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(TITLE UNCLASSIFIED) DEMONSTRATION OF AN ADVANCED SOLID PROPELLANT

Omar A. Dewhirst
Thomas N. Scortia
United Technology Center

TECHNICAL REPORT AFRPL-TR-66-299
October 1966

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Air Force Rocket Propulsion Laboratory
Research and Technology Division
Air Force Systems Command, United States Air Force
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United Technology Center DIVISION OF UNITED AIRCRAFT CORPORATION

9 November 1966

ADP-581-66-F

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

Attention: FTMKR-4

Subject: Final Report, AFRPL-TR-66-299
Contract AF 04(611)-10812

Reference: (a) AFFTC Letter FTMKR-4, 6 October 1966
(b) UTC Letter BKF 1427-66-F, 26 August 1966
(c) Contract AF 04(611)-10812

Gentlemen:

United Technology Center submits the subject report which has been corrected in accordance with the reference (a) letter. The report is being forwarded to all recipients designated on the CPIA Distribution List including Categories 1 and 2 in accordance with the information provided by your office on 26 October 1966.

This completes the effort under the reference (c) contract with the exception of the submission of a supplemental report (Surveillance List Data) as required by Change Order 2.

Very truly yours,

UNITED TECHNOLOGY CENTER
A Division of United Aircraft Corporation

A. D. Parker, Manager
Contract Management

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cc: AFPRO, UTC, Sunnyvale, Calif.
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CONFIDENTIAL 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, 1st Lt, USAF/RPMC, AFFTC, Edwards Air Force Base, California.

(C) The present report is the final report 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 sec 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 16 April 1965 through 31 July 1966. The following professional workers made significant contributions to progress on this program in the activities indicated.

Project Manager	T. N. Scortia	Mechanical and Hazard Property Testing	E. C. Francis
Project Engineer	O. A. Dewhirst	Polymer Chemistry Consultation	T. P. Rudy
Propellant Stabilization, Density, and Safety Studies	P. L. Allen G. J. Casaletto R. M. Kumagai	Theoretical Calculations	J. D. Breazeale R. I. Sutton
Burning Rate Studies	R. M. Kumagai	Processing Operations	W. E. Robertson F. K. Leuschner
Mechanical Property Improvement Studies	J. W. Allan J. K. West	Motor Test Operations	P. A. Heady K. L. Tacke

(U) This report contains classified information extracted from (1) "Demonstration of an Advanced Solid Propellant (U)," Report No. AFRPL-TR-65-195, August 1965, (2) "Demonstration of an Advanced Solid Propellant (U)," Report No. AFRPL-TR-65-225, November 1965, and (3) "Demonstration of an Advanced Solid Propellant (U)," February 1966.

(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, 1st Lt, USAF/RPMC, Project Officer

CONFIDENTIAL ABSTRACT

(C) Substantial progress has been made in developing and characterizing useful propellant systems containing aluminum hydride, ammonium perchlorate, and a nitrato-plasticized polyether having a theoretical I_{sp} of 280.9 and a theoretical density of 0.059 lb/in.^{3} The propellant formulation selected for characterization (UTP 8812) has delivered an average I_{sp} efficiency of 92.7% in 10-lb motors or a measured I_{sp} of 260.4 (1,000/14.7, 0°). The vacuum specific impulse measured in 10-lb motors at an ϵ of 80:1 and corrected for 0° exit angle is 314.2 sec for an I_{sp} efficiency of 90.9%. BATES motors containing this formulation were shipped to the Air Force for further evaluation. The propellant density of these motors was in excess of 99% of the theoretical value. The burning rate of this propellant is 0.395 in./sec at 1,000 psia. Inflection points in the burning rate curve occur at 490 and 720 psia. The slope of the burning rate graph below 490 psia is 0.2. Between 490 and 720 psia, the slope is 0.1; and above 720 psia, the slope is 0.54.

(C) Burning rates have been demonstrated in 10-lb motors from a low of 0.25 in./sec at 1,000 psia to a high of 1.48 in./sec. These rates can be obtained with negligible losses in performance efficiency. Burning rates have been found to be effected primarily by oxidizer particle size, TMETN plasticizer concentration, and O/F ratio. The principal of using aluminum powder in combination with aluminum hydride to achieve increased propellant density has been demonstrated. Good I_{sp} efficiencies have been demonstrated in 10-lb motors (91.3% and 91.6% for two formulations containing 3.6% aluminum). However, for upper-stage missions in which a density exponent of 0.3 is used as a criteria in the term of $I_{sp}\rho^n$, the increase in density cannot compensate for the loss in I_{sp}.

(C) Other notable advances made during the course of this program have been the rapid scaleup and utilization of a new proprietary carboxy-terminated polymer developed at UTC which provides improved physical properties and improved propellant stability. The use of magnesium modified aluminum hydride, developed by Dow Chemical Company, appears to result in a substantial increase in propellant storage life. A section of a large motor (6-in. web) containing this material is in storage.

