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QUARTERLY PROGRESS REPORT
THERMALLY STABLE
BURNING RATE ACCELERATORS (U)

Contract F04611-70-C-0066 ✓

October 1971

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Prepared for
Air Force Systems Command
Air Force ^{ROCKET} Propulsion Laboratory
Edwards Air Force Base, California 93523

Attn: RPOR-ST/INFO

Prepared by

Thiokol / HUNTSVILLE DIVISION

Huntsville, Alabama

A DIVISION OF THIOKOL CHEMICAL CORPORATION

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QUARTERLY PROGRESS REPORT

1 June through 31 August 1971.

⑥ THERMALLY STABLE BURNING RATE ACCELERATORS (U).

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FOREWORD

This report is prepared in accordance with the requirements of Contract F04611-70-C-0066.

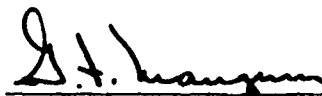
Program Structure Number	623148
Program Number	3148
Program Element Number	6.23.02F
Program Monitors	Capt C. L. Ennis and Capt D. J. Yardley USAF/RPMPC
Prime Contractor	Thiokol Chemical Corporation Huntsville Division Huntsville, Alabama

This report, the fifth quarterly progress report to be issued under this program, covers effort performed for the period 1 June 1971 through 31 August 1971. This report has been assigned the Thiokol internal number 32-71 (Control No. C-71-32A).

The principal investigators on the program are Dr. W. D. Stephens, Dr. D. A. Flanigan, and Mr. J. O. Hightower. Dr. M. Miller is serving as Program Manager. In addition to those contributions by the principal investigators, major contributions to the program were made by Dr. T. C. Willis, Messrs. J. W. Blank, C. M. Christian, Mrs. Patricia B. Walters, and Dr. R. E. Rogers. The over-all Project Director is Mr. G. F. Mangum.

STATEMENT OF APPROVAL

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



G. F. Mangum
Project Director

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ABSTRACT

(U) Experiments have shown that the thermal stability of propellants is a direct function of the UFAP and catalyst concentration. A new thermally stable catalyst (dimethylpolyferrocenylmethylene) has been synthesized and is undergoing preliminary evaluation. Indications are that fast burning propellants (2.5 in/sec at 1000 psi) which exhibit good thermal stability (less than 1% weight loss in 10 hours at 350° F), can be made using up to 30% UFAP and up to 3% catalyst.

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INTRODUCTION

(C) The purpose of this program is to research the methods and materials that will produce thermally stable AP and burning rate catalyst. They will be used with other thermally stable propellant ingredients to produce a solid propellant that is capable of withstanding a temperature environment of -75° to 350°F .

(C) With the present capability of aircraft to fly at supersonic speeds, a new requirement now exists for missiles which can be carried externally. The major development problem arises from the effects of aerodynamic heating, causing missile skin temperatures of several hundred degrees Fahrenheit. In addition, the external carry means that a missile in the air-stream is exposed to temperatures of about -65°F (the temperature in the isothermal layer). Missiles and missile components therefore are subject to temperatures from -65°F to well over 300°F .

(C) Propellants that will survive this treatment, and also possess the desirable ballistic properties, are not available. Two general approaches to assure the rocket motor's operation at all times are to insulate the motor or develop a propellant to withstand the environment. Internal or external insulations increase the motor cross sectional area and weight. Also, internal insulation decreases propellant volume. Since the missile is normally exposed to these conditions for a relatively short time, the insulation approach is inefficient. The penalties paid for insulation are too great; thermally stable propellant is a better solution.

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PROGRAM OBJECTIVES AND ORGANIZATION

OBJECTIVES:

(C). Very small particle oxidizer (less than one micron) and burning rate catalysts have been used to achieve high burning rates; however, commercially available ammonium perchlorate is prone to thermal decomposition which, in turn, degrades propellant. Some liquid burning rate catalysts also have undesirable characteristics such as crystallization, migration, or volatilization and are subject to thermal and oxidative decomposition. The objectives of this program are: (1) to develop methods of production of stable ultrafine ammonium perchlorate (UFAP), (2) to demonstrate improved methods of burning rate catalysis, and (3) utilization of these rate augmentation techniques in propellants which must survive temperature cycling up to 350°F.

