/91
7-78/88	7-81/95	7-72/87	7-72/87	7-71-86	7-88/95	7-77/90	7-73/90
9-78/97	9-80/94	13-67/84	13-75/87	13-76/84	13-76/91	13-78/88	13-70/84
12-76/88	12-76/90	21-72/84	21-74/83	21-72/82	21-69/83	21-68/85	21-70/79
14-73/91	14-73/90	---					
19-73/86	19-71/84	---					
21-71/88	21-69/84	---					
23-68/83	23-69/82	---					
26-72/87	26-66/83	---					
28-74/88	28-70/84	---					
30-72/86	---	---					
33-73/89	---	---					
35-72/85	---	---					
37-66/80	---	---					

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TABLE III
(U) COMBUSTION OF PROPELLANT A

<u>Sample</u>	<u>Storage History</u> <u>Days - Temperature</u>	<u>Combustion Tests</u> <u>Number -- Result</u>	<u>DTA</u> <u>Exotherm/Ignition</u> <u>° C</u>
A1D	13 - ambient	1 - normal	52/74 exploded
A1D	17 - ambient	[1 - normal 2 - exploded]	48/72
A1D	20 - ambient	3 - normal	58/76 exploded
A1D	23 - ambient	3 - normal	62/76
A2D	[10 - ambient 14 - 60° F]	1 - normal	62/75
A2D	[10 - ambient 24 - 60° F]	3 - normal	60/72
A2D	[10 - ambient 31 - 60° F]	2 - normal	57/72
A2D	[10 - ambient 35 - 60° F]	2 - normal	56/75
A2D	[10 - ambient 38 - 60° F]	2 - exploded	60/78

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SECTION III

EFFECT OF PROPELLANT COMPOSITION ON STABILITY

(U) In the last quarterly report, evidence was presented suggesting that increased concentration of the crosslinker, NTEB, might provide improved propellant stability. The effects of this and other formulation variables are discussed in the following sections.

1. INCREASED CROSSLINKER CONCENTRATION

(C) Several formulations containing increased concentrations of NTEB were prepared and tested for storage stability by DTA. Because the high concentrations of NTEB provided plasticization, the MRPX plasticizer used in previous formulations was omitted. This omission alone provided a dramatic improvement in propellant stability and will be discussed in detail in section 2. Propellants L, H, J, and K (UTX 9146, 9143, 9144, and 9145) contain respectively 1.2, 1.5, 2.0, and 2.5 equivalents of NTEB. Complete formulations are given in table I. The DTA results shown in table II for samples L60, H60, J60, and K60 lead to several important conclusions. First, in terms of absolute DTA values and age at occurrence of explosive combustion, these propellants are remarkably stable compared to all previous formulations with uncoated, particulate NP. Second, while stability is improved by increased NTEB concentration throughout the storage period, the greatest differences appear during the first month. Finally, when explosive combustion does occur, exotherm inception temperatures have dropped only to ca 60° C instead of the 50° to 55° C level characteristic of propellant A.

(U) Samples of propellants L, H, J, and K were initially stored at 35° F and at 10° F and subsequently transferred to storage at ambient temperature. DTA results are shown in table II. Again, stability improved with increased NTEB content. Storage at the lower temperatures further improved stability, but the superiority of samples stored at 35° F to those stored at 10° F observed with propellant A was not apparent in these studies. This conclusion applies to the period of low temperature storage as well as the period of subsequent storage at ambient temperature.

(U) All of the foregoing samples which were initially stored at 60° F required 2 weeks to cure at that temperature. The samples stored at

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35° F were just beginning to cure when transferred to ambient temperature storage after ca 39 days. After 1 week at ambient temperature, the H, J, and L samples were cured. The K samples (2.5 equivalent wt NTEB) required 12 days to cure under this condition. Stability of the samples at ambient temperature is much greater (ca 4 weeks or more) than that of propellant A (1 week).

(U) It should be pointed out that all the samples discussed above were exposed to increased atmospheric moisture during a 5 day period. This exposure produced no evident trend in thermal stability, but did cause surface softening of the propellant samples. Those containing the higher concentrations of NTEB (J-2.0 equivalent wt, K-2.5 equivalent wt) were much less affected, and when moisture discipline was restored, they recovered their original appearance within 3 days.

2. PLASTICIZERS

(C) The great improvement in stability of propellant L over propellant A must be attributed to the absence of MRPX plasticizer in the former. While considerable effort has been expended in the past to develop a plasticizer superior to MRPX, the results have been unrewarding. Available nitroparaffins have proved to be too volatile to be practical, and a variety of hydrocarbons have shown inferior compatibility with NP.

(C) A candidate branched paraffin, TMPD, is a more effective plasticizer than MRPX, but is inferior in compatibility with uncoated NP. Since the chemical structure of TMPD implies low reactivity, impurities were suspected as being responsible for the poor compatibility with NP. A. D. McElroy of Midwest Research Institut has reported* that the products of interaction of NP with hydrocarbons are apparently largely confined to the oxidizer-hydrocarbon interface. This observation suggested that a hydrocarbon might be freed of reactive impurities by contact with NP followed by removal of the NP along with adsorbed reaction products.