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ABBREVIATIONS AND SYMBOLS

AN	acrylonitrile
AP	ammonium perchlorate
Atlas G-2684	blend of sorbitan monooleate polyoxyethylene ester mixed acids (Atlas Chemical Industries, Inc.)
Atlas G-2410	a surfactant (Atlas Chemical Industries, Inc.)
Atlas G-2421	a surfactant (Atlas Chemical Industries, Inc.)
Atlas G-3300	alkyl aryl sulfonate (Atlas Chemical Industries, Inc.)
Atlas G-3570	high mol wt fatty amine blend (Atlas Chemical Industries, Inc.)
Atlas G-2406	a surfactant (Atlas Chemical Industries, Inc.)
Atlas G-3335	alkyl aryl sulfonate blended with polyoxyethylene sorbitan esters of mixed acids (Atlas Chemical Industries, Inc.)
Atlas G-2188	polyoxyethylene fatty glyceride (Atlas Chemical Industries, Inc.)
Atlas 8916P	polyoxyethylene sorbitan esters of mixed acids (Atlas Chemical Industries, Inc.)
BTTN	1,2,4 butanetriol trinitrate
cc	cubic centimeters
c^*	characteristic exhaust velocity (ft/sec)
C_f	thrust coefficient
DEGDN	diethyleneglycol dinitrate

(This page is unclassified)

DGAP	diethyleneglycol bis (bis [1-aziridinyl phosphate])
DNPN	2, 2 dinitropropylnitrate
DOA	dioctyl adipate
Dowane 1451	aluminum hydride (Dow Chemical Co.)
DPA	diphenyl acetylene
DTA	differential thermal analysis
E(t)	tensile modulus
E.C.	ethyl centralite
Epon 812	epoxy resin (Shell Chemical Co.)
Eq.	equivalents
g	gram
HAP	hydroxlamine perchlorate
HMAT	hexamethyl aziridinyl triazine (Interchemical Co.)
HMX	cyclotetramethylene tetranitramine
HP-2	hydrazine diperchlorate
HX-735	carboxy-terminated polyester (3M Co.)
HX-874	tris-(2-ethylaziridinyl)-s-triazine (3M Co.)
HX-858	propylene imine adduct of trimesic acid (3M Co.)
HX-868	butyl imine adduct of trimesic acid (3M Co.)
I_{sp}	specific impulse (sec)
K_n	ratio propellant surface to nozzle area
LMH-1	aluminum hydride

L-1806	experimental carboxy-terminated polyester (3M Co.)
L-1807	experimental carboxy-terminated polyester (3M Co)
MAM	proprietary imine polymer (Dow Chemical Co.)
MAES	bis (2-methyl) aziridinylethyl) sulfone
MAPO	tris(1-[2-methyl]aziridinyl) phosphine oxide (Interchemical Co.)
MBT	2-mercaptobenzothiazole
NC-1034	polyethylene imine polymer (Dow Chemical Co.)
NC-10262	polyethylene imine polymer (Dow Chemical Co.)
NTEB	nitrilotriethyl-β-ethyleniminebutyrate (American Cyanamid)
NTPB	nitrilotriethyl-β-propylenimonobutyrate (American Cyanamid)
SPAN 20	sorbitan monolaurate (Atlas Chemical Industries, Inc.)
O/F	oxidizer/fuel
PTA	phenolthiazine
P_{c_a}	average chamber pressure over action time (psia)
P_{c_b}	average chamber pressure over burning time (psia)
PEP	binder system based on a polyester plasticized by TMETN
r_b	propellant burn rate (in./sec)
TBM	tris-(2-ethylaziridinyl)-s-triazine (American Cyanamid)

TEGDN	triethylene glycol dinitrate
T_c	chamber temperature
TMETN	trimethylolethane trinitrate
TMNTN	trimethylolethane nitromethane trinitrate
Tween 20	polyoxyethylene (20) sorbitan monolaurate
Tween 21	polyoxyethylene (4) sorbitan monolaurate
Tween 80	polyoxyethylene (20) sorbitan monooleate
UNOX 201	epoxy resin (Union Carbide)
Uteflex	carboxy-terminated polyether (UTC proprietary polymer)
vol	volume
2-NDPA	2-nitrodiphenylamine
α_p	linear expansion
γ	specific heat ratio of gas mixture
ϵ	nozzle expansion ratio
ϵ_m	strain at maximum load
μ	microns
ν	Poisson's ratio
π_k	temperature coefficient of K_n
ρ	density
σ_m	stress at maximum load

SECTION I

INTRODUCTION

1. PROGRAM SCOPE

(C) Under Contract No. AF 04(611)-10812 with the Air Force Rocket Propulsion Laboratory (AFRPL), Research and Technology Division, Edwards Air Force Base (EAFB), UTC has conducted a program to continue the development of an advanced solid propellant based on aluminum hydride (AlH_3), ammonium perchlorate (AP), and nitrato-plasticized polyester and having a theoretical I_{sp} in excess of 280 lb-sec/lb at standard conditions. This program continued the efforts initiated under Contracts No. AF 04(611)-8513 and AF 04(611)-9570. It provides data on the important propellant parameters required to produce a family of usable propellants containing AlH_3.

(U) The program is divided into two phases: phase I, formulation improvement and tailoring experiments, and phase II, optimum formulation characterization. The program is described in greater detail in section 3, Program Outline.

2. REVIEW OF PREVIOUS PROGRAMS

(C) During 1962 to 1963, UTC completed an 8-month research program to develop a high-energy AlH_3 solid propellant with a theoretical I_{sp} of 280 sec or higher (standard conditions). Target goals were achieved in both a double-base system and in a nitrato-plasticized composite system.

(U) A theoretical performance study showed that only an oxygenated binder would allow the achievement of the target impulse. Both a trimethylol ethane trinitrate (TMETN)-plasticized double-base binder and a TMETN-plasticized polyester binder offered castable systems with acceptable I_{sp}.

(U) Under Contract No. AF 04(611)-9570 with AFRPL beginning in October 1963, UTC conducted a research program to continue the development of this system using a TMETN-plasticized polyester binder.

(U) This program included the development of a performance-optimized propellant and its demonstration in three nominal 50-lb firings and one nominal 350-lb firing. Included was an extensive propellant physical property and hazard characterization including a 12-month surveillance program based on 4-lb motors.