PROGRAM ORGANIZATION:

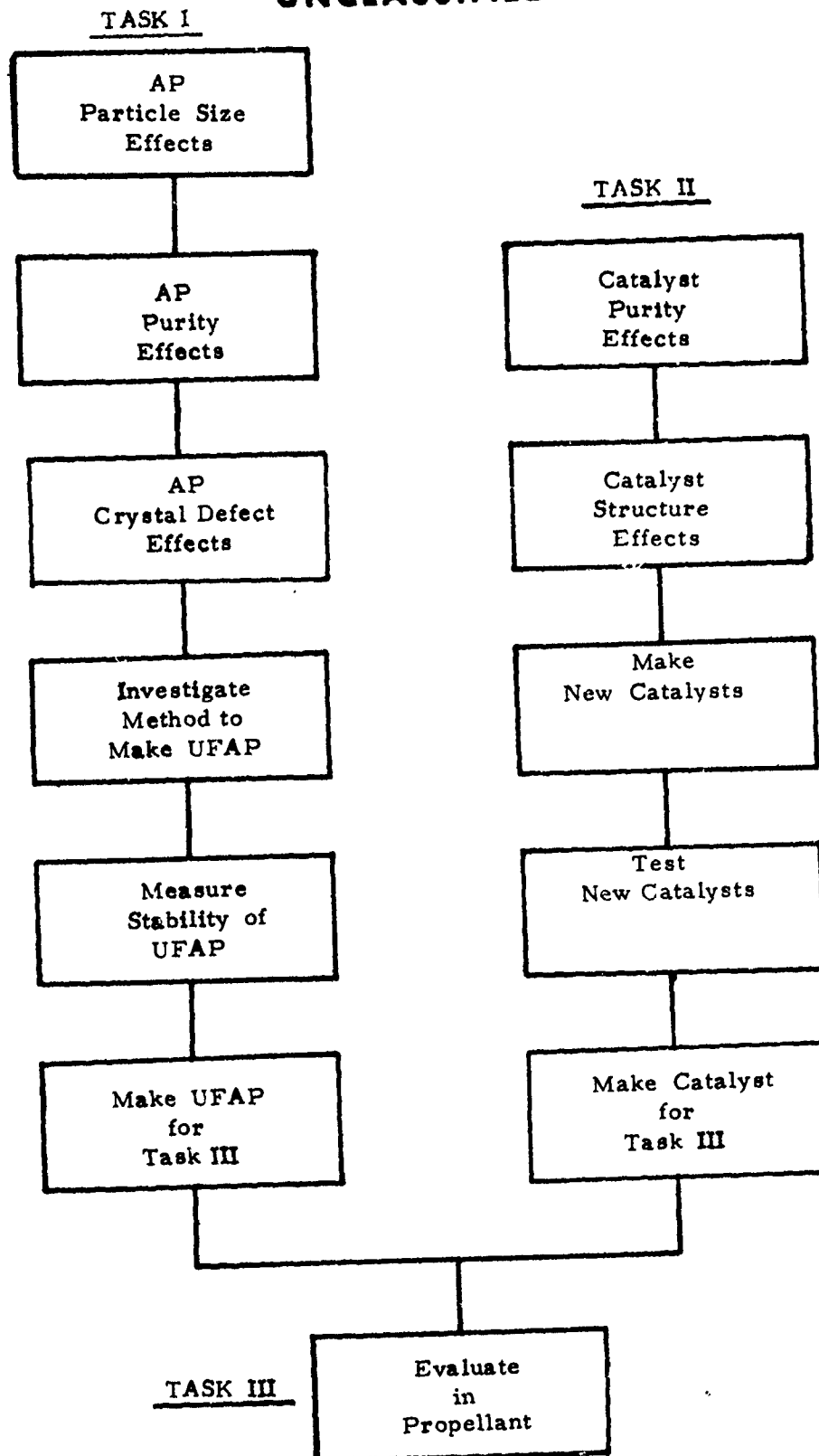
(U) This program is divided into three tasks as follows:

Task I, Thermally Stable UFAP, is subdivided into three subtasks. The first defines AP thermal stability and identifies the qualities that AP must have to be stable. In the second, new methods of preparing UFAP are examined and in the third subtask, the method of preparing UFAP is chosen and enough UFAP prepared for the demonstration part of the program.

