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(U) DEMONSTRATION OF AN ADVANCED SOLID PROPELLANT

REPORT NO. AFRPL-TR-65-225

NOVEMBER 1965

Prepared by

O.A. DEWHIRST, UTC

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for

AIR FORCE ROCKET PROPULSION LABORATORY

RESEARCH AND TECHNOLOGY DIVISION

AIR FORCE SYSTEMS COMMAND

UNITED STATES AIR FORCE

EDWARDS AIR FORCE BASE, CALIFORNIA

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United Technology Center DIVISION OF UNITED AIRCRAFT CORPORATION

15 November 1965

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Air Force Flight Test Center
Edwards Air Force Base,
California 93523

Attention: FTMKR-4/M. Racovich

Subject: Written Progress Report,
AFRPL-TR-65-225 (UTC 2146 QPR2)

Reference: Contract AF 04(611)-10812, DD 1423, Line Item No. 6

Gentlemen:

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

This report covers the period of 1 August 1965 through 31 October 1965.

Very truly yours,

UNITED TECHNOLOGY CENTER
A Division of United Aircraft Corporation

B. K. Forman, Manager
Contract Management

BKF:erb

Enclosure

cc: AFRPL, Edwards AFB, Calif. 93523
Attn: RPMC (w/3 encls.)
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Attn: CMRQK (w/1 encl)
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DEMONSTRATION OF AN ADVANCED SOLID PROPELLANT

Prepared by

O. A. Dewhirst, UTC
T. N. Scortia, UTC

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FOREWORD

(U) The work performed under this project is in response to requirements of AFFTC Project 3059, Program Structure No. 750G, BPSN 623059. The approving authority is Richard C. Miller, 2d Lt, USAF/RPMC, AFFTC, Edwards Air Force Base, California.

(C) The present report is the second of three quarterly reports on Contract No. AF 04(611)-10812 under which United Technology Center (UTC) is conducting a program to continue the development of an advanced solid propellant based on aluminum hydride, ammonium perchlorate, and nitrato-plasticized polyester and having a theoretical specific impulse (I_{sp}) in excess of 280 lb-sec/lb at standard conditions.

(U) This report covers experimental work conducted at UTC's Sunnyvale, California, research laboratories and at UTC's San Jose, California, processing laboratories during the period of 1 August 1965 through 31 October 1965. The following professional workers made significant contributions to progress on this program:

J. W. Allan	R. M. Kumagai
P. L. Allen	W. E. Robertson
J. D. Breazeale	R. I. Sutton
G. J. Casaletto	J. K. West
E. C. Francis	

(U) This report contains classified information extracted from "Demonstration of an Advanced Solid Propellant (U)," Report No. AFRPL-TR-65-195, August 1965.

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

Richard C. Miller, 2d Lt, USAF/RPMC, Project Officer

ABSTRACT

(U) An evaluation of surfactants for reducing propellant mix viscosity has resulted in the selection of Atlas G-2684. Particle size distribution studies have led to the use of a 50/50 coarse/fine ammonium perchlorate (AP) grind ratio to maximize propellant castability. Processing studies have also resulted in a capability of processing propellant at the 23 vol % binder level with a consequent improvement in theoretical performance and density.

(C) A series of 4-lb motors have been tested containing aluminum hydride/aluminum combinations representing the most dense formulations with a theoretical performance in excess of 280 sec and the most dense formulation with a theoretical performance in excess of 274 sec. Propellant UTP 6825 has a theoretical I_{sp} of 280.1 sec and a theoretical density of 0.0604 lb/in.^{3} Propellant UTP 6826 has a theoretical I_{sp} of 275.0 sec and a theoretical density of 0.0627 lb/in.^{3}

(U) A study to determine the effect of propellant formulation parameters on burning rates has been essentially completed. An end-burning micromotor was utilized for these tests. It was found that the oxidizer-to-fuel (O/F) ratio, AP concentration, and AP particle size distribution were the most critical parameters. Binder concentration and plasticizer level were found to be less significant.

(C) Efforts to minimize the handling hazards of aluminum hydride by increasing the electrical conductivity of the particles have been partially successful. A 1% coating of an antistatic agent reduced the electrical resistance of a 1 in. by 1 in. by 1/4 in. bed of particles from 1000 megohms for uncoated hydride to 40 to 70 megohms for the treated material.

(C) Propellant density studies indicate that in addition to the extensive drying of ingredients, propellant density can also be increased by sustained vibration and vacuum applied to the cast propellant. Surface treatment of the aluminum hydride with quinones, alizarin, or alizarin red S results in substantial improvements in propellant density. Testing with free-radical inhibitors has resulted in no noticeable improvements in propellant densities.

(U) The most promising curative systems evaluated to date are MAPO/Epon 812, MAPO/NTPB/Epon 812, and MAPO/TBM/Epon 812. Propellants containing these curative systems are in accelerated storage. Polyesters of increased molecular weight have been tested and appear to yield improved mechanical properties. Utilization of a molecular still for stripping moisture and light ends from the HX 735 polymer also results in some improvement in mechanical properties.

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I. INTRODUCTION

1.1 PROGRAM SCOPE

(C) Under Contract No. AF 04(611)-10812 with the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Edwards Air Force Base (EAFB), UTC is conducting a program to continue the development of an advanced solid propellant based on aluminum hydride, ammonium perchlorate, and nitreto-plasticized polyester and having a theoretical specific impulse in excess of 280 lb-sec/lb at standard conditions. This program continues the efforts initiated under Contracts No. AF 04(611)-8513 and AF 04(611)-9470. It will provide data on the important propellant parameters required to produce a family of usable propellants containing aluminum hydride.

(U) The program is divided into two phases: Phase I, formulation improvement and tailoring experiments, and Phase II, optimum formulation characterization.

1.2 PHASE I: FORMULATION IMPROVEMENT AND TAILORING EXPERIMENTS

(U) Phase I is divided into six tasks. These tasks, described in the following paragraphs, are interrelated and are being performed concurrently.

1.2.1 Task A10: Maximum I_{sp} and Solids Loading

(U) Task 1 has as its objective the achievement of the highest possible delivered I_{sp} consistent with the normal constraints on such a system. This objective will be accomplished by a study of processing parameters to achieve maximum solids loading and by formulation studies to optimize the concentration and type of oxygenated plasticizer. Results will be demonstrated in 10-lb motors

1.2.2 Task A20: Burning Rate Studies

(C) Task 2 has as its objective the development of a wide range of burning rates for this propellant system. Target burning rates of 0.25 in./sec and 1.0 in./sec at 1000 psi have been established. The following formulation parameters will be investigated: particle size, distribution of solid ingredients, plasticizer levels, use of burning-rate catalysts, flame-retardant coatings, O/F ratios, and the use of gassing agents. Four formulations

will be demonstrated in 4-lb motors: the fastest burning, the slowest burning, the most energetic slow burning, and the fastest burning with good physical properties. The two best formulations will subsequently be tested in 10-lb motors.

1.2.3 Task A30: Density-Isp Tradeoffs

(U) Task 3 involves a study of density-I_{sp} tradeoffs to achieve substantially increased propellant densities with minimum degradation of I_{sp}. A theoretical calculation effort will explore the influence of various parameters on density impulse. The two most promising formulations will be tested in 4-lb motors, and the best formulation will be demonstrated in 10-lb motors.

1.2.4 Task A40: Improvement of Safety Propellants

(U) Task 4 will have the objective of improving propellant safety properties. Propellant sensitivity will be reduced by the use of coatings and by changes in formulation parameters. Results will be measured by changes in impact sensitivity, differential thermal analysis (DTA), and spark sensitivity.

1.2.5 Task A50: Aging and Temperature Limits

(U) Task 5 will be a substantial effort devoted to defining the aging characteristics and the storage temperature limitations for this system. Techniques for improving storage limits will receive considerable attention. This work will include static and dynamic DTA studies, gas evolution studies as a function of temperature and humidity, gas diffusion through various web thicknesses, and the effects of constant strain on storability. The test program will include the use of analog motors and a propellant segment in a large motor. In addition, eighteen 10-lb motors and other specimens will be shipped to the Air Force for surveillance at their facilities.

1.2.6 Task A60: Improvement of Mechanical Properties

(U) Improved mechanical properties will be developed under task 6. The propellant curative system will be optimized for mechanical properties, new curatives will be evaluated, and testing will be performed at two strain rates over a range of temperatures from -60° to +130° F. Analog motor tests will also be performed.

1.3 PHASE II: OPTIMUM FORMULATION CHARACTERIZATION AND DEMONSTRATION

(U) Phase II involves a complete characterization of the propellant formulation resulting from the Phase I studies, including a complete ballistic characterization in 10-lb motors and mechanical property and hazardous property testing. Six Bates motors will be processed and shipped to the Air Force for scaleup testing.

1.4 REPORT STATUS

(U) The present report covers the experimental work performed during the second quarter, 1 August 1965 through 31 October 1965. The reporting status for the program is presented graphically in figure 1.

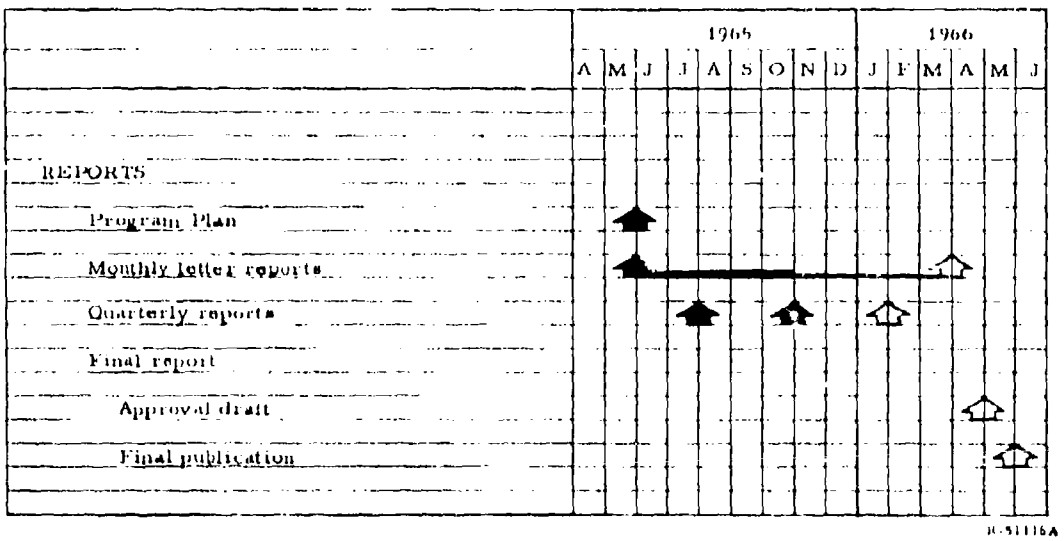

Figure 1. Report Status

II. TECHNICAL DISCUSSION

2.1 MAXIMUM I_{sp} AND SOLIDS LOADING (A10)

(C) This task has as its objective the formulation of a propellant system with the highest possible delivered specific impulse in a practical system. This objective is being accomplished by a study of processing parameters to achieve maximum solids loading and by formulation studies to optimize the concentration and type of oxygenated plasticizer. In addition, the effect of aluminum hydride concentration on specific impulse efficiency will be reexamined briefly at high O/F ratios (1.2 to 1.4).

2.1.1 Development of a Fuel Processing Simulant

(C) To allow the rapid evaluation of various processing parameters such as higher solids loading, particle size distribution, and processing time and temperature studies, a processing simulant for aluminum hydride was developed. Its utilization resulted in reduced cost of processing studies and minimized remote handling requirements during the preliminary stages of propellant processing studies. The material selected as the fuel simulant is ammonium oxalate. The evaluation of this material is described in the first quarterly report (AFRPL-TR-65-195).

2.1.2 Surfactant Studies

(U) A series of surface active agents were evaluated for their effect on propellant viscosity. This evaluation was conducted to determine which surfactant would provide the greatest reduction in mix viscosity so that a maximum solids loading could be achieved in a castable system.

(U) A number of different surfactants were evaluated during the first quarter with formulation UTP 6814 premix (binder and oxidizer only) at the laboratory scale at a concentration of 1%. Results of the laboratory investigation indicated that the following surfactants yielded significant processing improvements and were selected for studies at the 1-gal mix level:

Tween 20
Tween 21
Atlas G-2684.

These surfactants were investigated using UTP 6822, with ammonium oxalate as the control formulation.

UTP 6822

Ingredients	Wt %
PEP-150 binder	18.9
1451 simulant	23.0
Oxidizer	58.0
Surfactant	0.1
	100.0

(U) Of the three candidate surfactants, Atlas G-2684 was the most effective in reducing the viscosity of the simulated propellant mix. The discharge rates through a 0.25 in. by 3.00 in. rheometer orifice for mixes with the candidate surfactants are shown in figure 2. All mixes at the 1-gal mix scale now utilize Atlas G-2684 at a concentration of 0.1 wt % of the total propellant formulation.

2.1.3 Particle Size Distribution Studies

(C) An experimental processing program was conducted to optimize particle size distribution. To reduce the number of possible particle size combinations, it was decided to utilize the aluminum hydride in the particle size in which it is received and to vary the ratio of "coarse" oxidizer to "fine" oxidizer. The aluminum hydride particles are generally considered to have an average particle size of about 100 μ although there is considerable variability from lot to lot. However, since larger mixes require blends of several lots, the mix-to-mix variations of aluminum hydride particle distributions are minimized. The ammonium perchlorate size fractions which have been found to result in minimum mix viscosities in other propellant systems at UTC are the 300-μ (+48 mesh) size and the "fine" 8 to 12-μ size.

(U) A series of mixes were processed utilizing ammonium oxalate as a simulant for Dowane 1451. The propellant utilized a PEP-150 binder at 25 vol % binder and a constant 25 wt % Dowane 1451 simulant. The following ratios of coarse/fine oxidizer fractions were evaluated: 16/84, 23/77, 30/70, 50/50, and 60/40. The oxidizer size ratio which resulted in the maximum propellant fluidity was 50/50. The propellant fluidity as measured by its discharge rate through a 0.25 in. by 3.00 in. orifice at given rheometer pressures is shown at the various oxidizer size ratios in figure 3.