(C) The stability and decomposition kinetics of AlH_3 were studied in the early submicron particle-size material and in the later 30μ to 50μ material. A wide range of stabilities were encountered. Thermal stability was found to improve on treatment with mercury or organic nitriles, particularly acrylonitrile. However, it was found that mixtures of mercury-stabilized AlH_3 and nitrate esters gassed sufficiently to preclude the use of mercury-treated material in systems containing a nitrate ester.

(C) The thermal stability of AlH_3 has been constantly improving as manufacturing techniques have been refined and more effective treatments have been devised. Thermal stability of Mg-modified crystals represent an improvement of at least two orders of magnitude over early submicron-size material. The scaleup formulation, UTP 6814, which has a theoretical I_{sp} of 281. 1 sec, delivered 262. 3 sec (0° half-angle, 1,000 – 14. 7 psia) or 93. 4% of theoretical in the large motor.

(C) An extensive 4- and 10-lb motor program demonstrated that AlH_3 combustion efficiency is strongly dependent on oxidizer level and that, at sufficiently high oxidizer levels, up to 26% AlH_3 can be burned efficiently. Combustion temperatures above 3,000° K are necessary for good combustion. Combustion studies show that AlH_3 survives the surface of the burning propellant largely intact and dehydrogenates in the gas stream, with hydrogen and aluminum burning simultaneously. Because AlH_3 takes significantly longer to burn than comparable aluminum, residence time may be important to combustion efficiency. A steep scaleup mass discharge rate-performance efficiency curve tends to support this hypothesis. Nozzle losses appear less than with aluminum. This may be explained by the observation that combustion products include hollow oxide spheres of an average 4μ diameter.

(C) The uncatalyzed AlH_3 propellant shows inflection points in the burning rate-pressure log-log plot in the region of 800 to 1,100 psi. Temperature sensitivity (π_K) at K_n's in the 100 to 150 region is 0 22%/° F in the scaleup propellant. Aluminum hydride propellant shows little radial acceleration effect on burning rate in the plateau region at accelerations as high as 93 g.

(U) An Interstate Commerce Commission (ICC) classification of class B-unconfined has been given to the scaleup propellant. Although impact sensitivity of the propellant is in the 6-kg-cm region, the 20-ft drop test and

.50-caliber, machine-gun bullet test results on a 5-in. workhorse motor were negative. Similarly, no detonation was observed in an external heat test. Card-gap tests gave 34 cards for the scaleup propellant.

(U) A surveillance program using burning rate specimens, JANAF specimens, 4-lb motors, and uniaxial propellant-liner specimens was concluded after 60 weeks. The propellant deteriorated noticeably after a month's storage at 60° C (140° F). Major physical property changes were noted in 45° C (113° F) specimens after 81 days. Under ambient storage 4-lb motors showed essentially no physical or ballistic change for 1 year.

3. PROGRAM OUTLINE

a. 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 were performed concurrently.

(1) Task A10 — Maximum I_{sp} and Solids Loading

(U) Task 1 had as its objective the achievement of the highest possible delivered I_{sp} consistent with the normal constraints on such a system. This objective was 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 were demonstrated in 10-lb motors.

(2) Task A20 — Burning Rate Studies

(C) Task 2 had 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 1,000 psi have been established. The following formulation parameters were 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 were 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 were tested in 10-lb motors.

(3) Task A30 – Density I_{sp} Tradeoffs

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

(4) Task A40 – Improvement of Safety Propellants

(U) Task 4 had the objective of improving propellant safety properties. Propellant sensitivity reduction by the use of coatings and by changes in formulation parameters were explored.

(5) Task A50 – Aging and Temperature Limits

(U) Task 5 was devoted to defining the aging characteristics and the storage temperature limitations for this system. Techniques for improving storage limits received attention. This work included static and dynamic differential thermal analysis (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 involves the use of analog motors and a propellant segment in a large motor. Eighteen 10-lb motors and other specimens scheduled to be shipped to the Air Force for surveillance at their facilities were deleted from the program. In their place the following items were substituted on other tasks.

A. Viscoelastic tests on final scaleup propellant

   1. Biaxial strip tests

   2. Fiberglass analog motor tests

B. Eight 10-lb motors of two formulations to examine the specific impulse efficiency of:

   1. Low burning rate LMH-1 formulation

   2. Low burning rate mixed fuel LMH-1/Al formulation

C. Provide 24 test specimens to an AFRPL contractor for use in a gas diffusion study.

(6) Task A60 — Improvement of Mechanical Properties

(U) Improved mechanical properties were developed under task 6. The propellant curative system is optimized for mechanical properties and new curatives and new polymers were evaluated.

b. 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 were processed and shipped to the Air Force for scaleup testing.

4. REPORT STATUS

(U) The present report covers the experimental work performed from 16 April 1965 through 31 July 1966. The reporting status for the program is presented graphically in figure 1. An addendum report will be issued later on the results of the surveillance program and BATES motor firings.