Task II, Thermally Stable Catalysts, is divided into four subtasks. These include impurity investigations, structure investigations, improved catalysts, and synthesis of material for Task III.

Task III, Demonstration, is that part of the program in which the thermally stable UFAP and an improved catalyst are combined into a propellant for demonstration.

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PROGRAM OUTLINE

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TECHNICAL ACCOMPLISHMENTS

Task I - Thermally Stable Ammonium Perchlorate

Introduction

(U) With satisfactory thermal stability obtained with both the ultrafine ammonium perchlorate and ferrocene containing catalyst, experiments were designed to determine how much UFAP and catalyst could be incorporated into a high performance propellant without deleterious effect upon thermal stability.

UFAP Concentration Effects

(C) Small mixes (e.g., 10 g each) of propellant containing R45M HTPB/IPDI as the binder, 10% Al, 70% AP, and zero catalyst were prepared. The AP (pre-purified) was a blend of nominal 40 micron large fraction and 0.8 micron small fraction. The UFAP was prepared by spray freeze dry techniques and was incorporated in the various mixes at the 5, 15, 25, 40 and 60% levels. TGA data was obtained for 20 hours at 190°C on each cured sample. The propellant weight loss at the end of 20 hours of exposure served as the criterion for assessing the effect of UFAP concentration on thermal stability.

(U) The results are shown in the following table. The expected result of decreasing thermal stability with increasing UFAP concentration is noted.

UFAP Concentration Effects (375°F)

<u>% UFAP in Propellant</u>	<u>Time to 1% Wt. Loss</u> (hr)	<u>% Wt. Loss at 20 Hrs.</u>
5	11.00	2.09
15	8.42	3.15
25	6.33	3.75
40	6.20	4.37
60	5.10	5.67

Catalyst Concentration Effects

(U) Small mixes of propellant, such as described above, were prepared in which various amounts (0.2, 0.5, 1.0, 2.0, and 3.0%) of the binder were replaced with polyferrocenylmethylene (PFM) burn rate catalyst. The fine AP concentration was selected so that AP size effects, if any, were of secondary importance. Forty micron pre-purified AP was used as the single oxidizer fraction. TGA data were obtained for 6 hours at 160°C on each cured sample. Percent weight loss after 20 hours exposure was likewise noted.

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(U) The results are shown in the following table. The anomaly in the 20-hour data for 1% PFM was rechecked by running another isothermal TGA at 160°C. The resulting data (parentheses) is more in line with the expected trend.

Catalyst Concentration Effects (160°C)

<u>% PFM</u>	<u>% Wt. Loss at 6 Hrs.</u>	<u>% Wt. Loss at 20 Hrs.</u>
0.2	0.10	0.69
0.5	0.22	1.25
1.0	0.47 (0.59)	1.18 (1.80)
2.0	0.68	2.16
3.0	0.74	2.32

(C) Following a determination of the effect of UFAP concentration on thermal stability and a similar set of experiments designed to elucidate catalyst concentration effect upon thermal stability, a series of mixes was manufactured in which UFAP/catalyst interactions were examined. Small mixes of propellant (70AP/10A1/HTPB/IPDI) were manufactured. The UFAP utilized in the phase of testing was prepared by the spray freeze dried technique and exhibited an average particle diameter 0.78 microns. The remaining AP was prepurified by flash precipitation from isopropyl alcohol. The catalyst utilized in this study was polyferrocenyl methylene (PFM). The polymer, curing agent and aluminum were predried before mix manufacture.

(U) The mix matrix contained three separate UFAP concentrations and three catalyst concentrations at each UFAP concentration. The UFAP concentrations examined were 10, 30 and 60% and at each of these concentrations the catalyst content was varied from 0.2 to 3.0% (Table I).

(U) Isothermal TGA's were performed at 160°C and the weight loss at the end of 6 hours exposure served as a criterion for assessing any interaction effects on thermal stability. The test matrix and the results are shown on Table I. It is apparent from these experiments that increases in UFAP concentration do not appear to significantly degrade thermal stability. The expected effect of a decrease in thermal stability with increasing catalyst concentration is apparent. It appears from these data that good (less than 1% weight loss) thermal stability can be achieved at 350°F for at least ten hours in propellants containing 30% UFAP and 1-2% PFM. This judgement is based upon the fact that these samples examined by TGA were 1/4-inch cubes and exposed very large surface per unit weight. The percent weight loss will decrease markedly with decreasing surface area to weight ratios.