(C) A 2.5 g sample of TMPD was placed in contact with 0.15 g of 30-40 mesh NP in the dry box. After 7 days at ambient temperature, the NP appeared sticky, but not discolored. On the ninth day of contact, a slight yellowing of the TMPD was observed. After 13 days, both the NP and TMPD showed a brown discoloration. At intervals during the treatment period,

* Quarterly Progress Report No. 2, Contract AF 04(611)-11199, April 1966

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small samples of TMPD were removed and incorporated in propellant formulations containing uncoated NP. All the formulations were stored at ambient temperature. Within 3 to 4 days, all but one propellant sample had deteriorated visibly (swelled). The formulation containing TMPD which had been treated for 9 days survived 6 days before swelling was evident. On the basis of these unfavorable results, no further work in this direction is planned.

(C) Another hydrocarbon plasticizer, HMN, is presently under study. This compound has a structure very similar to that of the backbone of UTREZ prepolymer and would be expected to exhibit outstanding compatibility with NP. Spot plate tests show that HMN is, in fact, more compatible than MRPX. Tests of propellants containing HMN are in progress.

(C) The improved stability of propellants containing high levels of NTEB may be attributed to the basic, tertiary amine group or simply to the basic, polymerizable imino groups. If the latter is the case, other aziridines may be useful. Of course, it must be recognized that cure chemistry and stoichiometry cannot be completely subordinate to stabilization processes. An aziridinyl compound less reactive than NTEB might provide stabilization and protection against binder reversion without participating importantly in crosslinking reactions of the binder. MAPO is quite compatible with NP but is far less reactive than NTEB in crosslinking UTREZ. Therefore, MAPO has been evaluated as a potential stabilizing plasticizer in UTREZ/NTEB propellants.

(U) Formulations IA and IIA (see table I) were prepared using MAPO at 5% wt of the binder. Thermal stability of these formulations (table II) at ambient temperature is outstanding. Studies of formulations containing both MAPO and HMN are in progress.

3. OXIDIZER PARTICLE SIZE

(U) Limited studies have been conducted to explore the effect of oxidizer particle size on propellant stability. Since reactions leading to degradation presumably occur at binder-oxidizer interfaces, reduction of interfacial area by use of coarse oxidizer would be expected to promote stability. Of course, there are important factors limiting the maximum practical size of oxidizer particles. Some of these factors are processability, mechanical properties of the propellant, and, especially, combustion efficiency.

(C) Comparison of the thermal stabilities of propellants M (UTX-9147) and A1D (UTX-9118) suggests that 8-20 mesh NP is to be favored over

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30-40 mesh oxidizer. However, this does not appear to be the case when MRPX plasticizer is omitted from the formulation and a high concentration (2.0 equivalent wt) of NTEB is employed (compare propellants N and O). Again, in the series 109-1 through 109-8, the coarse oxidizer shows no superiority over the 30-40 mesh material. In this series, effects of aluminum and NTEB concentration (2.0 and 2.5 equivalent wt) were studied. Finally, in the MAPO-plasticized systems (I-A, -B, -C and II-A, -B, -C) there appears to be little difference in the behavior of 30-40 mesh, 8-20 mesh, and crushed 8-20 mesh oxidizer.

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SECTION IV

CONCLUSIONS AND FUTURE ACTIVITY

(C) Continuing testing confirms the value of reduced storage temperature in prolonging the life of NP propellants. However, the beneficial effect of prior low temperature storage on subsequent stability at ambient temperature does not appear to be general. DTA provides a rough, but useful, indication of the tendency of deteriorated propellant to burn explosively.

(U) Increased concentrations of the crosslinker, NTEB, significantly improve propellant stability. The isoparaffinic plasticizer, MRPX, greatly contributes to the degradation process. Two materials, HMN and MAPO, are promising alternate plasticizers and are under active study. Oxidizer particle size has surprisingly little effect on propellant stability except with the least stable binder formulations.

(C) Experimental formulations containing uncoated NP have exhibited thermal stabilities superior to those of previous composite propellants containing coated or uncoated particulate NP and a binder with useful fuel value.

(U) Optimization of these promising formulations will be conducted with the objective of developing processable, ballistically interesting propellants for motor tests.

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13. ABSTRACT Unclassified Abstract A 24-month program is being conducted to produce practical, castable, composite solid propellants containing a high-energy oxidizer. Emphasis is being placed on improving the thermal stability and increasing the attainable solids loadings of propellants containing particulate oxidizer, a compatible binder (carboxy-terminated polyisobutylene), and polyfunctional aziridinyll crosslinking agents. Work during this report period was concerned with systems containing uncoated oxidizer. The effectiveness of reduced storage temperature in extending propellant life has been further demonstrated. Differential thermal analysis (DTA) has been found to provide a rough, but useful indication of the tendency of deteriorated propellants to burn explosively. Increased concentrations of the binder crosslinking agent have significantly improved propellant stability. The isoparaffinic plasticizer used previously has been found to impair propellant stability. Improved plasticizers have been demonstrated. Propellant stability has thereby been increased dramatically. Oxidizer particle size has been found to exert little influence on the stability of improved formulations.		

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14 KEY WORDS	LINK A		LINK B		LINK C	
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High-energy solid oxidizer						
Carboxy-terminated polyisobutylene						

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