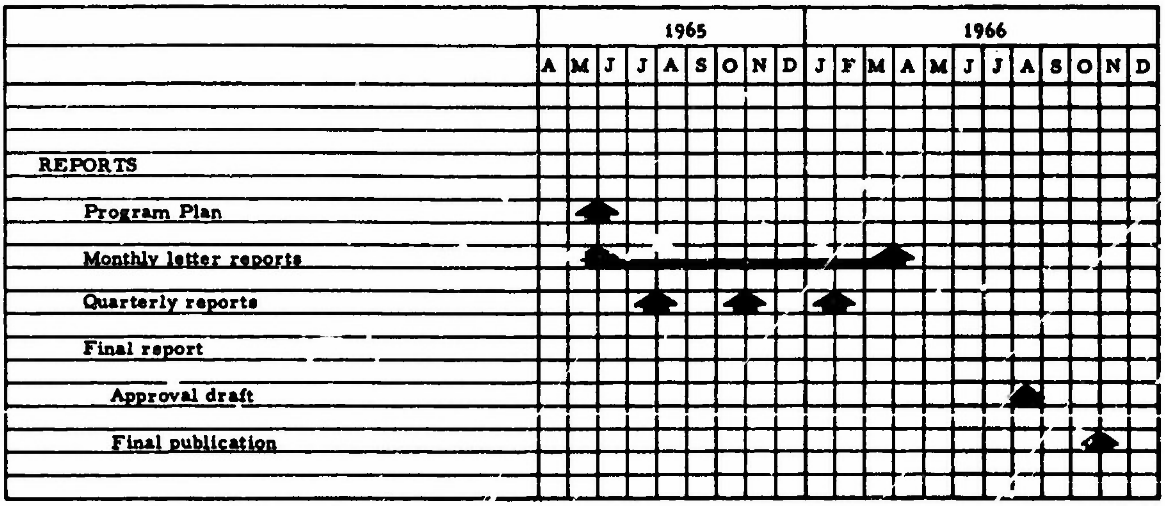


Figure 1. (U) Report Status

SECTION II

TECHNICAL ACTIVITIES

1. REVIEW OF ALUMINUM HYDRIDE CHARACTERISTICS

(C) The most stable polymorph of AlH_3, designated Dowane 1451 by Dow Chemical Company and Olane 58 by Olin Mathieson Chemical Corporation, is an off-white powder with a measured (pycnometric) density of 1.44 g/cc. Production samples historically have varied widely in thermal stability, crystal form, and bulk density, although continual improvements are being made. Aluminum hydride is compatible with nitrate esters, AP, carboxy-terminated and imine-terminated polyesters, MAPO and similar curatives, common acid scavengers, and most of the common ballistic modifiers. Aluminum hydride is also generally compatible with likely variations of the nitrato-composite binders designated as the PEP-100 series at UTC.

a. Physical Properties

(C) The stable polymorph of AlH_3 has a heat of formation of -2.17 kcal/mole and a crystal density of 1.474 g/cc by helium densitometer. Particle forms observed include bead-like crystals and strings of bead-like crystals. Large crystals may be single or fused of smaller crystals. The bulk densities of Dowane 1451 delivered during this program and the one previous have shown a steady improvement. The bulk density is now generally between 0.78 and 0.91 g/cc. The bulk density of lots in 1963 was between 0.43 and 0.62 g/cc.

b. Stability and Compatibility

(1) Stability History

(C) The thermal stability of LMH-1 has steadily improved. During the early development stages, the thermal stability appeared to be equated with particle size. The initial micro-crystalline material was considerably less stable than later macro-crystalline AlH_3 of about 50μ particle size. During 1964 a larger (approximately 100μ) crystalline material became available which represented a further improvement in stability. The most recent modification has been the modification of the AlH_3 crystal with magnesium. Pilot plant lots of such modified material have shown a five-fold improvement in stability compared to the best material obtained prior to this modification. This history of steady improvement in thermal stability is shown as a function of Taliani measurements at 60° C in figure 2.

(C) Taliani measurements at 60° C show a wide range of stabilities depending on the particle size, analysis, age, and previous chemical and thermal history of the sample. Figure 2 shows typical stability measurements for an early pilot-plant lot of macro-crystalline Dowane 1451 (DL-11) and a miniplant lot (DL-10). The two shaded portions of the graph define the range of stability measurements for the coarse grade of AlH_3 (100μ) and for the magnesium-treated hydride. This graph represents stability of the material before it is subjected to any stabilizing treatment. The two most successful stabilizing treatments used before the development of the magnesium additive have been the liquid acrylonitrile treatment and continuous ether extraction. A treatment for the magnesium-modified AlH_3, which is still in the development stages, involves the use of diphenylacetylene during the preparation of the hydride. Preliminary studies by Dow Chemical Company indicate a substantial improvement in thermal stability by this method.

2. PHASE I – FORMULATION IMPROVEMENT AND TAILORING EXPERIMENT

a. Maximum I_{sp}, Density and Solids Loading Studies – Tasks 1 and 3

(U) Tasks 1 and 3 have objectives which are closely interrelated. The objective of task 1 is the achievement of the highest possible delivered I_{sp} to be achieved by processing studies to obtain the maximum solids loading and by formulation studies to optimize the concentration and type of oxygenated plasticizer. Task 3 involves a study of density I_{sp} tradeoffs to achieve substantially increased propellant densities with minimum degradation of I_{sp}. As essentially the same theoretical calculations and the same processing studies were required to support the objectives of both tasks, these studies will be treated together in this section.