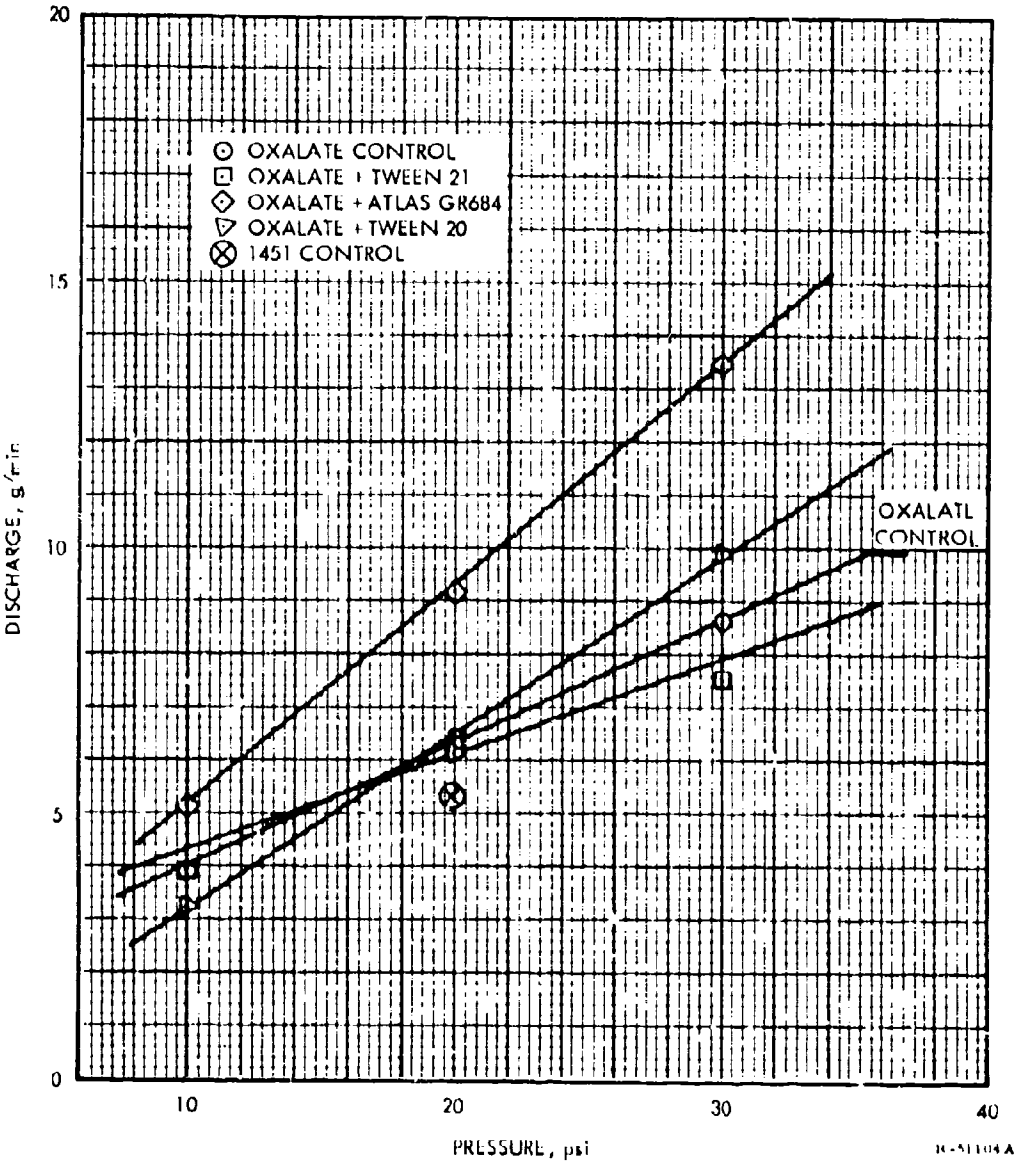

Figure 2. (U) Effect of Surfactants on Viscosity of Simulated UTP 6822, Rheometer at 120° F

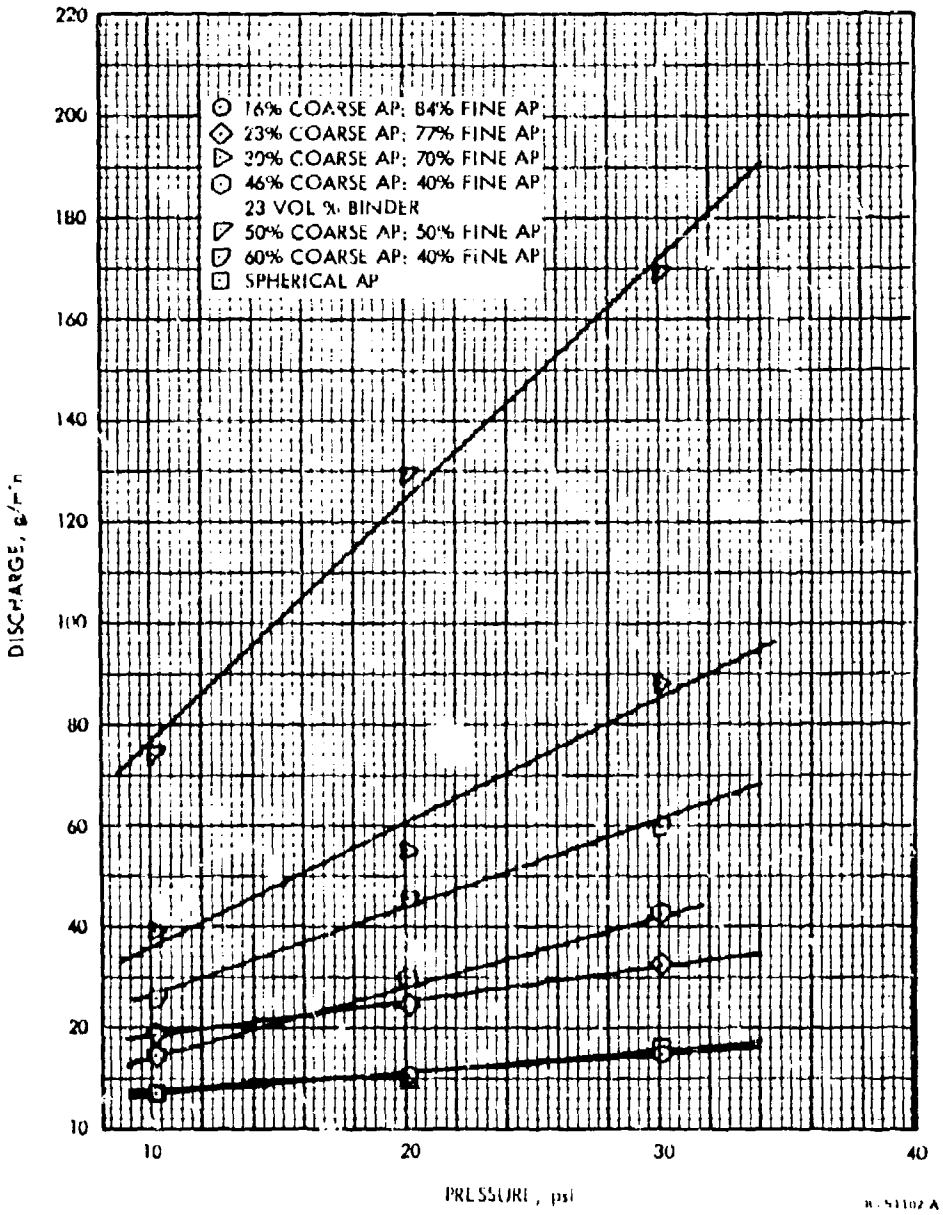

Figure 3. (U) Effect of Coarse/Fine Oxidizer Ratio on Castability of Simulated UTP 6822, Rheometer at 120° F

(U) An additional variable which is considered significant to propellant processability is the particle shape of the oxidizer. The fine oxidizer fractions utilized on Contract No. AF 04(611)-9570 consisted of rounded particles processed in a jet pulverizer and classifier. This product, obtained from an outside vendor, became unavailable. As a result the fine fraction was obtained from in-plant grinding. The in-plant material is processed in an impact mill and a more jagged, irregular particle is obtained. However, comparative mixes indicated no measurable difference in propellant processability when one material shape was substituted for the other. The similarity in processing characteristics for these two materials is illustrated in figure 3.

2.1.4 Plasticizer Evaluation

(C) In the previous quarterly report (AFRPL-TR-65-195), an extensive theoretical treatment of the affect of other nitrate plasticizers on the performance of the AP-oxidized aluminum hydride system was presented for propellants formulated at the 55 wt % plasticizer level. The plasticizers investigated as replacements for trimethylolethane trinitrate (TMETN) have been 1,2,4,-butanetriol trinitrate (BTTN), 2,2-dinitro propylnitrate (DNPN), and trimethylolnitromethane trinitrate (TMNTN or NIB-nitroglycerine). The ternary performance diagrams of these systems are shown in figures 7, 8, and 9 of AFRPL-TR-65-195.

(C) For purposes of comparing the effects of the three new plasticizers, the intersection of the 25 vol % binder line with the 25 wt % aluminum hydride line was selected on each of the appropriate ternary diagrams. The results were discussed in AFRPL-TR-65-195 and are summarized in table I.

(C) In view of the significant effect of plasticizer level on physical properties, as contrasted to the relatively minor effect of plasticizer level on specific impulse, it has been decided to maintain a plasticizer level of 50 wt % rather than the 55 wt % used in the previous program. Further, processing studies have resulted in a capability of reducing the binder level to 23 vol % with consequent modest improvements in theoretical performance and density. As a result of these changes, a further theoretical comparison was made of the effects of these plasticizers on performance at 50 wt % plasticizer, 23 vol % binder, and 25 wt % aluminum hydride. The results are compared in table II. As an additional point of comparison, the effect of nitroglycerine is also presented.

(U) Preliminary laboratory evaluation tests have been conducted with all of the candidate nitrate plasticizers. The data accumulated on BTTN was reported previously in the first quarterly report. Although BTTN was not miscible with HX-735 polymer above 50 wt % of plasticizer, some laboratory-scale mixes have been processed to further evaluate this plasticizer.

TABLE I

(C) 55% PLASTICIZER IN BINDER, 25 VOL % BINDER, 25 WT % ALUMINUM HYDRIDE

Plasticizer	Theoretical I_{sp}, sec	Improvement %	Theoretical Density lb/in.^{3}	Improvement %	O/F Ratio
TMETN	283	Control	0.0589	Control	1.35
BTTN	284	0.35	0.059	0.17	1.40
DNPN	284	0.35	0.059	0.17	1.40
TMNTN	284	0.35	0.0595	1.01	1.40

TABLE II

(C) 50% PLASTICIZER IN BINDER, 23 VOL % BINDER, 25 WT % ALUMINUM HYDRIDE

Plasticizer	Theoretical I_{sp}, sec	Improvement %	Theoretical Density lb/in.^{3}	Improvement %	O/F Ratio
TMETN	282.9	Control	0.0595	Control	1.38
BTTN	283.6	0.25	0.0596	0.17	1.42
DNPN	283.6	0.25	0.0596	0.17	1.42
TMNTN	283.8	0.31	0.0599	0.67	1.42
NG	283.1	0.07	0.0598	0.50	1.42

(U) The plasticizer TMNTN is apparently miscible with HX-735 polymer in all proportions. However, several small-scale mixes formulated with TMNTN at the 50 wt % plasticizer level did not cure. This sample of TMNTN may have a high acid analysis (0.002% according to the vendor). Additional TMNTN will be obtained from a different vendor.

(U) A DTA made on DNPN is reproduced in figure 4. An exotherm peak was observed at 185.3° C, but the trace returned to its original base line after this exotherm. The trace then became irregular, indicating some vaporization, and then boiling occurred at 233.4° C. No ignition occurred. The DNPN was subsequently mixed with HX-735 and curative in a polyethylene beaker. The polyethylene container was destroyed by heat generated from the mixture within a few minutes after mixing. Some further testing is required to establish the source of this problem.

2.2 DENSITY-I_{sp} TRADEOFFS

(C) This task is devoted to the improvement of both specific impulse and density in aluminum hydride systems. As a part of this task, two formulations will be chosen for evaluation in six 4-lb motors: the most dense formulation with a theoretical performance in excess of 274 sec and the

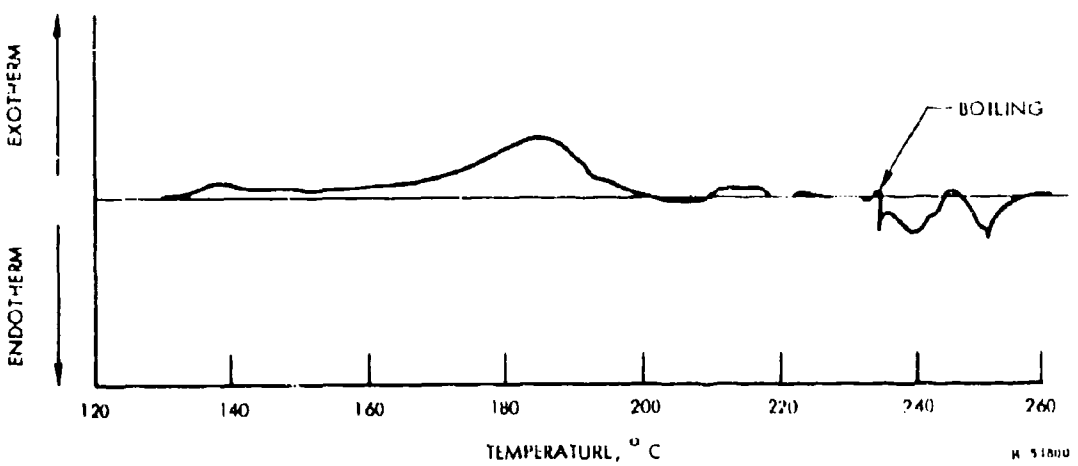

Figure 4. (U) DTA of 2,2-Dinitropropyl Nitrate

most dense formulation with a theoretical performance of 280 sec or above. One of these formulations will then be evaluated in three 10-lb motors.

(C) During the previous quarter, methods for increasing the density and specific impulse of aluminum hydride propellants were explored theoretically. Methods that might be used to increase the theoretical specific impulse of aluminum hydride propellants are: (1) the use of higher aluminum hydride loadings, (2) the use of advanced binders such as the NF_2 binders, (3) the improvement of the oxygenation of the CHON binder, (4) the increase of solids loading, and (5) the use of advanced oxidizers of greater oxygen content, density, or more favorable heat of formation.

(C) The density of aluminum hydride propellants may be increased by such measures as: (1) reduction of aluminum hydride content, (2) the use of a secondary metallic fuel in a mixed fuel system, (3) the reduction of binder content and or current solids loading increase, and (4) the use of more dense oxidizers resulting in increased solids loading.

(C) The use of increased aluminum hydride for increased performance is limited by the problem of combustion efficiency and two-phase flow losses. Extensive performance efficiency studies under Contract No. AF 04(611)-9570 indicate that levels above 25 wt % are probably not desirable. Calculations under Contract No. AF 04(611)-10540, "Evaluation of an Advanced Binder," and calculations on the present program indicate that substantial improvements in performance can be achieved by use of the PBEP difluoramino binder and by use of advanced oxidizers such as hydroxylamine perchlorate (HAP). However, experimentation on the present program is limited to nitrato-plasticized binders of more conventional analysis and to conventional oxidizers. In systems oxidized with ammonium perchlorate, the greatest improvements in density result from the use of mixed hydride/metal fuels.

(C) The tradeoffs between increasing density and decreasing performance were examined in systems containing mixtures of aluminum hydride and aluminum metal and in systems containing mixtures of aluminum hydride and zirconium metal. Generally, performance losses with increasing density were greater in the zirconium- than in the aluminum-containing systems.

(C) The effect of aluminum addition on I_{sp} and density is seen in figures 5 and 6 where aluminum analysis in the composition is held to 18.0% and to 22.5%, respectively. Two lines are shown for 25 vol % binder and 30 vol % binder to include the areas of maximum processability. In the compositions containing 18% metal, the first target impulse of 280 sec cannot be achieved. However, the second target of 274 sec can be achieved at between 4.0 and 5.0% aluminum metal with a resulting density between 0.0602 and 0.0613 lb/in.^{3}

(C) In the compositions in figure 6 where the aluminum analysis is 22.5%, the 280-sec target can be achieved at 2.75 and 3.75% metal with a resulting density between 0.0590 and 0.0597 lb/in.^{3} The secondary target of 274 sec can be achieved at a metal content of approximately 11.0% to yield a density of between 0.0620 and 0.0635 lb/in.^{3} From this it is apparent that the secondary target impulse in the 22.5% aluminum analysis system will have the best density.