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TABLE I
UFAP/CATALYST INTERACTIONS
ISOTHERMAL TGA (160°C)

<u>Sample</u>	<u>% UFAP</u>	<u>% PFM</u>	<u>% Wt. Loss</u>		<u>Time to 1% wt loss</u>
			<u>6 hours</u>	<u>20 hours</u>	
1	10	0.2	0.33	1.22	16.25
2	10	1.0	0.69	2.30	8.10
3	10	3.0	0.91	2.77	6.5
4	30	0.2	0.28	0.96	21.0
5	30	1.0	0.60	2.05	9.5
6	30	3.0	1.22	3.12	5.0
7	60	0.2	0.18	0.93	> 21.0
8	60	1.0	0.62	1.62	10.0
9	60	3.0	1.32	5.04	4.5

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Propellant Sample Size Effects

(U) A formulation of high burn rate thermally stable propellant was selected for manufacture as a master batch of propellant to be used in a series of small mixes to compare the relative stability of several burn rate catalysts and to determine if the sample size affects the previously drawn conclusions, since previous TGA data were obtained on small cubes of propellant. The master batch was to be subdivided into 600 gram lots. Into each of these 600 gram samples will be added 2% of several thermally stable burn rate catalysts.

(C) The formulation selected was one containing 70% AP and 16% Al powder. The polymer utilized was ARCO's R45M/HTPB polymer cured with IPDI. The oxidizer blend consisted of 25% freeze-dried AP (particle size 0.88 microns), 35% fluid-energy milled AP exhibiting a particle size of 2.1 micron and 40% 50 micron rounded AP. Each of the oxidizer samples were predried to minimum moisture contents and thermal stabilities determined on each of the fractions before mix manufacture. Thermal stabilities in excess of 30 hours at 375°F were obtained. The aluminum powder was predried at 110° under vacuum for 24 hours and the HTPB polymer was likewise vacuum dried and an antioxidant (A.O. 2246, 1%) was added to the polymer. The curing agent was dried over molecular sieves for 24 hours before utilization.

(U) The mix was manufactured in a one-gallon vertical mixer (9 lbs.) and mixed under vacuum. After all the ingredients were added (no curing agent nor catalyst was incorporated at this point), the mix was mixed at 170°F for 5 hours to eliminate moisture and other volatiles. The EOM viscosity was 18 Kp.

(U) Following completion of the mix, 600 gram samples were taken and sealed in aluminum foil. To each of the samples will be added 2% of several different thermally stable burning rate catalysts and two-inch cubes will be manufactured in order to determine the thermal stability character of each of these catalyzed formulations.

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Task II. Thermally Stable Catalysts

(U) Efforts to obtain thermally stable catalysts are proceeding essentially as planned. In Subtask 1, critical impurities were identified, and methods for removal were developed. In Subtask 2, molecular characteristics which contribute most to thermally stable catalysts were identified. The current report is concerned primarily with Subtask 3 which is the synthesis of improved catalysts based upon information gained in the two prior Subtasks.

(U) In the previous report, the stability data of propellants containing AP which had been aged at 25% relative humidity were reported. Fine, stable AP showed a marked improvement in a propellant catalyzed with benzoyl ferrocene. The large, stable AP showed a slight decrease in stability (Figures 1 and 2). It has been learned that the particle size of the fine, stable oxidizer approximately doubled during aging while that of the large, stable AP remained unchanged. This probably accounts for the change in stability of propellant containing fine, stable AP.

(U) Insoluble products which have no alpha hydrogen atoms present are being sought through the co-polymerization of ferrocene with acetone. It is hoped that polymerization conditions can be found which will yield an α, α -dimethylpolyferrocenylmethylene (DMPFM). Table I is a summary of reactions which have been attempted in order to prepare the desired products. A large number of variables has been investigated in this study. These include reaction time, temperature, order of addition or reaction sequence, catalyst and co-catalyst ratio, and acetone to ferrocene ratio.