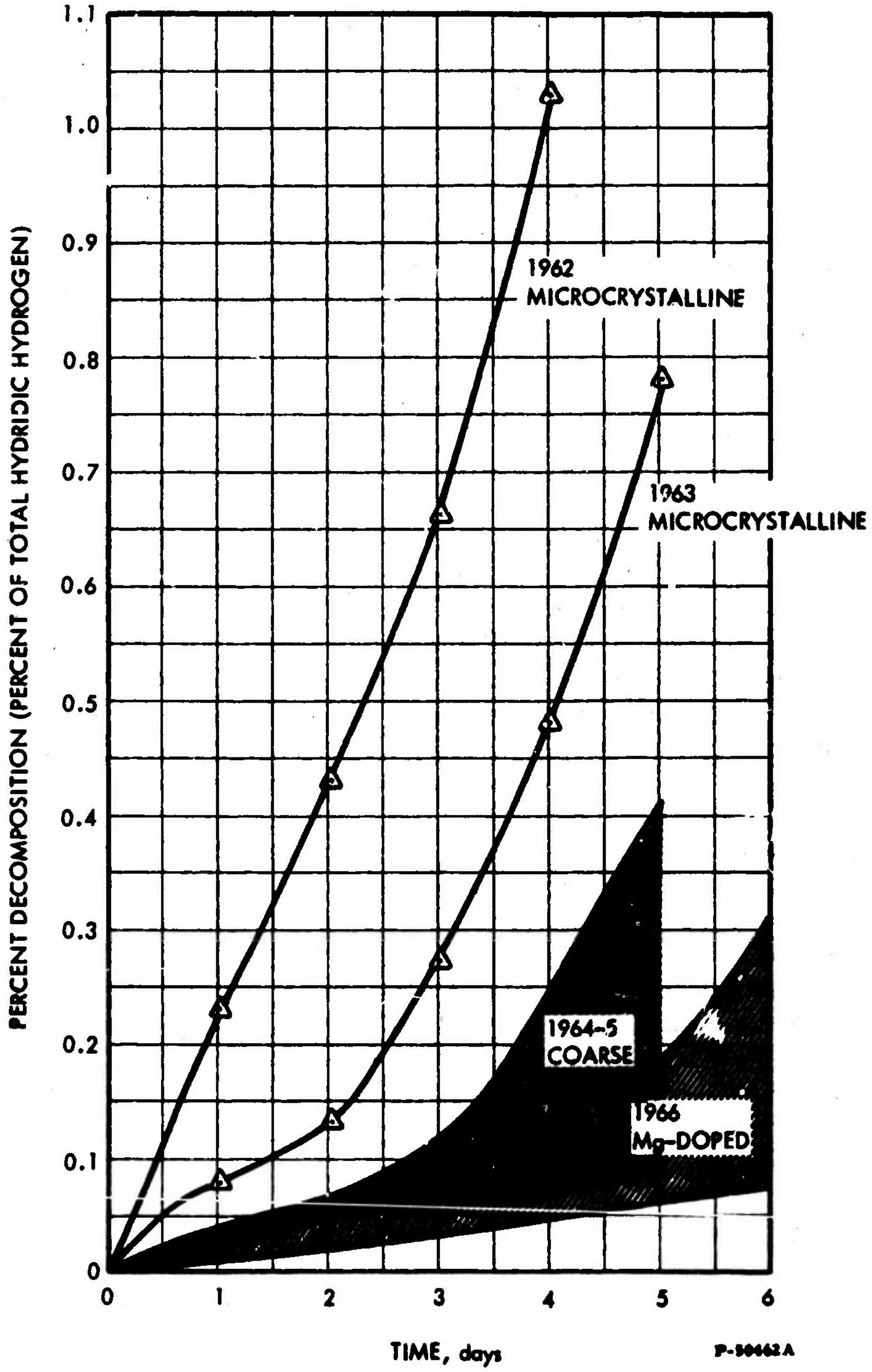


Figure 2. (C) Stability of Various Aluminum Hydrides at 60° C

(1) Theoretical Studies

(C) The theoretical I_{sp} of AlH_3 propellants may be increased by such measures as (1) the use of higher AlH_3 loadings, (2) the use of NF_2 binders, (3) the use of more highly oxygenated binders, (4) the use of higher solids loading, and (5) the use of advanced oxidizers.

(C) The density of AlH_3 propellants may be increased by such measures as (1) reduction of AlH_3 content, (2) the use of a secondary metallic fuel in a mixed fuel system, (3) reduction of binder content, and (4) the use of more dense oxidizers.

(C) The use of increased AlH_3 for increased performance is limited by the problems of combustion efficiency and two-phase flow losses. Extensive performance efficiency studies under Contract AF 04(611)-9570 indicate that levels above 25 wt-% are not desirable. Although calculations under Contract AF 04(611)-10540, "Evaluation of an Advanced Binder," indicate that theoretical I_{sp} of 295 sec in a processable region may be achieved in the PBEP difluoramino binder, the scope of the present program limits binder experimentation to nitrato-plasticized binders of more conventional analysis. Within this limitation, however, theoretical performance gains can be realized with more highly oxygenated plasticizers.

(C) Increased solids loading and increased binder density through the use of more dense plasticizers contribute to improved propellant density. The most significant improvement without performance loss, however, stems from the use of such dense oxidizers as hydroxylamine perchlorate (HAP) and hydrazine diperchlorate (HP-2). In systems oxidized with AP, the greatest improvements in density result from the use of mixed hydride-metal fuels. However, this approach results in performance losses which are quite significant in the case of zirconium.

(a) Effect of Plasticizer Level

(C) All of the AlH_3 formulations under Contract No. AF 04(611)-9570 were based upon the PEP-155 binder, a MAPO-crosslinked carboxy-terminated polyester (Minnesota Mining and Manufacturing Company's HX-735) plasticized with 55 wt-% TMETN.

The general effect of increasing the TMETN content of the PEP-100 system is to decrease the binder carbon and hydrogen analysis, and to increase the I_{sp} and density of a particular composition in a metal hydride system. However, the greater density of the plasticized binder results in some unfavorable reduction of the binder vol-%. The net results of these two opposed effects is an increase in performance and propellant density.

(C) Figures 3, 4, and 5 are ternary plots of the performance of AlH_3 in AP-oxidized systems with PEP-100 binders containing 50 wt-%, 55 wt-%, and 60 wt-%, respectively, of TMETN. It may be seen that, in addition to the other affects mentioned above, the binders with greater plasticizer levels display higher O/F ratios for particular compositions.