(C) Examination of the effect of zirconium in the AlH_3/AP/PEP-155 system shows less favorable compositions. While the O/F ratios are more favorable than in the aluminum system by virtue of the lower number of equivalent weights of zirconium used, the performance appears to drop much faster with increasing density. This is borne out in the presentation in figure 7 in which specific impulse and density are plotted against the percent of zirconium in a system whose metal analysis is 22.5%. The primary target of 280 sec is achieved at 0.9 to 1.20% zirconium at a density of from 0.0589 to 0.0598 lb/in.^{3} The secondary target of 274 sec is achieved at a zirconium content of from 3.5 to 3.7% with a resulting density of from 0.0605 to 0.0612 lb/in.^{3} In each instance the densities achieved at the target impulse are less than the densities achieved in the aluminum/aluminum hydride system. For this reason, no experimental work with zirconium is planned.

(C) Processing studies have demonstrated that PEP/AP/AlH_3 propellant systems can be processed at 25 vol % binder. Therefore, two propellant formulations containing aluminum hydride/aluminum combinations were selected on the basis of figure 6 to yield maximum densities at theoretical I_{sp} values of 280 and 275 sec. These formulations are:

Formulation	UTP 6825	UTP 6826
PEP-155 binder	18.72	18.00
NH_4ClO_4	56.63	58.00
Al	3.65	9.75
AlH_3	21.00	14.25
Theoretical I_{sp}, sec	280.1	275.0
Theoretical Density, lb/in.	0.0604	0.0627

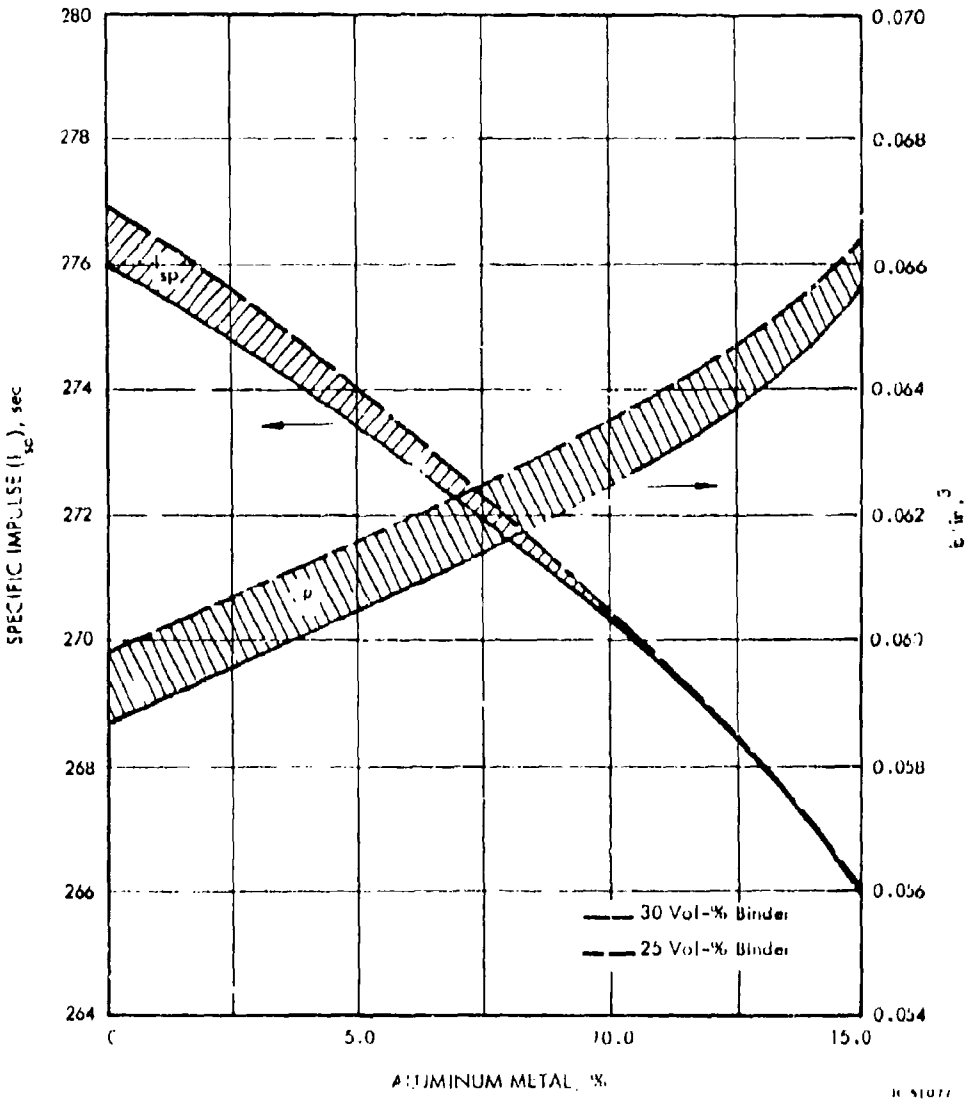

Figure 5. (U) Performance-Density Tradeoffs at Various Metal Loadings for a Constant 18.0 Wt % Aluminum Analysis

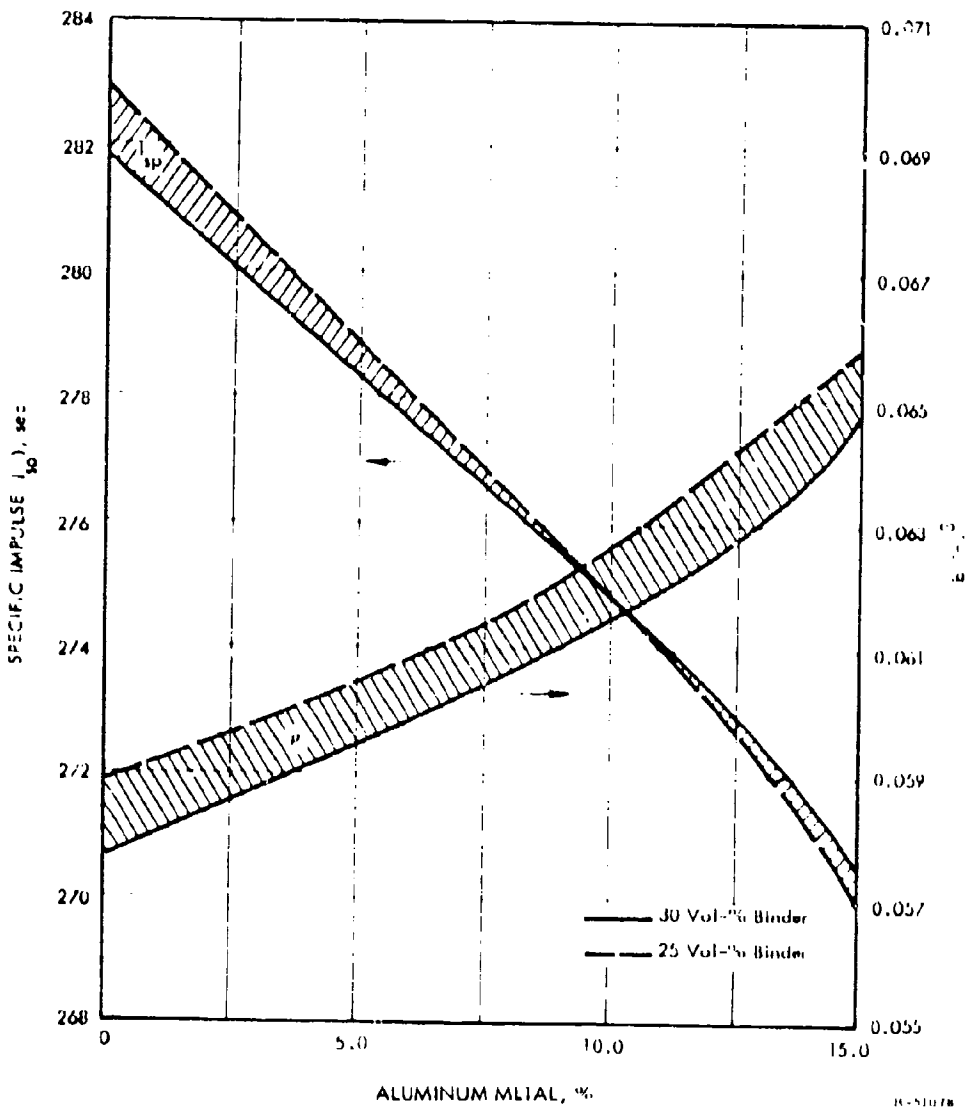

Figure 6. (U) Performance-Density Tradeoffs at Various Metal Loadings for a Constant 22.5 Wt % Aluminum Analysis

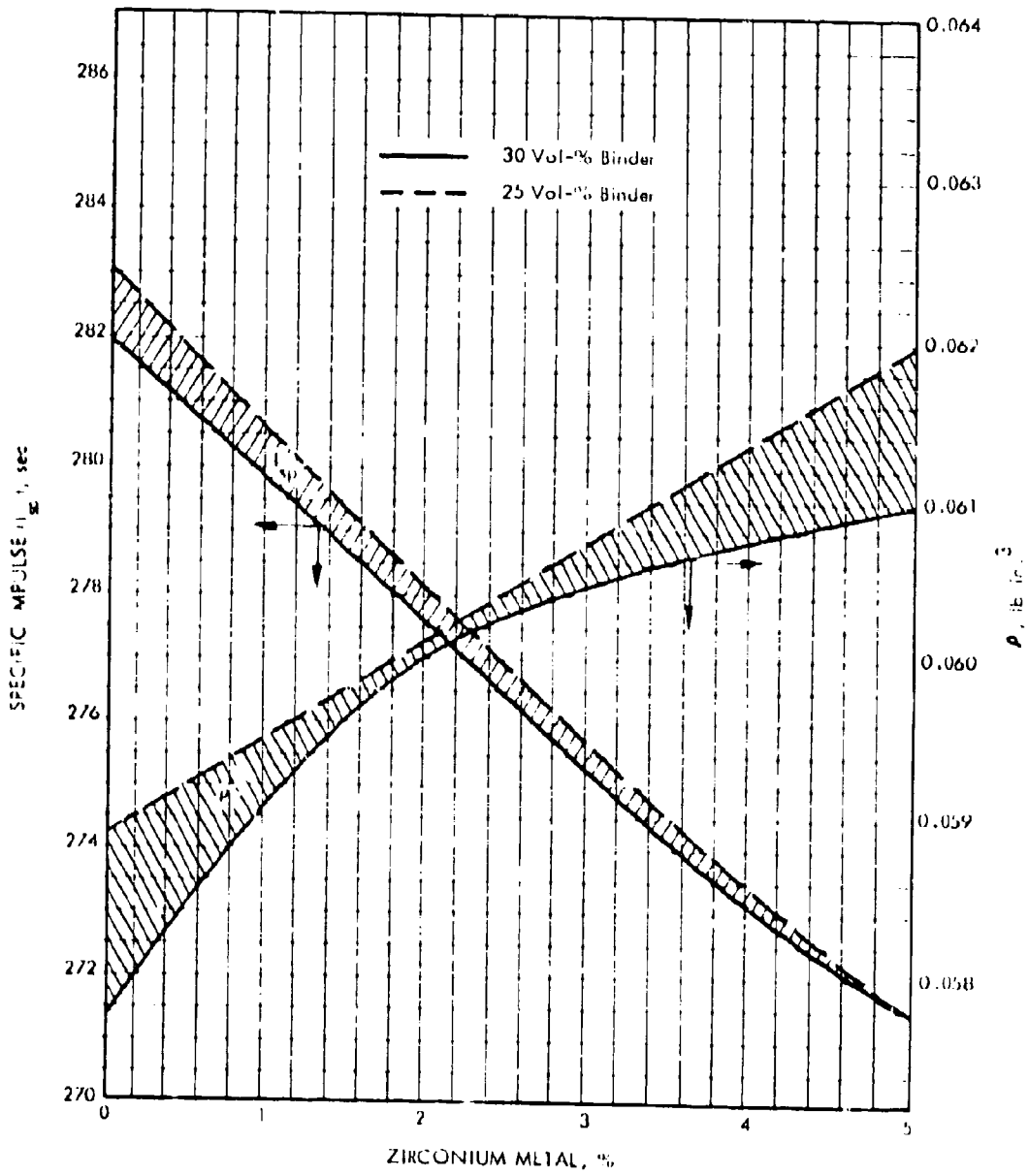

Figure 7. (U) Performance-Density Tradeoffs in Zirconium-AlH_3 Mixed Fuels

A total of four 4-lb motors of each formulation have been tested. The test results are presented in table III. The densities for these motors were below normal. Somewhat more gassing was evident in these motors, apparently because the aluminum powder had not been previously dried. Such a procedure would not normally be required except in cases where the aluminum and the aluminum hydride are processed together.

2.3 BURNING RATE STUDIES

(C) To make this propellant system adaptable to a wide range of potential missile systems, a study has been initiated to develop a spectrum of available burning rates. Target burning rates of 0.25 in./sec and 1.0 in./sec at 1000 psia have been established. Generally speaking, the higher burning rates in the range of about 0.4 to about 1.0 in./sec at 1000 psia offer no serious problems since a number of effective burning rate additives are available which can be varied in concentration sufficiently to achieve any burning rates in this range.

(C) Under Contract No. AF 04(611)-9270, propellant burning rates typically ranged from about 0.50 to about 0.7 in./sec for propellants containing no burning rate additives. Therefore, it was assumed that burning rates in the lower ranges, say 0.30 to 0.20 in./sec at 1000 psia would be somewhat more difficult to achieve. A systematic study was conducted to determine the effect of the normal propellant formulation parameters on burning rate prior to devoting any substantial effort to a study of burning rate catalysts or burning rate retardants.

(C) In the propellant system under study, the primary formulation parameters available for study include oxidizer (AP) concentration, PEP binder concentration, TMETN plasticizer concentration in the binder, and aluminum hydride concentration. For purposes of the burning rate investigation, the particle size distribution of the two solid ingredients, i.e., ammonium perchlorate and aluminum hydride, are added parameters. Since the effect of these last two parameters (particle sizes) can generally be predicted qualitatively, they do not offer any unusual problems and it remains merely to measure quantitatively the effect of AP and aluminum hydride particle sizes in this particular system.