(U) Two very attractive products have come from this work; the product from reaction 7 in Table II, and that from reaction 19. The elemental analysis of the reaction product number 7 (FeAc-7) is as follows: C64.79, 64.92; H6.17, 6.45; Fe22.57, 22.42. If it is assumed that the unaccounted composition is present as oxygen (halogens, sulfur, and nitrogen have been shown to be absent), then the empirical formula calculated is $C_{13}H_{16}FeO$. The product from reaction 19 (FeAc-19) gave the following analysis: C69.8; H6.5 (theoretical values for DMPFM are C69.09; H6.19; Fe24.71).

(U) Both of these products were totally insoluble in all solvents investigated (14 solvents). Purification of these compounds was accomplished by continuous Soxhlet extraction to remove all soluble components. Several solvents were used. The solubility properties and composition of the product are distinctly different from those reported by Paushkin.¹

1. Y. M. Paushkin, T. P. Vishnyakova, I. I. Patalakh, T. A. Sokolinskaya and F. F. Machus, Doklady Akad. Nauk. S. S. S. R., 149, No. 4, p. 856 (1963).

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TABLE II
REACTION CONDITIONS FOR DMPFM

No.	Fc(g)	$\text{CH}_3-\overset{\text{O}}{\underset{\text{ }}{\text{C}}}-\text{CH}_3(\text{g})$	Catalyst	Co-Catalyst	Product
1.	10	xs	5.3g H_2SO_4		Black insoluble solid
2.	10	4	xs H_3PO_4		Black insoluble solid
3.	10	4	Alc. KOH		Very little reaction
4.	10	xs	1 ml H_2SO_4	6.8 g ZnCl_2	Slight reaction
5.	10	6	5 ml H_2SO_4	0.1 g ZnCl_2	Orange solid
6.	10	6	5 ml H_2SO_4	0.1 g ZnCl_2	Orange solid
7.	10	6	5 ml H_2SO_4	0.1 g ZnCl_2	63% yield, orange solid
8.	10	6	5 ml H_2SO_4	0.1 g ZnCl_2	No reaction at 0°C
9.	10	6	5 ml H_2SO_4	0.1 g ZnCl_2	Charred black product at elevated temp.
10.	10	6	1 ml H_2SO_4	0.1 g ZnCl_2	Slight reaction
11.	100	60	50 ml H_2SO_4	1.0 g ZnCl_2	Reaction fumed off
12.	100	60	50 ml H_2SO_4	1.0 g ZnCl_2	32% yield, orange solid
13.	18.6	2.9	Na-Hg		No reaction
14.	18.6	116	HCl	4.8 g ZnCl_2	Low yield, soluble polymer
15.	18.6	116 (dry)	HCl	4.8 g dry ZnCl_2	11.2 g hexane soluble polymer
16.	18.6	11.6	HCl	4.8 g ZnCl_2	Ether solvent, low yield of product
17.	93	58	HCl	24.55 g ZnCl_2	Ether solvent. Carbonized product.
18.	18.6	11.6	HCl	13.6 g ZnCl_2	Ether, low yield
19.	18.6	17.4	HCl	13.6 g ZnCl_2	50% yield, yellow insoluble solid
20.	9.3	5.8	HCl	6.8 g ZnCl_2	Ether pre-saturated with HCl no reaction
21.	9.3	5.8	HCl	6.8 g ZnCl_2	Ether 50% saturated with HCl no reaction
22.	9.3	20	HCl	6.8 g ZnCl_2	10% soluble polymer some insoluble polymer
23.	9.3	20	HCl	6.8 g ZnCl_2	1% soluble polymer, no insoluble polymer
24.	18.6	11.6	HCl	9.5 g ZnCl_2	No reaction at 25°C

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(U) Small propellant mixes were made using these materials and the propellants were investigated in the standard TGA weight loss test as shown in Figure 1. Both materials appeared to be improvements over PFM, and are quite attractive. Note that at 8 hours, propellants made from the two new polymers had lost only 60% as much weight as those from PFM, which was the best catalyst of all the models studied.