(C) The four primary formulation parameters, AP, AlH_3, binder, and plasticizer level, are most conveniently treated in terms of binder volume concentration, O/F ratio, and plasticizer level. The O/F ratio essentially

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TABLE III

(C) DENSITY-I_{sp} TRADEOFF STUDIES, 4-LB MOTOR TESTS

Test No.	UTP No.	Motor and Propellant Parameters: Density lb/in³	Density % of Theor.	Motor Test Results: r_b in./sec	P_{c_b} psia	P_{c_a} psia	c^* ft/sec	Specific Impulse: I_{ca}	1000 θ*	P_{ca} θ*	Theoretical Calculations: Theor. I_{sp}, sec	Theor. c^* ft/sec	T_c °K	Efficiencies %: c^*	I_{sp}
233	6825	0.0538	89.1	*	*	1231.4	5280	245.[illegible]	[illegible]	249.4	284.4	5529	3517	95.5	87.7
234		0.0542	89.8	0.972	1078.6	964.9	5216	235.0	243.6	243.1	278.9	5520	3496	*	87.2
244		0.0582	96.4	0.899	1056.9	971.2	5279	243.1	247.8	247.2	278.9	5520	3496	*	88.6
246		0.0582	96.4	0.856	1012.1	946.9	5292	244.5	249.[illegible]	248.7	278.2	5520	3494	[illegible]	89.4
												Avg.		95.4	88.2
231	6826	0.0617	98.5	0.633	1346.7	1266.5	5118	244.5	244.4	248.7	273.7	5420	3663	94.4	88.9
232		0.0612	97.7	0.592	1081.8	1021.9	516[illegible]	236.4	240.2	240.4	274.7	5412	3641	95.4	87.5
240		0.0582	92.9	0.734	1182.4	1090.7	5348	236.7	238.9	240.7	277.3	5415	3648	98.8	86.8
242		0.0580	92.6	0.731	1089.2	1016.9	5185	234.4	237.9	238.4	275.5	5412	3640	95.8	86.5
												Avg.		96.1	87.4
239	5001	0.0639	99.7	0.338	940.1	919.2	5245	225.4	230.8	229.2	259.7	5178	3403	97.4	88.3
241		0.0635	99.1	0.345	1027.1	1000.5	5155	232.4	236.2	236.4	261.5	5179	3409	99.5	90.3
243		0.0641	100.0	0.342	1025.9	997.2	5101	217.6	221.2	221.3	261.5	5179	3409	98.5	84.6
245		0.0642	100.2	0.329	989.3	964.1	5202	236.1	240.7	240.1	260.8	5178	3406	[illegible]	92.1
												Avg.		99.0	88.8

* Long tailoff, no r_b

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represents a tradeoff between aluminum hydride and ammonium perchlorate at a given binder volume level. The following experimental plan was designed to explore these parameters in 1-in. micromotor tests:

BURNING RATE STUDIES EXPERIMENTAL PLAN

Variables

Binder volume levels:	25, 30, 32.5
O/F ratios:	1.0, 1.2, 1.4
Plasticizer levels:	30, 40, 50, 60
AP grind ratio:	Four ratios
LMH-1 size:	Two fractions

Constants	Variables	No. Mixes
30% binder (PEP-150) 50:50 AP ratio	O/F: 1.0, 1.2, 1.4	3
30% binder, 1.2 O/F ratio, 50:50 AP ratio	Plasticizer: 30, 40, 50, 60	4
PEP-150, 1.2 O/F ratio, 50:50 AP ratio	Binder volume: 25, 30, 32.5%	3
30% binder (PEP-150), 1.2 O/F ratio	AP ratio (coarse/fine): 60/40, 50/50, 35/65, 20/80	4
30% binder (PEP-150), 1.2 O/F ratio	Screen LMH-1 into two fractions	2
		16

(C) Most of the mixes required for this experiment have been processed and tested with the exception of the two designed to explore the effect of aluminum hydride particle size. One mix (UTX 6847) at the 50% plasticizer level was not castable and is being repeated. In addition, the test results for UTX 6846 at the 40% plasticizer level appear to be anomalous and this mix will also be repeated. Table IV summarizes the formulations tested and the test results.

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TABLE IV

(C) BURNING RATE STUDY (MICROMOTOR TEST RESULTS)

Formulation	Volume % Binder	Plasticizer Level	O/F Ratio	AP coarse/fine	Weight % Binder	Weight % AP	Weight % LMH-1	r_c	P_c
6842-1 r_{1000} = 0.301	30	50	1.0	50/50	23.5	48.5	28.0	0.274 0.273 0.303 0.255 0.266	at 667 at 752 at 1035 at 530 at 444
6843-1 r_{1000} = 0.320	30	50	1.2	50/50	23.5	52.0	24.5	0.284 0.275 0.296	at 601 at 609 at 745
6844-1 r_{1000} = 0.38	30	50	1.4	50/50	23.0	55.0	22.0	0.385 0.35 [illegible] 0.337	at 1019 at 818 at 735 at 532
6845-1 r_{1000} = 0.40	30	30	1.2	50/50	22.2	55.2	22.6	0.342 0.386 0.50 0.347	at 610 at 935 at [illegible] at 611
6846-1 r_{1000} = 027	30	40	1.2	50/50	22.7	54.0	23.2	0.241 0.244 0.260 0.270	at 476 at 642 at 837 at 955
6848-1 r_{1000} = 0.33	25	50	1.2	50/50	19.2	54.1	26.7	0.297 0.317 0.314 0.303	at 661 at 745 at 820 at 728
6849-1 r_{1000} = C_p32	32.5	50	1.2	50/50	25.8	51.0	23.2	0.289 0.340 0.321	at 545 at 759 at 1042
6850-1 r_{1000} = 0.31	30	50	1.2	60/40	23.5	52.0	24.5	0.260 0.282 0.296	at 702 at 839 at 389
684[illegible]	30	60	1.2	50/50	24.1	49.4	26.5	To be repeated. Mix was not castable.	
6850-[illegible] r_{1000} = 0.46	30	50	1.[illegible]	20/80	23.5	52.0	24.5	0.408 0.443 0.540 0.444	at 771 at 96[illegible] at 1385 at 443
6850-2 r_{1000} = 0.40	30	50	1.2	35/65	23.[illegible]	52.0	24.5	0.357 0.386 0.42	at 745 at 920 at 1140

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(U) The effect of binder concentration on burning rate in terms of volume percent binder is illustrated in figure 8. The binder concentration within the range of interest appears to have essentially no effect on burning rate. The effect of O/F ratio is significant as indicated by figure 8. An increase in O/F ratio from 1.0 to 1.4 results in an increase in burning rate of over 25%.

(C) Figure 8 presents the available data on the effect of plasticizer loading at constant O/F ratio. The low burning rate at 40% TMETN is apparently in error since it also f[illegible] substantially out of the curves in figure 9 in which burning rate is tra[illegible]d as a function of weight percent AP and weight percent aluminum hydride On the basis of the limited data available for this parameter, it appears that burning rate decreases as t[illegible] loading of TMETN increases at a constant O/F ratio.

(C) This interpretation is consistent with the data presented in figure 9, in which all of the formulations (at the same AP grind ratio) are treated without regard for O/F ratio, plasticizer loading, or binder concentration. There is a strong trend toward higher burning rates at increased AP concentration or reduced rates at increasing aluminum hydride concentration. There is obviously some interaction between the effects of AP concentration, per se, and the effect of O/F ratio. The separate effects of these two parameters may be somewhat easier to separate after the rest of the mixes treating TMETN level at a constant O/F ratio have been tested.

(C) These findings offer an approach to meeting one of the objectives of the study, a burning rate of 0.25 in./sec at 1000 psia. If the burning rate is primarily a function of AP concentration, it should be possible to reduce AP and increase the TMETN level at a constant O/F ratio (say 1.2) and achieve reduced burning rates with no significant loss in performance although at a sacrifice in mechanical properties.

(C) Figure 10 illustrates the effect of oxidizer particle size distribution. The coarse fraction of oxidizer has an average particle diameter above 300 μ. The fine fraction has an average particle diameter of 8 to 10 μ. Within the range of coarse/fine ratios tested, the burning rate v[illegible]ried from 0.31 in./sec to 0.40 in./sec. Some additional testing is planned to explore the effect of increased concentrations of coarse oxidizer on burning rate. It appears from the shape of the curve in figure 10 that a further reduction in fine AP below that represented will not result in further substantial reductions in burning rate. Further increases in the "fine" fraction of AP would no doubt result in increased burning rates since such an approach has been amply demonstrated in other propellant systems. In the propellant system being developed on the present program, however, any substantial increase in the "fine" AP fraction would seriously jeopardize the processing capabilities of the system and, to a lesser extent, the mechanical properties also.

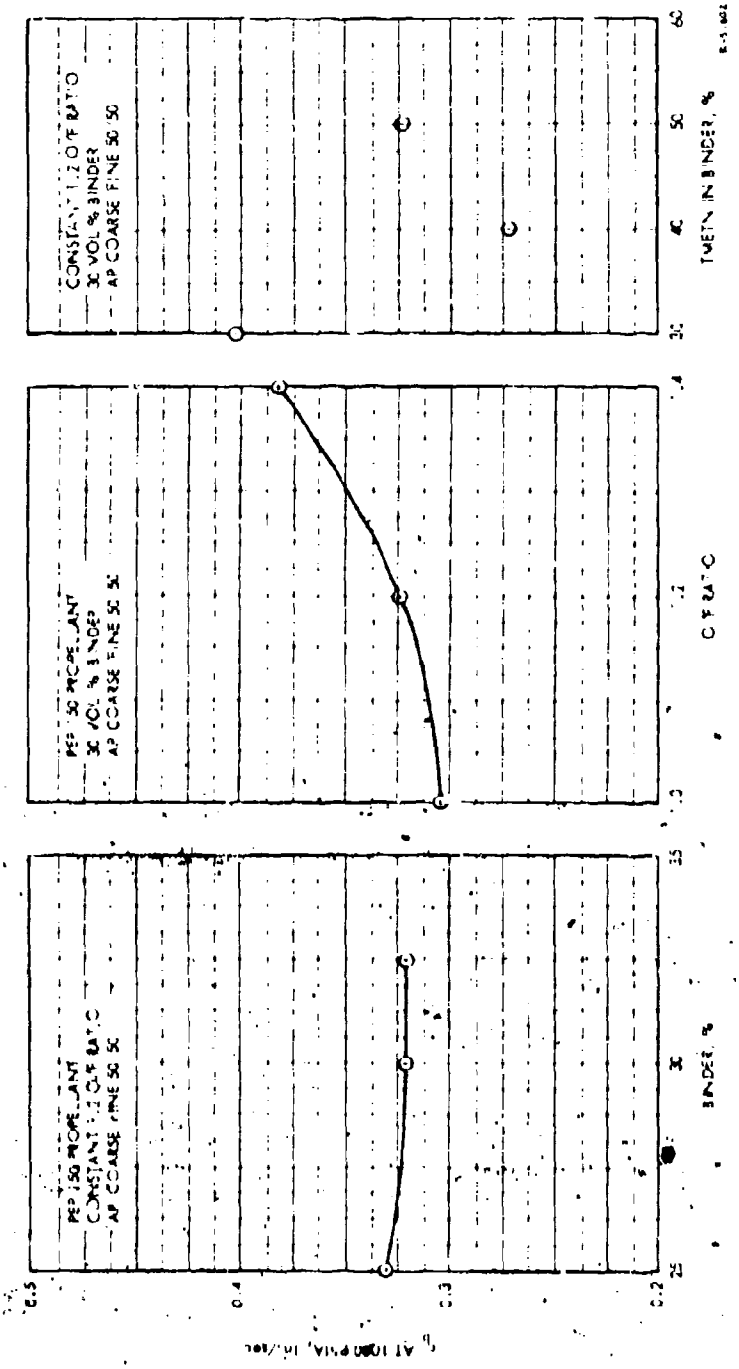

Figure 5. (U) Effect of Binder Concentration, O/F Ratio, and Plasticizer Loading on Burning Rate

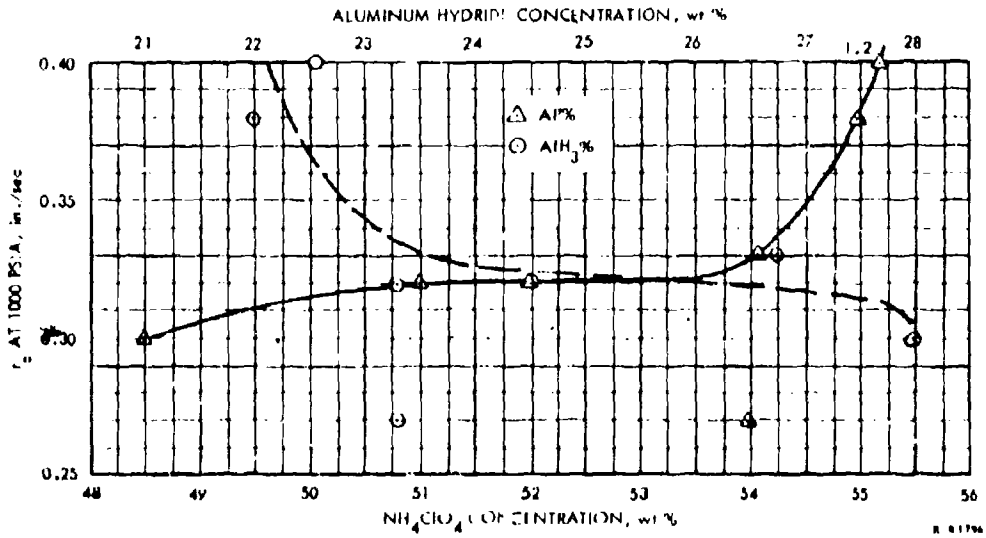

Figure 9. (U) Effect of NH_4ClO_4 and AlH_3 Concentrations on Burning Rate

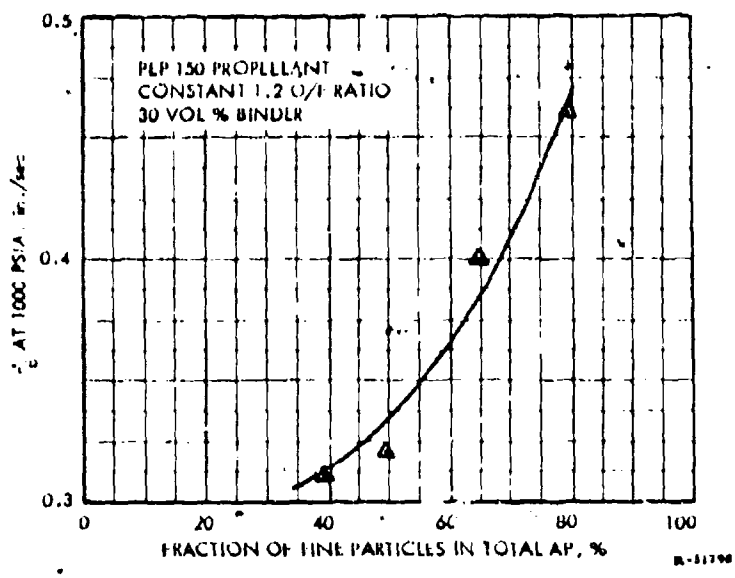

Figure 10. (U) Effect of AP Particle Size Distribution on Burning Rate

(C) Since the effect of the various formulation parameters on propellant burning rate have been defined, the next step in this study will involve defining the approximate upper and lower limits of the burning rate spectrum. On the basis of the test data obtained so far and the range of formulation parameters being studied, it appears that all of the burning rate objectives can be met without reducing the theoretical specific impulse below 280 sec.

(C) Some studies have also been conducted to determine the effect of grinding aluminum hydride. The hydride was ground for different time periods in a ball mill to reduce particle sizes and mixed in UTX 6814 formulations. The immediate goal was to determine the effect of grinding on processability and cured propellant density. The ultimate goal is to increase the fraction of coarse AP and reduce the fraction of fine oxidizer for the purpose of achieving reduced burning rates.