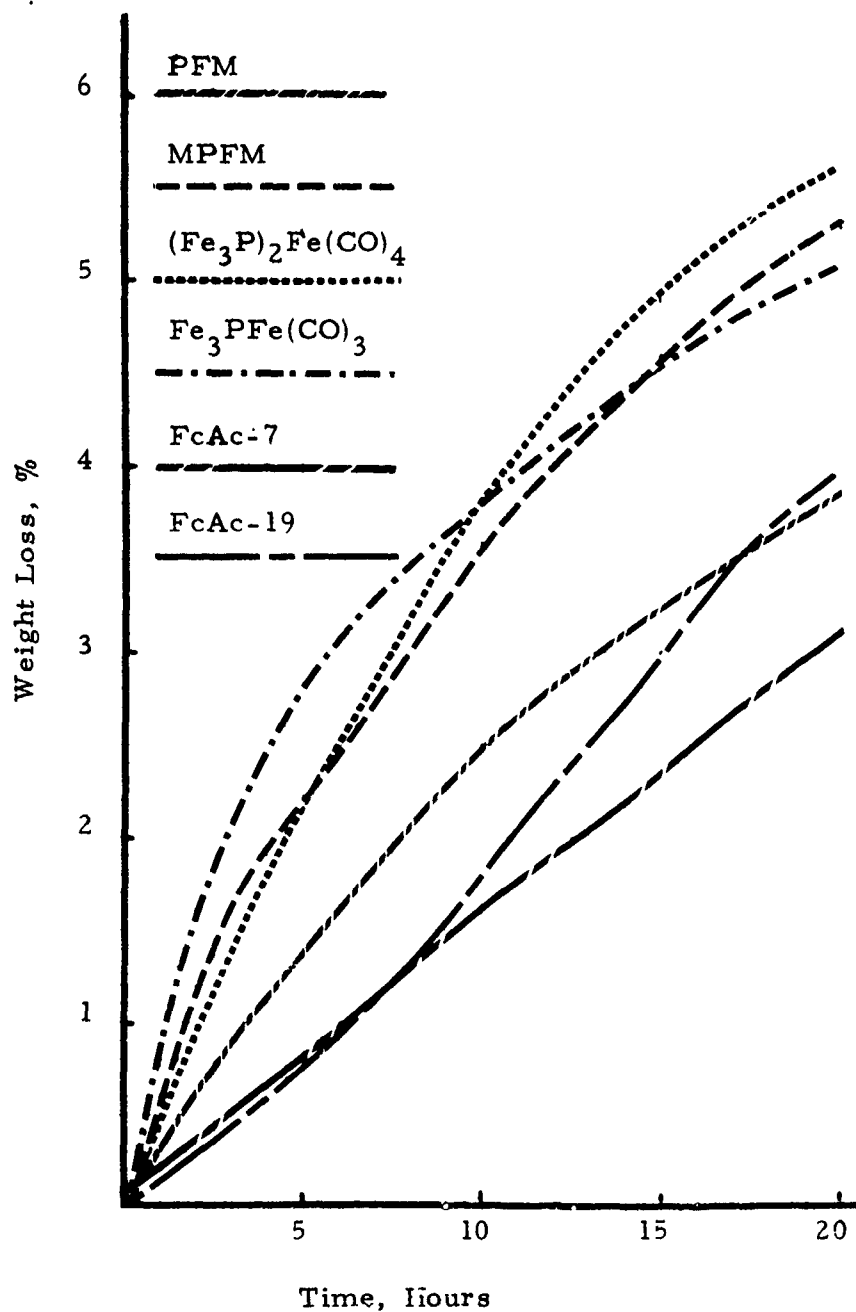


Figure 1. TGA of Propellant at 320°F

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(U) Two components from Frankfort Arsenal have been made into propellant and evaluated in this same test. These include triferrocenylphosphineiron tetracarbonyl, and bis-(triferrocenylphosphine) iron tricarbonyl. These compounds were relatively quite good with regard to thermal stability, being among the better compounds tested, but not quite as good as the most stable model (PFM, see Figure 1.).

(U) A polymeric product similar to PFM in which one of the benzylic type hydrogen atoms had been replaced by a methyl group was prepared from hydroxyethylferrocene. The polymer was formulated into propellant and evaluated by a TGA test. It was not quite as attractive as PFM.

(U) Attempts have been made to obtain similar polymers, but these have not given the encouraging results seen with the acetone-ferrocene products.

(U) The polymerization of 2-ferrocenyl-2-butanol was attempted using the same conditions as those used for polymerizing hydroxymethylferrocene. However, no polymeric products were obtained.

(U) 2-Ferrocenylisopropanol was prepared from acetylferrocene and methyl-magnesium bromide. This was used in polymerization reactions to obtain a polyferrocenyl-methylene type polymer which contains no alpha hydrogens, however, better results were obtained in going directly from ferrocene and acetone.

(U) The polyrecombination of ferrocene to make polyferrocenylene was abandoned because of experimental difficulties. At the reaction temperature of 200°C ferrocene sublimed rapidly and flask outlets stopped up. Fires occurred during the reaction.

(U) The two insoluble ferrocene polymers; FcAc-7, the polymer obtained from ferrocene, acetone, zinc chloride, and sulfuric acid, and FcAc-19, the polymer using hydrogen chloride in place of sulfuric acid were made in 50-grain batches.

(U) Thermogravimetric analyses in nitrogen indicated that both polymers are stable up to approximately 750°F. In air the limit is 340°F for FcAc-7 and 355°F for FcAc-19. Differential scanning calorimetry indicated similar results.

(C) A cross-check of propellant burning rate efficiency was made on the two new catalysts. Propellant containing 89.5% solids was prepared using a formulation which is essentially the same as that of a propellant containing 2.5% n-butyl ferrocene and which has been evaluated in large motors and laboratory tests for several years. The formulation included 71% oxidizer (69% unground and 31% high speed grind, 16 micron) 16% aluminum, 2.5%

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(C)
catalyst, and 10.5% HC binder with a mixed curing system using a 5/1 ratio of MAPO to ERLA-0510, with 0.1% iron linoleate as a cure catalyst. The propellant was cured using a 1/0.84 ratio of carboxylic acid to curing agent. Mix 13Q-633 was made using butylferrocene as a standard; the end-of-mix viscosity was 14 Kp at 137°F. Mix 2198 used FcAc-19 as the catalyst at 2.5%, and had an end-of-mix viscosity of 140 Kp at 120°F. Mix 2199 used FcAc-7 as a catalyst and the end-of-mix viscosity was off-scale. The burning rate curves are shown in Figure 2. The new burning catalysts are about 90% as effective as P-IV even though they are insoluble solids.