(C) The experimental data are summarized in table V. The propellant viscosities were directly proportional to grinding time. As grinding times increased, higher propellant viscosities resulted. Propellant densities in which the aluminum hydride was "wet ground" in acrylonitrile had densities corresponding to time of treatment as the hydride was not further surface treated after grinding. The hydride in batches 120 and 121 was "dry ground" and then acrylonitrile treated for 17 hr prior to drying. The densities in these two mixes were proportional to grinding them. The longer the grinding period, the lower the propellant density.

TABLE V

(C) EFFECT OF GRINDING VARIABLES ON PROPELLANT MIX VISCOSITY AND DENSITY*

UTX 6814	Grind Time min	Viscosity poises	Density g/cc	% Theoretical Density
116	0	1800	1.420	89.5
117	15 in AN	1800	1.402	88.3
118	30 in AN	5500	1.416	89.2
119	60 in AN	9400	1.516	95.5
120	15 dry	1200	1.457	91.8
121	30 dry	4200	1.440	90.7

* Dowane 1451 mixed in formulation UTX 6814

(C) Considerable difficulty was experienced on previous programs in obtaining reliable strand burning rate data because of the problems associated with obtaining a satisfactory coating on the strands. Many of the coating materials either reacted with the aluminum hydride or could not be cured at the low temperatures required. An end-burning micromotor has been designed and constructed for measuring propellant burning rates on this program. On the basis of micromotor tests and 4-lb motor tests from the same mixes, there is no measurable difference in burning rates obtained from micromotors or 4-lb motors. The micromotor design is illustrated in figures 11 and 12.

2.4 IMPROVEMENT OF SAFETY PROPERTIES

2.4.1 Conductivity Experiments

(C) Efforts were continued this period to develop methods for minimizing the hazards inherent in handling aluminum hydride. The primary approach being investigated at this time is to increase the electrical conductivity of the Dowane 1451 particles. Previous attempts to coat the particles with graphite by mechanical tumbling and by solvent dispersion did not result in a successful coating; that is, the measured electrical resistance of a bed of Dowane 1451 particles was not reduced as a result of the attempted coating.

(U) Two other materials have been investigated for their effect in increasing the electrical conductivity of the Dowane 1451 particles. One material is a commercial antistatic spray with the trade name Statikill. The other material is Catanac SN antistatic agent (stearamidopropyldimethyl-B-hydroxethylammonium nitrate) manufactured by American Cyanamid. These materials were evaluated as 1% coating on the Dowane 1451. The coated particles were tested in an electrical conductivity cell which consists of two 1-in. brass plates held 0.25 in. apart by a plexiglass spacer. The cell is shown in figure 13. A vacuum tube volt-ohmeter (RCA Senior Volt Ohmyst) was used to read the electrical resistance of the sample. The following results were obtained on the materials tested:

Sample	Electrical Resistance megohms
Pure Dowane 1451	1000
Dowane 1451, 1% graphite coating	1000
Dowane 1451, 1% Statikill coating	40 to 70
Dowane 1451, 1% Catanac SN coating	750

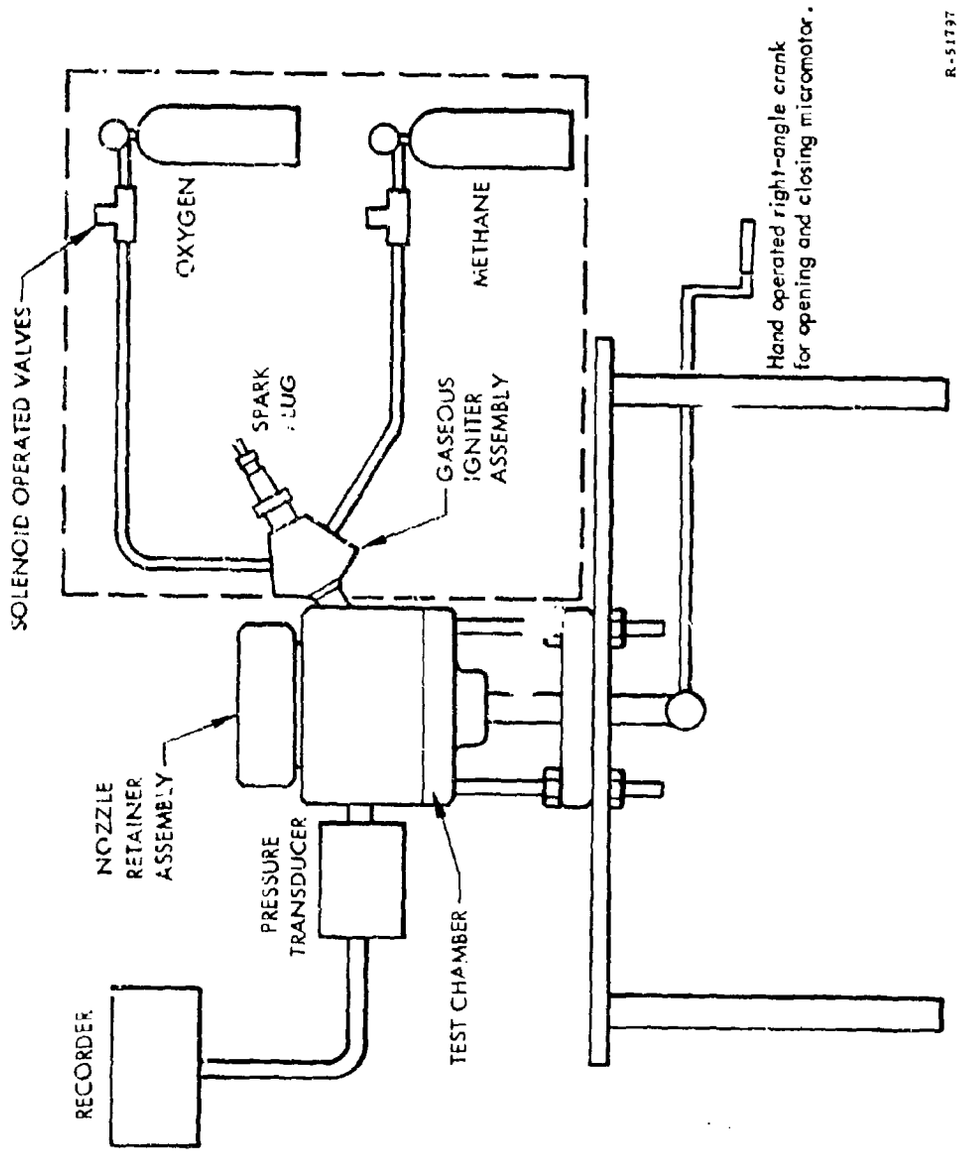

Figure 11. (U) Micromotor Test System for Burning Rate Studies

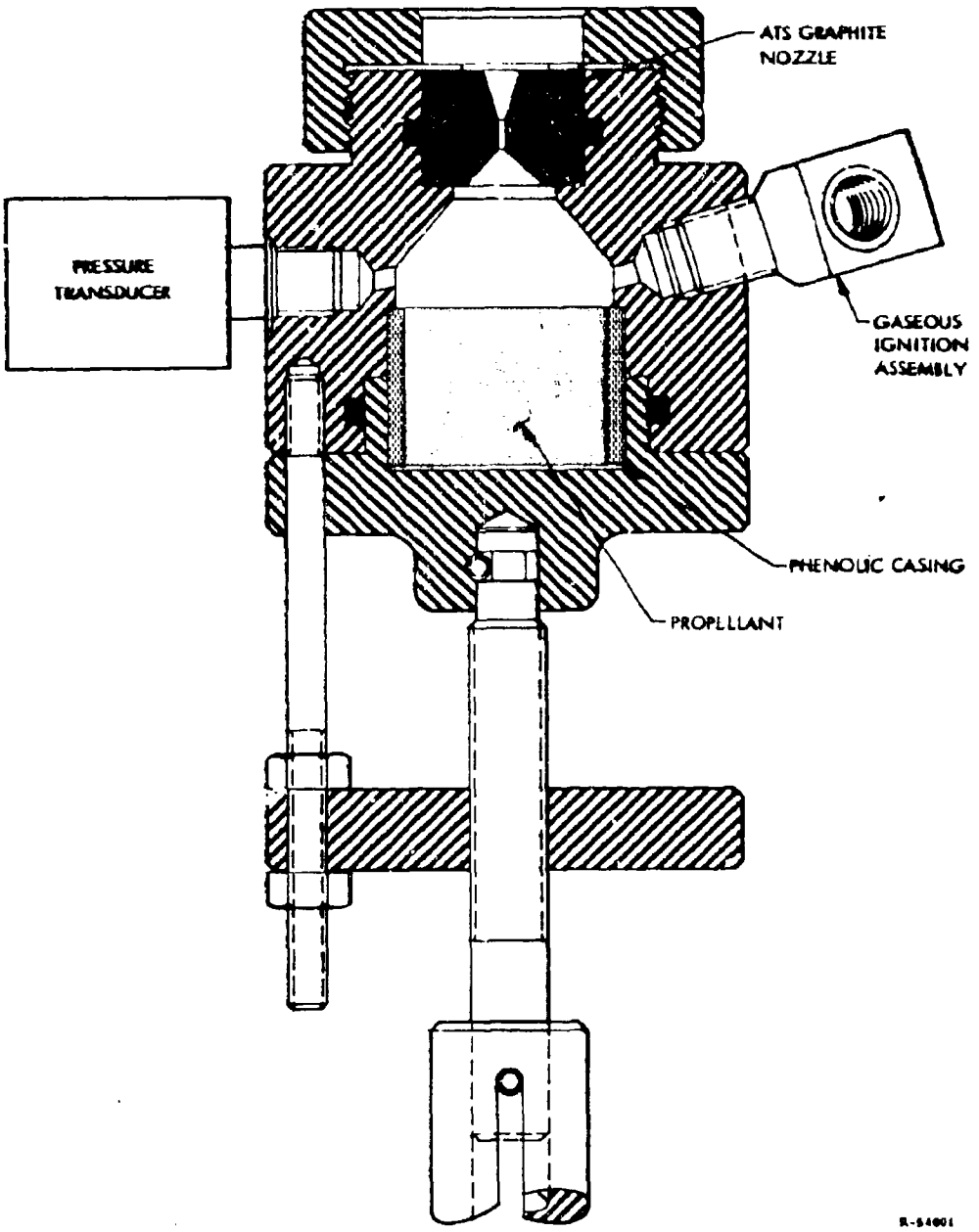

Figure 12. (U) Micromotor Chamber

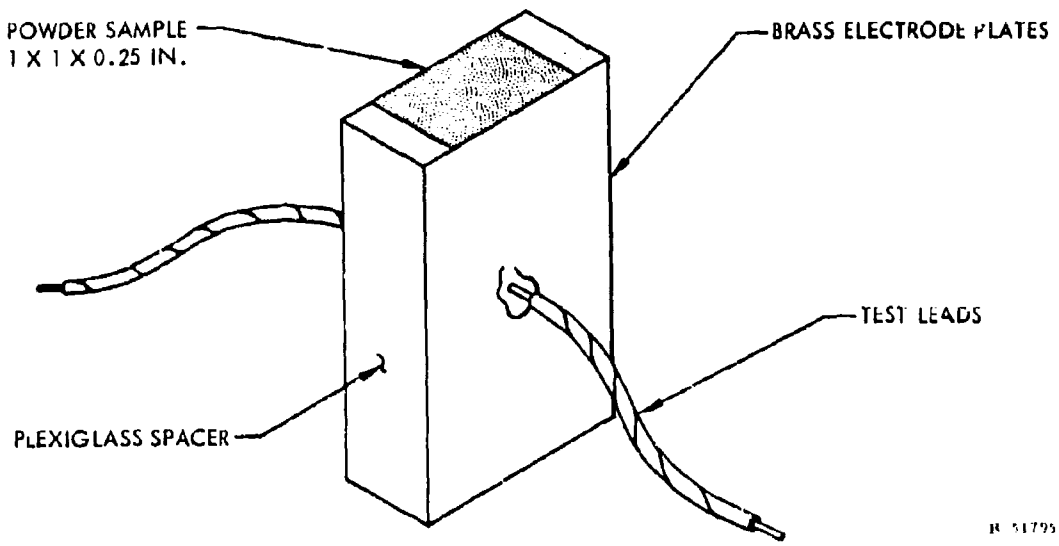

Figure 13. (U) Electrical Conductivity Cell

(U) So far, the Statikill is the only material sufficiently effective to warrant further study. It is planned to evaluate several surfactants which have been shown to be effective in improving processability of the propellant.

2.4.2 Impact Sensitivity Testing

(C) In connection with the burning rate studies described in section 2.3, a series of impact tests were conducted on uncured propellant samples which represented variations in O/F ratio, binder level, and plasticizer level. The data are presented in table VI. Impact sensitivity values for the uncured specimens ranged from 7.3 to 9.4 kg-cm. The most sensitive specimen in this series (UTX 6848) represented the lower binder level, 25 vol %. Neither the concentration of TMETN in the binder nor the O/F ratio of the propellant appeared to have any effect on propellant sensitivity within the ranges tested. There is also no apparent effect due to the individual ingredients, ammonium perchlorate or Dowanol 1451.

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TABLE VI

(C) IMPACT SENSITIVITY OF UNCURED PROPELLANTS

UTX No.	Vol % Binder	TMETN Loading	O/F Ratio	Wt % AP	Wt % Dowane 1451	Impact Sensitivity kg-cm
6842-1	30	50	1.0	48.5	28	9.3
6844-1	30	50	1.4	55.0	22	9.4
6845-1	30	30	1.2	55.2	22.6	8.6
6846-1	30	40	1.2	54	23.3	8.6
6847-1	30	50	1.2	49	26.5	8.2
6846-1	25	50	1.2	54.1	26.7	7.3

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2.5 AGING AND TEMPERATURE LIMITS

2.5.1 Formulation Improvement Studies

2.5.1.1 Effect of Purification of Ingredients on Propellant Density and Aging Properties

(C) One of the fundamental problems associated with the use of aluminum hydride containing propellants in operational motor systems is the limitation on storage times and storage temperatures. Another problem associated with the utilization of aluminum hydride for high-energy propellants is the lot-to-lot variation in the thermal and chemical stability of the material. The effect of thermal stability on the propellant properties has shown up as gassing during cure, resulting in a low-density propellant. The primary effect of the chemical reactivity has been shown in the gassing reactions that occur during cure, again resulting in a low-density propellant.