◇ 13Q633, P-IV

○ 2198, FcAc-19

□ 2199, FcAc-7

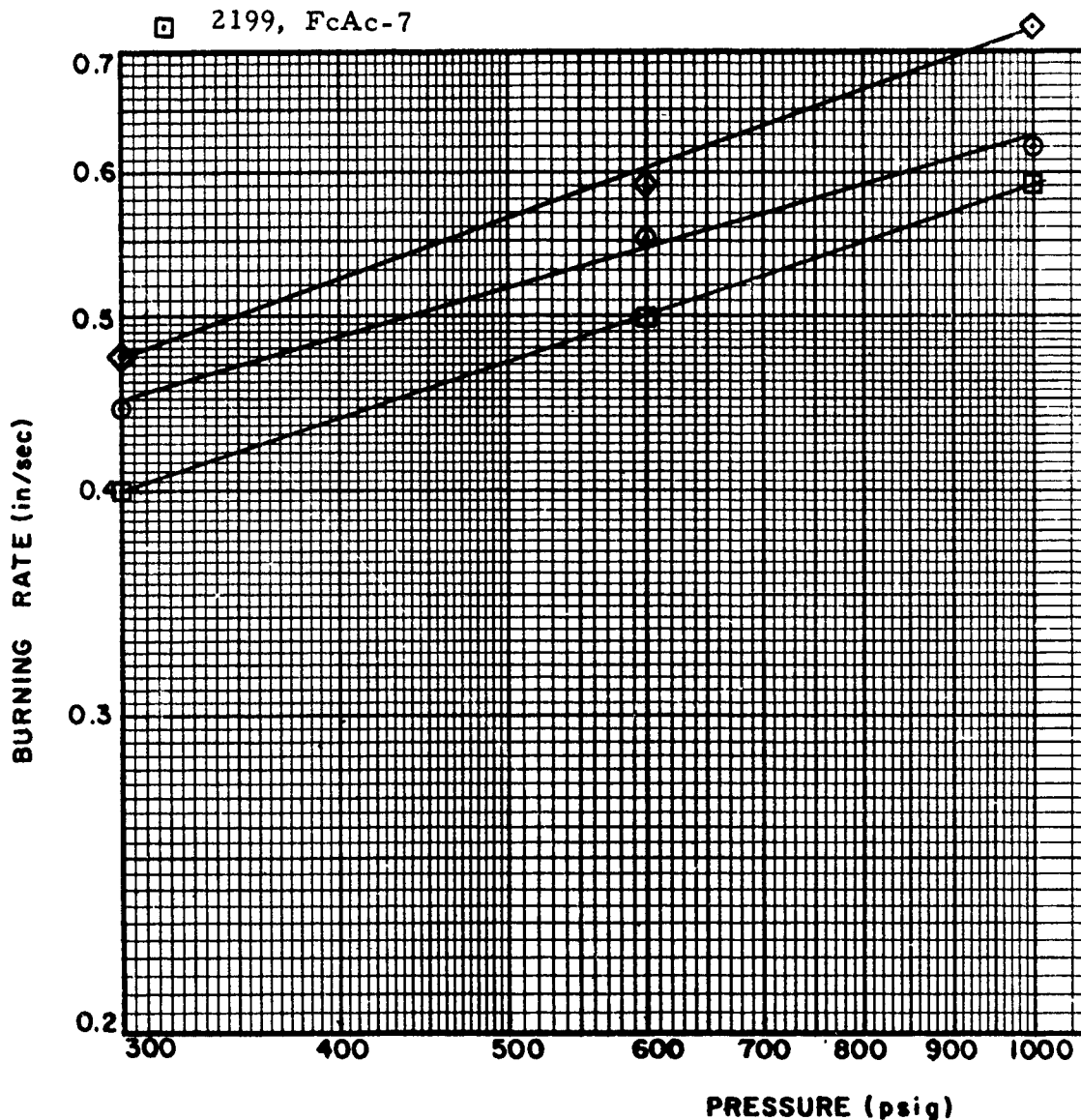


Figure 2. Burning Rate Comparisons (U)

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(U) Sensitivity comparisons were made by performing impact, friction, and spark sensitivity determinations as well as differential thermal analyses upon propellants from the mixes mentioned above. This data is shown in Table III. The sensitivity of propellants made using the two new catalysts is comparable with the sensitivity of propellants made with butyl ferrocene. With regard to the differential thermal analysis, slightly higher temperatures are achieved before the initial exotherm occurs.

TABLE III

SENSITIVITY DATA (U)

<u>Catalyst (mix no.)</u>	<u>Spark (joules)</u>	<u>Friction (lbs)</u>	<u>Impact (Eo, Kg-cm)</u>	<u>DTA (initial exo.)</u>
FcAc-19 (2198)	-25	-85	-40 +50	386°F
FcAc-7 (2199)	-25	-75 +85	-50 +60	377°F
P-IV (13Q633)	-25	-75 +85	-40 +50	341°F

CONCLUSIONS:

(U) Two new catalysts, FcAc-7 and FcAc-19 have shown promise of good thermal stability in propellants by several criteria including DSC in air and in nitrogen, and TGA tests on neat catalyst and propellant. Burning rates have been evaluated in strands. The FcAc-19 product appears to be cleaner and more uniform, and to consist entirely of carbon, hydrogen and iron. The structure of FcAc-7 is less certain. Primarily for this reason, as well as the slight advantage in burning rate, it has been recommended that FcAc-19 be scaled-up for further evaluation under Task III. This recommendation has been accepted and scale-up will be instituted during the next reporting period.

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		2b GROUP iv
3 REPORT TITLE Quarterly Progress Report, Thermally Stable Burning Rate Accelerators 1 June through 31 August 1971		
4 DESCRIPTIVE NOTES (Type of report and inclusive dates) Quarterly Progress Report, 1 June through 31 August 1971		
5 AUTHOR(S) (Last name, first name, initial) Flanigan, D.A. Stephens, W.D.		
6 REPORT DATE October	7a. TOTAL NO. OF PAGES 20	7b NO OF REFS 1
8a CONTRACT OR GRANT NO F04611-70-C-0066	9a. ORIGINATOR'S REPORT NUMBER(S) 32-71 Control No. C-71-32A	
b PROJECT NO 3148		
c		
d	9b OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
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