(C) Inasmuch as aluminum hydride is known to react with moisture, one approach to eliminating the gassing problem has been the evaluation of a rigorous procedure of drying and purifying all propellant ingredients prior to their utilization in the propellant. The HX-735 polyester was stripped of azelaic acid and moisture in a molecular still at 125° C and an absolute pressure of approximately 10 to 20 μ. The MAPO was vacuum distilled. The Epon 812 was diluted in methylene chloride, dried over molecular sieves for 48 hr, and then the supernatant liquid was decanted off and the solvent vacuum stripped. The AP was dried for a minimum of 24 hr in a vacuum oven at 190° F. All materials after purification were kept in a dry box with a relative dew point of -30° C or below. All weighings were done in the dry box and subsequent exposure to the atmosphere was kept to a minimum. In table VII, a comparison of batch 116, which was prepared by the standard procedure previously used, with batch 112, in which all ingredients have been purified, indicates an improvement in density from 89.5% to 95.6% of the theoretical value. A systematic substitution of ingredients one by one was made, starting with batch 106, to determine which of the ingredients was most critical. One ingredient at a time was used in the as-received condition and a subsequent propellant density determined. In this series of mixes, a blend of several lots of aluminum hydride was utilized to simulate the conditions existing in the scale-up facilities. This particular blended material contained lots of Dowane 1451 which are considered marginal. In this test series, the density of batch 108 indicated that the utilization of Epon 812 "as-received" had the most deleterious effect on the propellant density. However, when the effect of Epon 812 was examined with a lot of very good hydride, DL-482, no effect on the propellant density was noted, as is shown in batches 124 and 138 in table VII. Treatment of TMETN had the least effect on propellant density. The ether extraction

TABLE VII

(U) PURIFIED INGREDIENT DATA
BL 28 UTC AN

UTX 6814	Formulation Variable	Shore A	Cure Time days	Density g/cc	% Theoretical Density
106	HX-735 A. R., remainder purified	56	4	1.486	93.6
107	MAPO A. R., remainder purified	60	4	1.504	94.8
108	EPON 812 A. R., remainder purified	60	4	1.461	92.0
109	TMETN A. R., remainder purified	52	3	1.535	98.0
110	TMETN + M. S., remainder purified	52	3	1.513	95.3
111	TMETN + 1% E. C., remainder purified	60	3	1.526	96.1
113	AP oven dried, remainder purified	50	3	1.507	94.9
116	A. R. binder, AP oven dried, BL-28 UTC AN	46	5	1.420	89.5
112	All ingredients purified (control)	45	3	1.518	95.6
129	A. R. binder, BL 28, 3-day SOX Et_2O, oven dried AP	58	4	1.492	94.0
124	Purified ingredient, (DL-482)	69	4	1.568	98.8
138	EPON 812 A. R., (DL-482) remainder purified	72	4	1.574	99.2

procedure (using a soxhlet extractor) was again evaluated as a stabilization technique for aluminum hydride in mix 129. A significant improvement in propellant density was obtained utilizing "as-received" binder over AP with the soxhlet ether-treated aluminum hydride as compared to that treated with acrylonitrile. Further work is planned on this procedure utilizing all purified ingredients to see if a further density improvement can be obtained by using purified ingredients with soxhlet-treated Dowane 1451.

2.5.1.2 Vacuum Cure Effects

(C) The most severe gassing of the propellant occurs during the first 24 hr of cure. Accordingly, a series of experiments were conducted to determine the effect of curing the propellant under vacuum or of subjecting the mixed propellant to a vacuum prior to cure. The results of this study are shown in table VIII. Batch 123 is the control for this study. A propellant density of 98.2% of theoretical was obtained in the control. Aluminum hydride lot DL-482, which had been treated by Dow with wet acrylonitrile (0.3% water), was used in this test series. In batch 135, duplicate samples were degassed at 1, 2, 3, and 4 days, respectively, under static vacuum at 120° F. The same degassing periods were used with batch 136 only at ambient temperature. All samples were cured for a total of 4 days at 120° F. For example, sample 135A was cured under static vacuum at 120° F for 1 day followed by 3 days ambient pressure and cured. Sample 136A was cured under ambient temperature and static vacuum for 1 day followed by 4 days cured at ambient pressure. A slight improvement in density was obtained with the 1- and 2-day vacuum treatment at 120° F, but a reduction in density occurred on the third and fourth day. This is not surprising since the effect of vacuum during gelation should result in a swelling of the propellant. The vacuum conditioning of ambient temperature samples, however, gave reproducibility high densities. On all four samples, densities from 99.9 to 100.3% of theoretical were obtained. It is apparent that at ambient temperature, no cure occurs in the 6814 formulation in this time period. Therefore, when the sample was placed in the 120° F oven for cure after ambient degassing, the propellant retained enough fluidity to reconsolidate itself.

(U) Since the initial study here had been with a lot of Dowane 1451 that already exhibited good properties, a check was made on the effect of this technique with blend 28. A modification made to the procedure here, however, was to leave the mixed propellant in the mixer under vacuum for approximately 17 hr followed by a 1/2-hr mix cycle and then casting. It can be seen that with this lot of hydride and this particular technique, no gain in density was obtained.

TABLE VIII

(U) VACUUM CURE DATA; DL-482 (AN TREATED BY DOW)

UTX 6814	Variable	Density g/cc	% Theoretical Density
122	A.R. ingredient	1.529	96.3
123	Purified ingredient, control	1.559	98.2
135A	One day 120° F + vacuum purified ingredient	1.567	98.7
135B	Two days 120° F + vacuum purified ingredient	1.571	99.0
135C	Three days 120° F + vacuum purified ingredient	1.542	97.1
135D	Four days 120° F + vacuum purified ingredient	1.523	95.9
136A	One day ambient + vacuum purified ingredient	1.545	99.9
136B	Two days ambient + vacuum purified ingredient	1.589	100.1
136C	Three days ambient + vacuum purified ingredient	1.589	100.1
136D	Four days ambient + vacuum purified ingredient	1.592	100.3
140	Purified ingredient	1.522	95.9
	BL-28 UTC AN		
	Left in mixer 17 hr, 80° F under vacuum		
112	Purified ingredient (control for batch 140)	1.518	95.6
	BL-28 UTC AN		

2.5.1.3 Surface Treatments

(C) Among the many techniques for improving the stability and compatibility of aluminum hydride, perhaps the most commonly used is that of a surface passivation obtained by immersing the hydride in a medium such as acrylonitrile. Advancements in this technique have been made by adding fractions of a percent of water to the acrylonitrile. The technique favored by Aerojet-General is the treatment of the hydride with hot, moist air. Currently it is believed that this passivation is occurring through the surface oxidation of the hydride. Dow Chemical Company has demonstrated that the Taliani stability of aluminum hydride is significantly improved by reacting aluminum hydride directly with water. Surface oxidations of the order of 0.3 to 1% yielded significant improvements in thermal stability.

(C) The basic reaction occurring in the water treatment is one of oxidation and reduction. A technique originally studied under Contract No. AF 04(611)-9570 by UTC for measuring the degree of reactivity of aluminum hydride was evaluated as a surface oxidation technique for improving the stability and compatibility of aluminum hydride. This approach involves reacting the Dowanee 1451 surface with the quinones, alizarin (1, 2-dihydroxyanthroquinone) and alizarin red S, the sodium alizarin sulfonate salt. The alizarin was dissolved in benzene and Dowane 1451 was added to the mixture. No immediate color change was noticed, but after several hours a red color was observed on the hydride surface. The exact reaction products are not currently known. It is assumed that a chelate was formed causing the red coloration on the Dowanee 1451 surface. Table IX is a summary of the experimental data. The difference between batches 130 and 139 was that the excess alizarin was removed in batch 139. It is interesting to note that the hydride with no excess alizarin had a red coated surface. This surface is not soluble in benzene and could not be removed from the hydride. It is possible, therefore, that an organic coating of the reduced alizarin has been formed by chelate formation with aluminum. To obtain the maximum effect from the alizarin treatment in batches 131 and 132, the aluminum hydride (lot DL-458) was ground in benzene with dissolved alizarin or alizarin red S in it. Such treatment results in exposure of fresh surfaces for reaction with the oxidizing agents. The biggest gain in density was obtained with the alizarin red S treated fuel. This batch did have a soft cure, however, as evidenced by a low Shore A rating, indicating the possibility of an interference with cure mechanism.

(C) To separate the effects of the surface oxidation obtained by chelate formation of the quinone and aluminum hydride from that of the subsequent reaction of chelate formation with the oxidized aluminum, another quinone, 2, 6-dibromo quinone chloroimide, is being evaluated. This material should react without any subsequent side reactions such as the chelate formation obtained with the alizarin.

TABLE IX

(U) ALIZARIN DATA

UTX 6814		Density g/cc	% Theoretical Density	Shore A
142	BL-28 A.R., A.R. ingredient	1.388	87.5	43
116	BL-28 UTC AN, A.R. ingredient	1.420	89.5	46
130	BL-28 + 1% alizarin, A.R. ingredient	1.503	94.7	52
139	BL-28 treated with alizarin with excess alizarin removed purified ingredient	1.476	93.0	83
89	DL-458, A.R. ingredient		92.0	
131	DL-458 + 1% alizarin, ground 15 min, A.R. ingredient	1.493	94.1	55
132	DL-458 + 1% alizarin red S, ground 15 min A.R. ingredient	1.555	98.0	35 soft cure

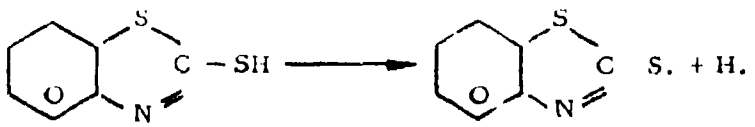
(C) To obtain the maximum effects of the surface treatment studies, all these reactions and mixes were run with "as-received" ingredients. Comparison batches of 116 and 142 shows that an improvement of only 2% in density was obtained by acrylonitrile treating the blend 28, whereas improvements up to a maximum of 98% of theoretical density were obtained through the alizarin studies. Further studies will, of course be conducted to thoroughly evaluate the use of these ingredients as stabilizing agents, as well as their effect on the long-term stability and aging characteristics of aluminum hydride propellant.

2.5.1.4 Free-Radical Inhibition

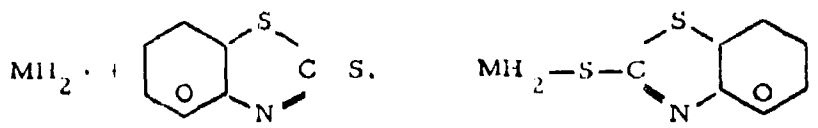
(C) A third approach to improvement in propellant density was the use of free-radical inhibitors. Olin-Mathieson and Dow Chemical Company have been experimenting with 2-mercaptobenzothiazole (MBT) and phenothiazine (PTA). They have added the above materials during and after

formation of aluminum hydride and propose that the following MBT-Dowane 1451 reactions occur:

A. Initiation: $AlH_3 \longrightarrow MH_2\cdot + H.$



B. Termination:



(C) The termination reaction would prevent the further decomposition of a Dowane 1451 polymer chain with a minimum of hydrogen gas involved. It was speculated that if this mechanism was correct, the aging characteristics of aluminum hydride propellant could be improved.

(C) Table X summarizes the experimental data of both PTA- and MBT-coated Dowane 1451 propellant. The fuels with lower weight coatings (1/2%) had higher densities than 1% coated materials. If the hydride was ground and treated simultaneously, marginal or no density improvements were obtained. Another part of this study was to add MBT directly to the binder instead of coating the hydride. Batch 141 contains added MBT and batch 124 contains no additional ingredients. Aluminum hydride lot DL-482 was acrylonitrile treated. A definite drop in density (4%) occurred when MBT was added to the mix containing this lot.

(U) Although some improvement in density may be obtained from the use of the two free-radical inhibitors tested, their use in all the propellants tested resulted in discoloration and poor cures. It is possible that other free-radical inhibitors may be found that will give a significant improvement to the stability of the propellant without affecting the propellant cure.

2.5.1.5 Cure Studies

(U) A minor phase of this study was to evaluate the effect of cure and casting conditions. The results of air curing versus nitrogen cure and the effect of vibration following casting are shown in table XI. Curing under

TABLE X

(U) FREE-RADICAL INHIBITOR DATA

UTX 6814	Formulation Variable	Density g/cc	% Theoretical Density
142	BL-28 A.R., A.R. binder	1.388	87.5
125	1/2% PTA + BL 28 AR, A.R. binder	1.461	92.0
126	1% PTA + BL 28 AR, A.R. binder	1.420	89.5
127	1/2% MBT + BL 28 AR, A.R. binder	1.472	92.7
128	1% MBT + BL 28 AR, A.R. binder	1.420	89.5
89	DL-458, A.R. binder		92
133	DL-458 + 1% PTA ground 15 min, A.R. binder	1.480	93.2
134	DL-458 + 1% MBT ground 15 min, A.R. binder	1.384	87.1
137	DL-458 + Sv. dried An. ground 15 min A.R. binder	1.464	92.2
141	DL-482, MBT added to purified binder (1% of binder)	1.495	94.2
124	DL-482, purified binder, control	1.568	98.8

TABLE XI

(U) EFFECT OF PROCESSING VARIABLES ON PROPELLANT DENSITY

UTX 14	Variable	Shore A	Cure Temperature days	Density g/cc	% Theoretical Density
112	All ingredients purified (control)	45	3	1.52	95.6
114A	Air cure, purified ingredients	46	3	1.53	96.1
114B	N_2 cure, purified ingredients	48	3	1.58	99.4
115A	No vibration, purified ingredients	45	3	1.54	96.2

nitrogen results in a significant density improvement. The data shown for batch 115B are questionable as the sample foamed over during the vacuum degassing step.

2.5.2 Testing and Evaluation

2.5.2.1 Static DTA Experiment

(U) Three micromotors of UTP 6814 composition were placed in storage at 50° C at relative humidity levels of 0, 50, and 90%, respectively. There was no thermal event recorded up to 1000 hr.

(U) The static DTA experiment is designed to detect any thermal event occurring within the propellant which might lead to spontaneous cook-off in stored motors. In the UTP 6814 formulation, such an event would be most likely to be caused by an accumulation of oxides of nitrogen produced by slow decomposition of the TMETN plasticizer in the binder.

(U) The samples tested in controlled humidity environments were suspended in 250-ml Dewar flasks over glycerine-water solutions of appropriate composition to give the desired humidity level (71 wt % glycerine for 50% relative burn, 29 wt % for 90% relative humidity*). The sample tested under anhydrous conditions was suspended over Drierite. An iron-Constantan thermocouple was imbedded in each sample, and the temperatures were read daily. A fourth thermocouple was inserted in an empty Dewar flask to serve as a reference.

(U) The samples were stored at temperatures ranging from 50° to 65° C for 1000 hr. Although deterioration of the propellant was evident, there was no thermal event recorded.

(C) The lack of a thermal event at these elevated temperatures does not represent conclusive evidence that an exotherm might not take place on longer storage at a lower temperature, since the visible deterioration at the higher temperature is probably due to decomposition of the hydride. This experiment will be repeated at 35° C, a temperature at which the hydride will be stable for a longer period of time.

2.5.2.2 Effect of Web Thickness on Stability

(U) Samples of UTP 6814 were cast in glass tubes of various lengths and stored at 50° C. The experiment did not yield meaningful data because rapid swelling accompanied by bond failure occurred within the first week. This experiment will be repeated at a lower temperature.

* International Critical Tables Vol. III

(U) Glass tubes of 1-in. ID and lengths of 1/2, 1, 2, and 3 in. were cast full of UTP 6814. Preliminary samples cast on glass slides showed that a satisfactory bond was formed between the propellant and clean glass. The first samples cast in glass tubes were stored at 50° C. Decomposition was rapid at this temperature, and the 2- and 3-in. samples showed bond failure due to excessive swelling within 1 wk. This experiment is being repeated at lower temperatures.

2.5.2.3 Effect of Temperature and Humidity

(U) Samples of UTP 6814 in the form of 2-in. cubes are being stored at various temperatures and humidities. The cube samples presently in storage are summarized in table XII.

(U) These samples have been in storage 1 wk at the present writing. Measurements will be made at weekly intervals.

TABLE XII

(U) TWO-IN. CUBE SAMPLES IN STORAGE

Relative Humidity %	Temperature		
	25° C	35° C	45° C
0	3 cubes*	3 cubes*	3 cubes*
50	2 cubes		
90	1 cube		

* All samples over 90% density, except where marked (*) to indicate one sample over 80% density.

2.6 IMPROVEMENT OF PHYSICAL PROPERTIES

(U) The objective of this task is to improve the mechanical properties of the propellant and to improve the stability of the mechanical properties. The mechanical properties of UTP 6814 developed under Contract No. AF 04(611)-9570 were in the range of 72-psi maximum load and 16.5% at maximum load. The objective of the current study is to obtain measured values of 100-psi maximum stress and 20% strain at maximum stress.

2.6.1 Evaluation of Curative Systems

(U) In the previous report results of an evaluation of candidate curative systems with a terminally carboxylated polyester (HX-735) in an unfilled binder system were presented. In addition, data were presented on Instron testing of aluminized propellants made with these curatives considered most promising as a result of the unfilled binder tests. All of the curatives tested were trifunctional imine type materials which were tested generally at four or more equivalent ratio levels and also in combination with other curatives. Those materials evaluated were NTEB, NTPB, HMAT, NC 1034, NC 1026L, MAM, MAES, HX-868, HX-858, HX-874, DGAP, and MAPO/Epon 812. It was concluded that none of the curatives evaluated were as effective as the MAPO/Epon 812 combination. The best mechanical properties were obtained on a MAPO/Epon 812 cured formulation, [illegible]X 7902. Crosshead data obtained on specimens of this formulation were [illegible] - 62 psi and e_m = 25%.

(U) It has been found that calcium hydroxide accelerates the cure of the UTX 7902 binder. A series of mixes were made to determine the effect of the addition of calcium hydroxide on the mechanical properties of the propellant. A summary of these data is presented in table XI. It was found that 0.05 wt % calcium hydroxide gave what was believed to be a satisfactory pot life for casting. Loadings in excess of this amount adversely affected the propellant castability. The data show that improved tensile strength occurs with the use of calcium hydroxide. When loadings in excess of 0.05 wt % are used, the elongation of the propellant is affected adversely.

(U) Nitrilotriethyl-β propylenminobutryate (NTPB) was tested in two aluminized PEP-155 propellant formulations to evaluate the effect of this imine curative in combination with MAPO and Epon 812. Test results are presented in table XIII. Good tensile strength and elongation values were observed. Since previous tests have shown cure reversion when NTPB was used alone, additional UTX 7942 propellant was prepared for accelerated aging tests.

TABLE XIII

(C) EFFECT OF VARIOUS CURATIVES ON PEP-155 PROPELLANTS*

Formulation UTX	Curative and Equivalents	Crosshead Stress at Maximum psi	Crosshead Strain at Maximum %
7902	MAPO (1.5) EPON 812 (0.7)	62	25
7938	MAPO (1.5) EPON 812 (0.7) $Ca(OH)_2$ 0.05 wt %	97	23
7945	MAPO (1.5) EPON 812 (0.7) $Ca(OH)_2$ 0.075 wt %	170	16
7939	MAPO (1.5) $CA(OH)_2$ 0.10 wt %	115	17
7942	MAPO (1.5) EPON 812 (0.7) NTPB (0.3)	139	22
7943	MAPO (1.5) EPON 812 (0.7) NTPB (0.5)	152	20
7934	HX-874 (1.4)	11	5
7935	HX-874 (1.5)	53	7
7936	HX-874 (1.05) EPON 812 (0.35)	98	13
7937	HX-874 (1.125) EPON 812 (0.375)	115	10

* Formulation:

16.0% PEP 155
16.0% Al
47.5% coarse AP (190 μ)
20.5% fine AP (6.5 μ)

(U) Formulation tests were made with a proprietary aziridine curative (HX-874) obtained from 3M Company. A summary of the mechanical property data is presented in table XIII. The addition of Epon 812 to the cure system did improve the tensile properties. However, the tested formulations did not give equivalent elongation values observed in other propellant systems.

(U) Another recently acquired curative (TBM) is a trifunctional aziridine, tris-(2-ethylaziridinyl)-s-triazine. This curative was tested by itself and also in blends with other curatives. A summary of these tests is presented in table XIV. The use of TBM in combination with MAPO and Epon 812 results in an apparent improvement in mechanical properties. These properties should be amenable to further improvement by optimizing curative ratios. Furthermore, cured samples of UTX 7578 and UTX 7579 have been stored 10 wk at 160° F and no reversion has been observed.

(U) A review of the data has been made on all of the propellants processed with the new curatives used in the current program. The best mechanical properties have been from UTX 7902, UTX 7942, and UTX 7592. The formulations are based on the following combinations of curatives, MAPO/Epon, MAPO/Epon/NTPB, and MAPO/Epon/TBM. The curative systems are being tested to determine storage stability. These formulations were tested at the end of 1 wk cure at 120° F and then stored at 140° F. The stored propellant is being sampled at weekly intervals for stress and strain properties.

(C) Since there were some differences in the volume percent binder used in the preliminary propellant tests the binder content was adjusted to 24 vol % for all of the propellant storage specimens. The adjusted formulations are identified by the designations UTX 7700, UTX 7701, and UTX 7702. These formulations are similar to UTX 7942, UTX 7902, and UTX 7592. Table XV presents the three formulations that are in the accelerated aging program and the mechanical properties available at this time. These are aluminized propellants. Following initial evaluation in the PEP-Al system, limited evaluation of TBM in an aluminum hydride system was initiated. These data are presented in table XVI. Propellant UTX 6834 essentially represents what will probably be the final propellant selection for this program in terms of total solids loading and in terms of plasticizer level in the binder. Measured values of S_m = 104 psi and e_m = 6.8% indicate that further tailoring of the curative system and perhaps the polymer is required.

TABLE XIV

(C) EFFECT OF TBM AND MIXTURES ON MECHANICAL PROPERTIES OF PEP-15' PROPELLANT*

Formulation UTX	Curative and Equivalents	Crosshead Stress at Maximum psi	Crosshead Strain at Maximum %
7577	TBM (1.3)	46	13
7578	TBM (1.25) EPON 812 (0.7)	90	16
7579	TBM (1.0) EPON 812 (0.55)	67	26
7580	TBM (1.25) HX-874 (0.7)	77	8
7581	TBM (1.0) XH-874 (0.55)	61	10
7584	TBM (1.25) NTPB (0.70)	61	11
7585	TBM (1.0) NTPB (0.55)	44	15
7586	TBM (1.25) UNOX 201 (0.70)	34	17
7587	TBM (1.00) UNOX 201 (0.55)	18	34
7588	TBM (0.50) MAPO (0.70) EPON 812 (0.7)	58	46
7589	TBM (0.60) MAPO (0.60) EPON 812 (0.70)	74	42
7590	TBM (0.40) MAPO (0.80) EPON 812 (0.70)	44	51
7591	TBM (0.3) MAPO (0.9) EPON 812 (0.7)	47	52

TABLE XIV

(C) EFFECT OF TEM AND MIXTURES UPON MECHANICAL PROPERTIES OF PEP-150 PROPELLANT* (Continued)

Formulat UTX	Curative and Equivalents	Crosshead Stress at Maximum psi	Crosshead Strain at Maximum %
7592	TBM (0.7) MAPO (0.5) EPON 812 (0.7)	83	41
7593	TBM (0.6) MAPO (0.6) EPON 812 (0.7)	49	45
7594	TBM (0.8) MAPO (0.4) EPON 812 (0.7)	76	32
7595	TBM (0.9) MAPO (0.3) EPON 812 (0.7)	78	31
7596	TBM (0.5) MAPO (1.0) EPON 812 (0.7)	77	37
7597	TBM (1.5) EPON 812 (0.5)	109	13
7598	TBM (1.75) EPON 812 (0.25)	112	11
7599	TBM (1.25) EPON 812 (0.75)	98	14
8200	TBM (1.50) EPON 812 (0.75)	119	11
8201	TBM (1.60) EPON 812 (0.75)	114	12

* Formulation:

25% Binder { 12.5% Polymer, 12.5% TMETN }

21% Al

32.4 Coarse AP (190 μ)

21.6 Fine AP (6.5 μ)

TABLE XV

(C) EVALUATION OF PROSPECTIVE IMPROVED PEP-150 BINDERS

Material	UTX 7700 Equivalent	UTX 7701 Equivalent	UTX 7702 Equivalent	Wt %
HX-735	1.0	1.0	1.0	8.80
MAPO	0.5	1.5	1.5	
TBM	0.7	-	-	
EPON 812	0.7	0.7	0.7	
NTPB	-	-	0.3	
TMETN				8.80
Aluminum				16.00
AP (as received)				45.78
AP (Ground)				19.62
Ethyl Centralite				1.00
Propellant cured 7 days at 120° F				
Stress at Maximum, psi				
Storage, wk at 140° F				
0	41	46	85	
1	97	78	186	
2	86	84	155	
Strain at Maximum, %				
Storage, wk at 140° F				
0	37	31	17	
1	18	22	14	
2	15	21	14	

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TABLE XVI

(C) PRELIMINARY EVALUATION OF TBM CURATIVE IN ALUMINUM HYDRIDE PROPELLANT

(Cure Temperature = 120° F)

Formulation No.*	Curative Equivalents	Crosshead S_m	Crosshead e_m	Measured S_m	Measured e_m	Cure Time days	Vol % Binder	Density g/cc	% Theoretical Density
UTX 6827-1 PEP-155	1.0 TBM 0.7 Epon 812	38.0	30.0	44.0	19.2	3	32	1.472	92.7
UTX 6829-1 PEP-155	1.5 TBM 0.7 Epon 812	88.0	14.8	95.0	8.4	3	32	1.524	96.0
UTX 6841-1** PEP-155	1.5 TBM 0.7 Epon 812	92.0	13.8	100.0	8.6	3	32	1.791	96.1
UTX 6834-1 PEP-150	All purified ingredients 0.9 TBM 0.1 MAPO 0.5 Epon 812	97.5	11.8	104	6.8	4	23	1.546	94.4

* 1.0 equivalent HX-7135 polymer in all mixes
** Aluminized mix

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2.6.2 New Polymers

(U) Two new polymers have been tested as prospective replacements for the currently used HX-735. These polymers are being used in an effort to improve the propellant mechanical properties.

(U) Both the polymers are neopentyl glycol azelaic acid polyesters supplied by Emery Industries. The first of these is identified as Emery 1025-9-R and has a molecular weight of 1900. The second is Emery 1025-94-R which has a molecular weight of 2950. Table XVII presents a summary of the formulations tested and the resulting mechanical properties. Each of the polymers appears to show some improvements in the mechanical properties. The improved elongation of the high molecular weight polymer (Emery 1025-94-R) and the good tensile strength of the low molecular weight polymer (Emery 1025-9-R) offer a method of mechanical property tailoring by blending the two polymers. Propellant mixes have been made to evaluate the effect of blending the two Emery polymers.

2.6.3 Effect of Polymer Treatment on Mechanical Properties

(U) The HX-735 polymer used in the PEP propellant binder system is frequently treated in a molecular still for the purpose of removing moisture and other low molecular weight materials which might prove to be reactive with aluminum hydride. It has been observed that as a result of this operation the equivalent weight of the polymer is usually raised about 10% and physical properties are considerably improved.

(U) A summary of the effect of operating variables involved in operation of the molecular still on HX-735 equivalent weight is shown in table XVIII. The effect on physical properties of propellant made from polymer subjected to the variations in treatment is shown in table XIX. An increase in tensile and Shore A hardness was obtained with the higher purity polymer. Undoubtedly some of this increase is due to the increased density of the propellant.

(C) The effect of adding back the azelaic acid to the stripped HX is shown in mix UTX 6812-2. A decrease in density was obtained indicating an interaction between the aluminum hydride and the azelaic acid.

2.6.4 Effect of TMETN Loading on Propellant Mechanical Properties

(U) A series of mixes was tested for the purpose of evaluating the effect of TMETN level in the binder on propellant mechanical properties. The results of these tests are presented in table XX and in figure 14. It is apparent that propellant stress values are quite sensitive to plasticizer level whereas the increase in strain capability with increasing plasticizer

TABLE XVII

(C) EVALUATION OF OTHER POLYMERS (ALUMINIZED PROPELLANT, 24 VOL % BINDER)

Formulation UTX	Binder Materials and Equivalents	Crosshead Data: Stress at Maximum psi	Crosshead Data: Strain at Maximum %
8226	Emery 1025-9R (1.0) TBM (1.5) EPON 812 (0.7)	91	19
8227	Emery 1025-94R (1.0) TBM (1.5) EPON 812 (0.7)	66	32

TABLE XVIII

(C) EFFECT OF MOLECULAR STILL STRIPPING ON HX 735 EQUIVALENT WEIGHT

HX 735 variable	Equivalent Weight
As received	874

Molecular Still Operating Variables	Equivalent Weight
No trap used	873
Hot trap used	892
Cold trap used	983

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TABLE XIX

(C) EFFECT OF POLYMER TREATMENT ON PROPELLANT MECHANICAL PROPERTIES

Formulation*	Variable	Crosshead S_m, psi	Crosshead E_m, %	Measured S_m, psi	Measured E_m, %	Cure Time days	Shore A Hardness	Density g/cc	% Theoretical Density
UTX 6814-144	All purified ingredients	79.5	18.1	89.0	11.0	4	70	1.510	95.1
UTX 6814-145	As received HX	65.0	17.9	72	11.1	4	65	1.456	91.7
UTX 6814-146	Cold finger not used on molecular still	86.0	17.2	94.5	12.5	4	70	1.518	95.6
UTX 6814-147	Hot water (115° C) circulated through finger trap of still	50.5	18.6	56.5	11.1	4	60	1.418	89.3
UTX 6832-2	0.09% azelaic acid added to stripped HX	40.5	21.2	46.0	13.0	4	50	1.394	87.8

* Lot 2 HX-735 used in all mixes.

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TABLE XX

(C) EFFECT OF TMETN ON PEP PROPELLANT MECHANICAL PROPERTIES

Formulation UTX	TMETN wt %	Stress at Maximum psi	Strain at Maximum %
7703	0	194	16
7704	10	165	14
7705	20	151	18
7706	30	134	19
7707	40	97	19
7701	50	86	22

Formulation

Material	Equivalent	Wt %
HX-735	1.0	17.60
MAPO	1.5	
EPON 812	0.7	
TMETN	As noted	
Aluminum		16.00
AP (as received)		45.78
AP (ground)		19.62
Ethyl Centralite		1.00

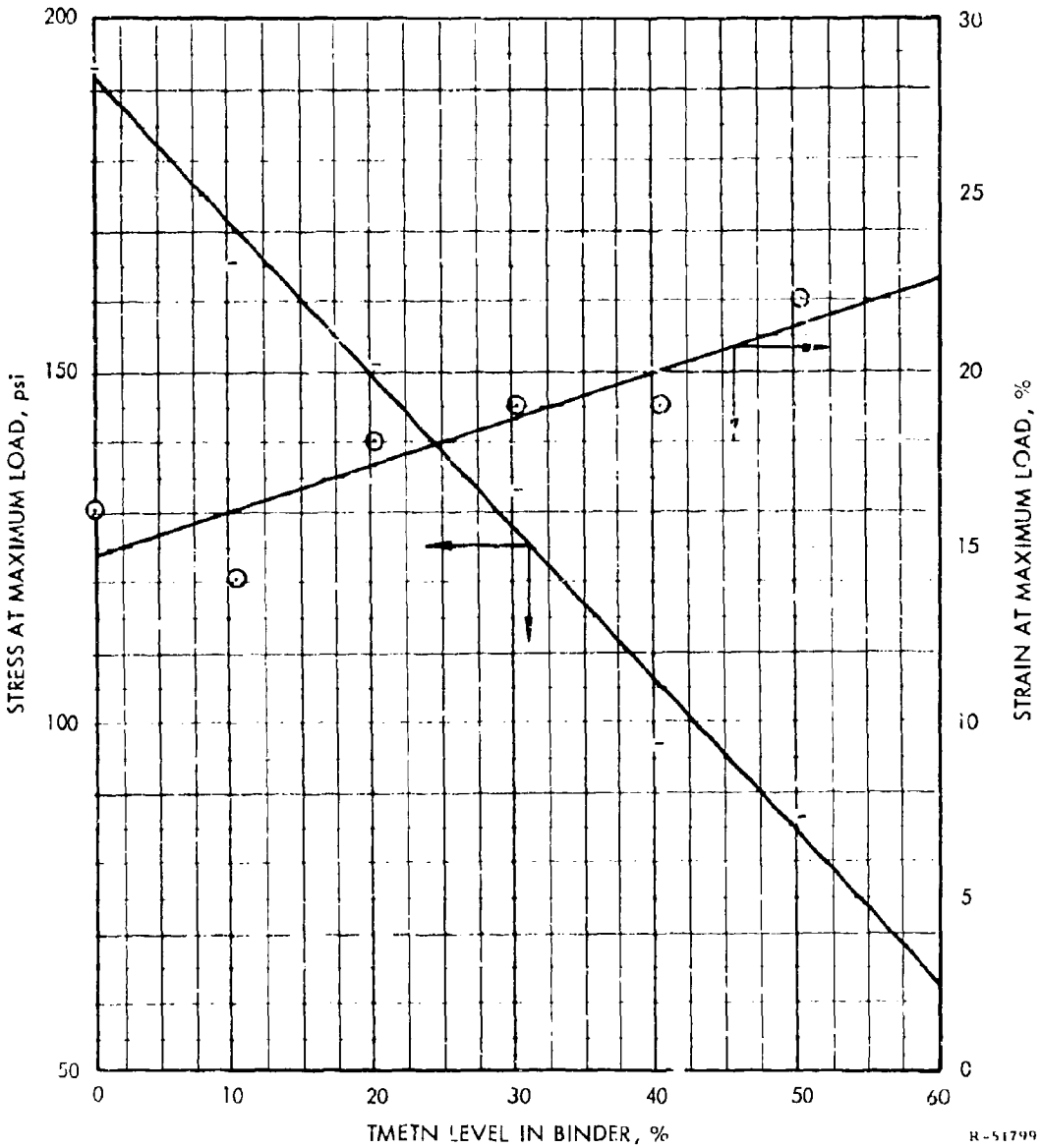

Figure 14. (U) Effect of Plasticizer Level on Propellant Mechanical Properties

content is minimal. It is because of this effect that the plasticizer level is being maintained at 50% rather than being increased to 55 or 60% to gain modest performance increase.

2.6.5 Liner Evaluation

(U) The liner development and improvement program has been based on the results of binder tests used in the propellant evaluations. Ten basic formulations were chosen as prospective candidates. Each was mixed with carbon black (Elftex-5) and wollastonite (Cab-O-Lite) as fillers. Small cup samples were prepared and cured 16 hr at 140° F. Propellant UTX 7702 was cast over the liner and cured 168 hr at 120° F. Qualitative analysis of the interfacial bond showed L200-36-4, L200-38-10, and L200-41-1 liners to be the best of the series. Shear tests are now being prepared to evaluate each of these liners with UTX 7700, UTX 7701, and UTX 7702. Quantitative data will be available from these tests.

III. FUTURE WORK

(C) The most promising formulations in the PEP-150/AlH_3/AP system at 23 vol % binder will be scaled up to 10-lb motors and tested to obtain maximum delivered specific impulse. The compound BTTN will be scaled up to 10-lb motor testing if laboratory-scale evaluation indicates that such scale-up is warranted.

(C) Both formulations from the mixed aluminum/aluminum hydride series will be tested in 10-lb motors for final evaluation.

(U) Burning rate studies currently in progress on the micromotor scale will be completed and promising formulations will be evaluated through the 10-lb motor scale during the next quarter.

(C) Studies to reduce the hazards of static discharge during handling of aluminum hydride and uncured propellants should be completed. In addition, a study to determine the effect of formulation parameters on impact sensitivity of uncured propellants will be completed.

(C) Studies to improve the aging and temperature limits of aluminum hydride propellants will emphasize the evaluation of several promising aluminum hydride treatments in 2-in. cubes and Taliani tests.

(U) The evaluation of reactive curative systems and of variations in polymer molecular weights will be completed early in the next quarter. An improved liner system will also be evaluated.

(U) Activities on Phase II, to characterize an improved propellant formulation, will begin on about 1 December 1965.

APPENDIX A

FORMULATION INDEX
ALUMINIZED PROPELLANTS

TMETN	12.50	12.50	12.50	12.50	12.50	12.50	12.50	12.50
Aluminum	21.00	21.00	21.00	21.00	21.00	21.00	21.00	21.00
AP	54.00	54.00	54.00	54.00	54.00	54.00	54.00	54.00
Ethyl Centralite	—	—	—	—	—	—	—	—
HX 874	—	—	—	—	—	—	—	—
NTPB	—	—	—	—	—	—	—	—
UNOX 201	0.85	—	—	—	—	—	—	—

	7595	7596	7597	7598	7599	7700	7701	7702
HX 735	10.40	10.31	10.34	10.43	10.24	7.32	7.30	6.98
MAPO	0.22	0.72	—	—	—	0.26	0.77	0.74
Epon 812	1.00	0.99	0.71	0.36	1.06	0.74	0.73	0.74
TBM	0.88	0.48	1.45	1.71	1.20	0.48	—	—
TMETN	12.50	12.50	12.50	12.50	12.50	8.80	8.80	8.80
Aluminum	21.00	21.00	21.00	21.00	21.00	16.00	16.00	16.00
AP	54.00	54.00	54.00	54.00	54.00	65.40	65.40	65.40
Ethyl Centralite	—	—	—	—	—	1.00	1.00	1.00
HX 874	—	—	—	—	—	—	—	—
NTPB	—	—	—	—	—	—	—	0.34
UNOX 201	—	—	—	—	—	—	—	—

	7703	7704	7705	7706	7707
HX 735	14.59	13.13	11.68	10.22	8.75
MAPO	1.53	1.38	1.22	1.07	0.92
Epon 812	1.48	1.33	1.18	1.03	0.84
TBM	—	—	—	—	—
TMETN	—	1.76	3.52	5.28	7.04
Aluminum	16.00	16.00	16.00	16.00	16.00
AP	65.40	65.40	65.40	65.40	65.40
Ethyl Centralite	1.00	1.00	1.00	1.00	1.00
Emery 1025-9R	—	—	—	—	—
Emery 1025-94R	—	—	—	—	—

	8200	8201	3226	8227	6641
HX 735	9.78	10.05	—	—	7.71
MAPO	—	—	—	—	—
Epon 812	1.35	1.04	0.76	0.51	0.87
TBM	1.37	1.41	1.10	0.75	1.27
TMETN	12.50	12.50	8.80	8.80	11.93
Aluminum	21.00	21.00	16.00	16.00	22.50
AP	54.00	54.00	66.40	66.40	55.60
Ethyl Centralite	—	—	—	—	0.12
Emery 1025-9R	—	—	6.94	—	—
Emery 1025-94R	—	—	—	7.54	—

Formulation No.

	7902	7934	7935	7936	7937	7938	7939	7942	7943
HX 735	5.99	6.36	6.31	6.27	6.22	5.99	5.99	5.76	5.61
MAPO	0.63	—	—	—	—	0.63	0.63	0.61	0.59
Epon 812	0.58	—	—	0.31	0.32	0.58	0.58	0.55	0.54
TBM	—	—	—	—	—	—	—	—	—
TMETN	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80
Aluminum	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00
AP	67.90	67.90	67.90	67.90	67.90	67.85	67.80	67.90	67.90
Ethyl Centralite	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
HX 874	—	0.84	0.89	0.62	0.66	—	—	—	—
NTPB	—	—	—	—	—	—	—	0.28	0.46
UNOX 201	—	—	—	—	—	—	—	—	—
Calcium Hydroxide	—	—	—	—	—	0.05	0.10	—	—

	7945	7577	7578	7579	75[illegible]0	7581	7584	7585	7586
HX 735	5.99	11.15	10.29	10.6[illegible]	10.56	10.91	10.15	10.36	10.26
MAPO	0.63	—	—	—	—	—	—	—	—
Epon 812	0.58	—	1.00	0.81	—	—	—	—	—
TBM	—	1.35	1.21	1.01	1.24	1.03	1.19	0.99	1.21
TMETN	8.80	12.50	12.50	12.50	12.50	12.50	12.50	12.50	12.50
Aluminum	16.00	21.00	21.00	21.00	21.00	21.00	21.00	21.00	21.00
AP	67.825	53.50	53.50	53.50	53.50	53.50	53.50	53.50	54.00
Ethyl Centralite	0.10	0.50	0.50	0.50	0.50	0.50	0.50	0.50	—
HX 874	—	—	—	—	0.70	0.56	—	—	—
NTPB	—	—	—	—	—	—	1.16	0.95	—
UNOX 201	—	—	—	—	—	—	—	—	1.04
Calcium Hydroxide	0.075	—	—	—	—	—	—	—	—

	7587	7588	7589	7590	7591	7592	7593	7594
HX 735	10.65	10.48	10.46	10.50	10.52	10.44	10.46	10.42
MAPO	—	0.51	0.44	0.59	0.66	0.37	0.44	0.29
Epon 812	—	1.02	1.01	1.02	1.02	1.01	1.01	1.01
TBM	1.00	0.49	0.59	0.39	0.30	0.68	0.59	0.78
TMETN	12.50	12.50	12.50	12.50	12.50	12.50	12.50	12.50
Aluminum	21.00	21.00	21.00	21.00	21.00	21.00	21.00	21.00
AP	54.00	54.00	54.00	54.00	54.00	54.00	54.00	54.00
Ethyl Centralite	—	—	—	—	—	—	—	—
HX 874	—	—	—	—	—	—	—	—
NTPB	—	—	—	—	—	—	—	—
UNOX 201	0.85	—						

APPENDIX B

FORMULATION INDEX
DOWANE 1451 PROPELLANTS

Formulation No.

	6814	6822	6825	6826	6827	6829	6834	6842
HX 735	9.40	7.81	6.91	6.64	9.26	3.86	7.76	9.74
MAPO	1.16	0.96	0.85	0.82	--	—	0.35	1.21
Epon 812	0.76	0.63	0.56	0.54	1.04	1.00	0.58	0.80
TBM	—	—	—	—	1.02	1.46	0.71	—
TMETN	14.01	9.40	10.20	9.80	14.01	14.01	9.40	11.75*
Dowane 1451	25.00	23.00	21.00	14.25	25.00	25.00	23.00	28.00
Aluminum	—	—	3.65	9.75	—	—	—	—
AP	49.53	58.00	56.63	58.00	49.53	49.53	58.00	48.50
Ethyl Centralite	0.14	0.10	0.10	0.10	0.15	0.15	0.10	—
Atlas G-2684	—	0.10	0.10	0.10	—	—	0.10	—

	6843	6844	6845	6846	6847	6848	6849	6850
HX 735	9.74	9.53	12.88	11.29	9.65	7.96	10.70	9.74
MAPO	1.21	1.19	1.61	1.41	1.20	0.99	1.33	1.21
Epon 812	0.80	0.78	1.05	0.92	0.79	0.65	0.87	0.80
TBM	—	—	—	—	—	—	—	—
TMETN	11.75*	11.50*	6.66*	9.08*	12.46*	9.60*	12.90*	11.75*
Dowane 1451	24.50	22.00	22.60	23.30	26.50	26.70	23.20	24.50
Aluminum	—	—	—	—	—	—	—	—
AP	52.00	55.00	55.20	54.00	49.40	54.10	51.00	52.00
Ethyl Centralite	—	—	—	—	—	—	—	—
Atlas G-2684	—	—	—	—	—	—	—	—

* TMETN contains 1% ethyl centralite

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Quarterly Progress Report for the period 1 August 1965 through 31 October 1965

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Rocket Propulsion Laboratory U.S. Air Force Edwards Air Force Base, California

13. ABSTRACT

(U) An evaluation of surfactants for reducing propellant mix viscosity has resulted in the selection of Atlas G-2684. Particle size distribution studies have led to the use of a 50/50 coarse/fine ammonium perchlorate (AP) grind ratio to maximize propellant castability. Processing studies have also resulted in a capability of processing propellant at the 23 vol % binder level with a consequent improvement in theoretical performance and density.

(C) A series of 4-lb motors have been tested containing aluminum hydride/aluminum combinations representing the most dense formulations with a theoretical performance in excess of 280 sec and the most dense formulation with a theoretical performance in excess of 274 sec. Propellant UTP 6825 has a theoretical I_{sp} of 280.1 sec, and a theoretical density of 0.0604 lb/in.^{3}. Propellant UTP 6826 has a theoretical I_{sp} of 275.0 sec and a theoretical density of 0.0627 lb/in.^{3}

(U) A study to determine the effect of propellant formulation parameters on burning rates has been essentially completed. An end-burning micromotor was utilized for these tests. It was found that the oxidizer-to-fuel (O/F) ratio, AP concentration, and AP particle size distribution were the most critical parameters. Binder concentration and plasticizer level were found to be less significant

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13. Abstract

(C) Efforts to minimize the handling hazards of aluminum hydride by increasing the electrical conductivity of the particles have been partially successful. A 1% coating of an antistatic agent reduced the electrical resistance of a 1 in. by 1 in. by 1/4 in. bed of particles from 1000 megohms for uncoated hydride to 40 to 70 megohms for the treated material.

(C) Propellant density studies indicate that in addition to the extensive drying of ingredients, propellant density can also be increased by sustained vibration and vacuum applied to the cast propellant. Surface treatment of the aluminum hydride with quinones, alizarin, or alizarin red S results in substantial improvements in propellant density. Testing with free-radical inhibitors has resulted in no noticeable improvements in propellant densities.

(U) The most promising curative systems evaluated to date are MAPO/Epon 812, MAPO/NTPB/Epon 812, and MAPO/TBM/Epon 812. Propellants containing these curative systems are in accelerated storage. Polyesters of increased molecular weight have been tested and appear to yield improved mechanical properties. Utilization of a molecular still for stripping moisture and light ends from the HX 735 polymer also results in some improvement in mechanical properties.

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