(C) The effect of increasing plasticizer levels on both performance and density may best be observed by the treatment of the data in figure 6. In this figure both I_{sp} and density are plotted against plasticizer level for formulations containing a constant 25 wt-% AlH_3 for two binder levels — 25 vol-% and 30 vol-%. A gain of 1 sec at either binder level is observed in going from 50 wt-% to 60 wt-% plasticizer with the major gain occurring in the 50 wt-% to 55 wt-% interval. This performance gain is accompanied by modest improvement in density.

(C) If only the PEP-100 systems having a theoretical specific impulse of 280 sec are considered, the treatment in figure 7 results. In figure 7 the wt-% of AlH_3 and the density are plotted as a function of the plasticizer level for compositions with both 25 vol-% and 30 vol-% binder. The AlH_3 requirements for a theoretical 280-sec decrease with increasing plasticizer level but at lower binder levels the change is not significant unless the binder contains greater than 55 wt-% TMETN. In these systems, the density rises rapidly at both binder levels with decreasing AlH_3 content.

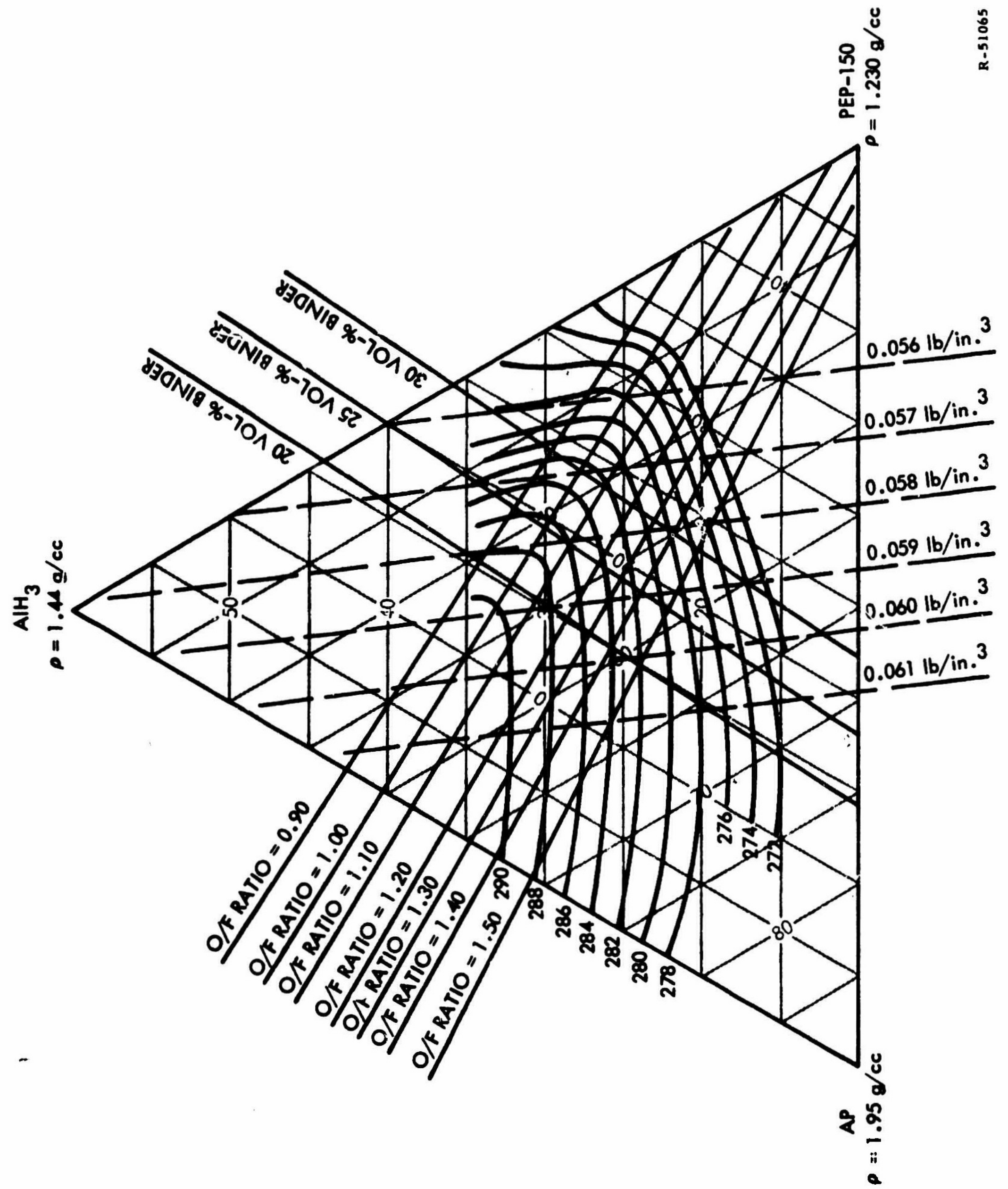


Figure 3. (C) Performance in the AlH$_3$/AP/PEP-150 System

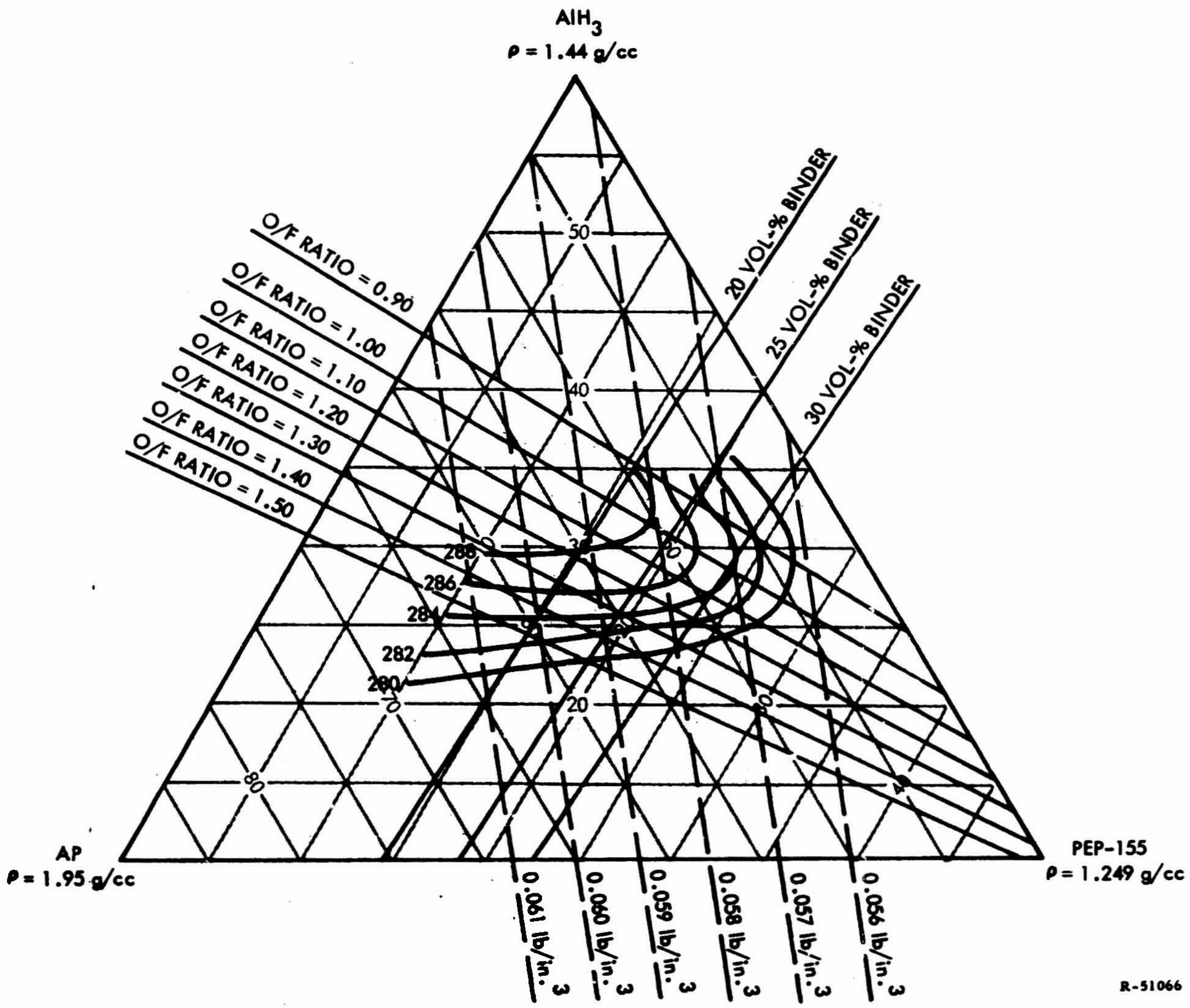


Figure 4. (C) Performance in the AlH$_3$/AP/PEP-155 System

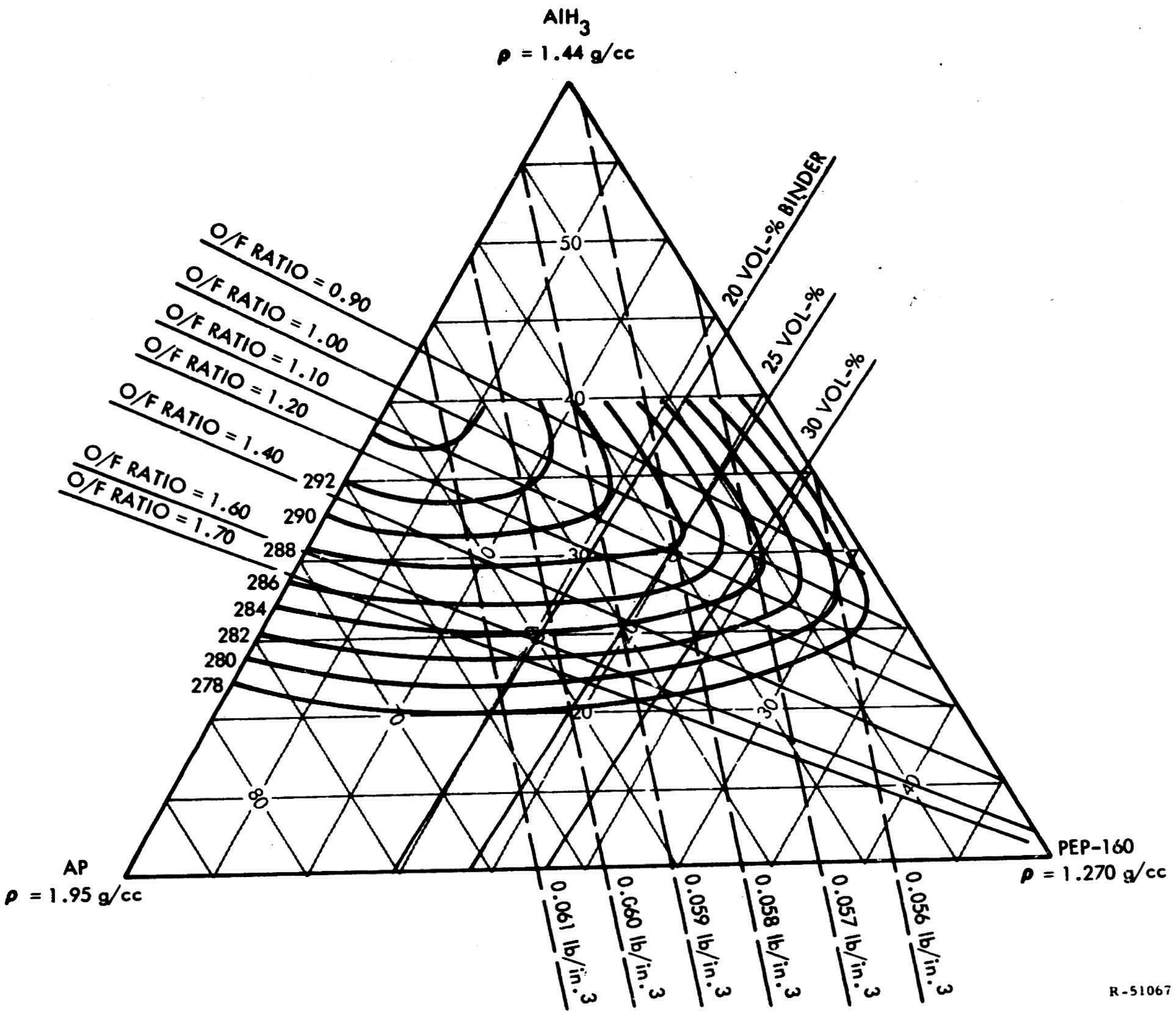


Figure 5. (C) Performance in the AlH$_3$/AP/PEP-160 System

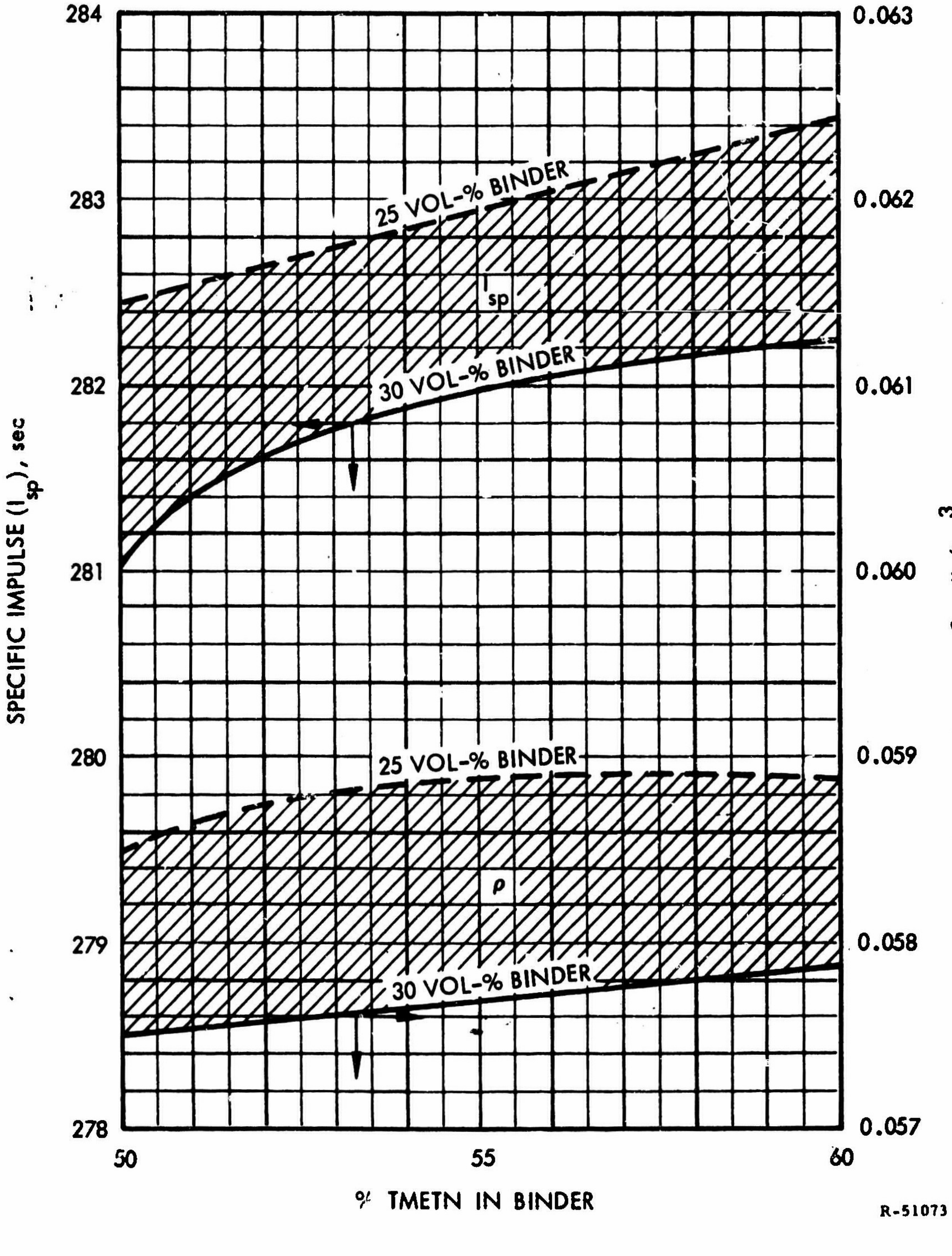


Figure 6. (C) Performance-Density Gains with Increasing Binder-TMETN Level for a Constant 25 wt-% AlH_3 Loading in the AlH_3/AP/PEP Systems

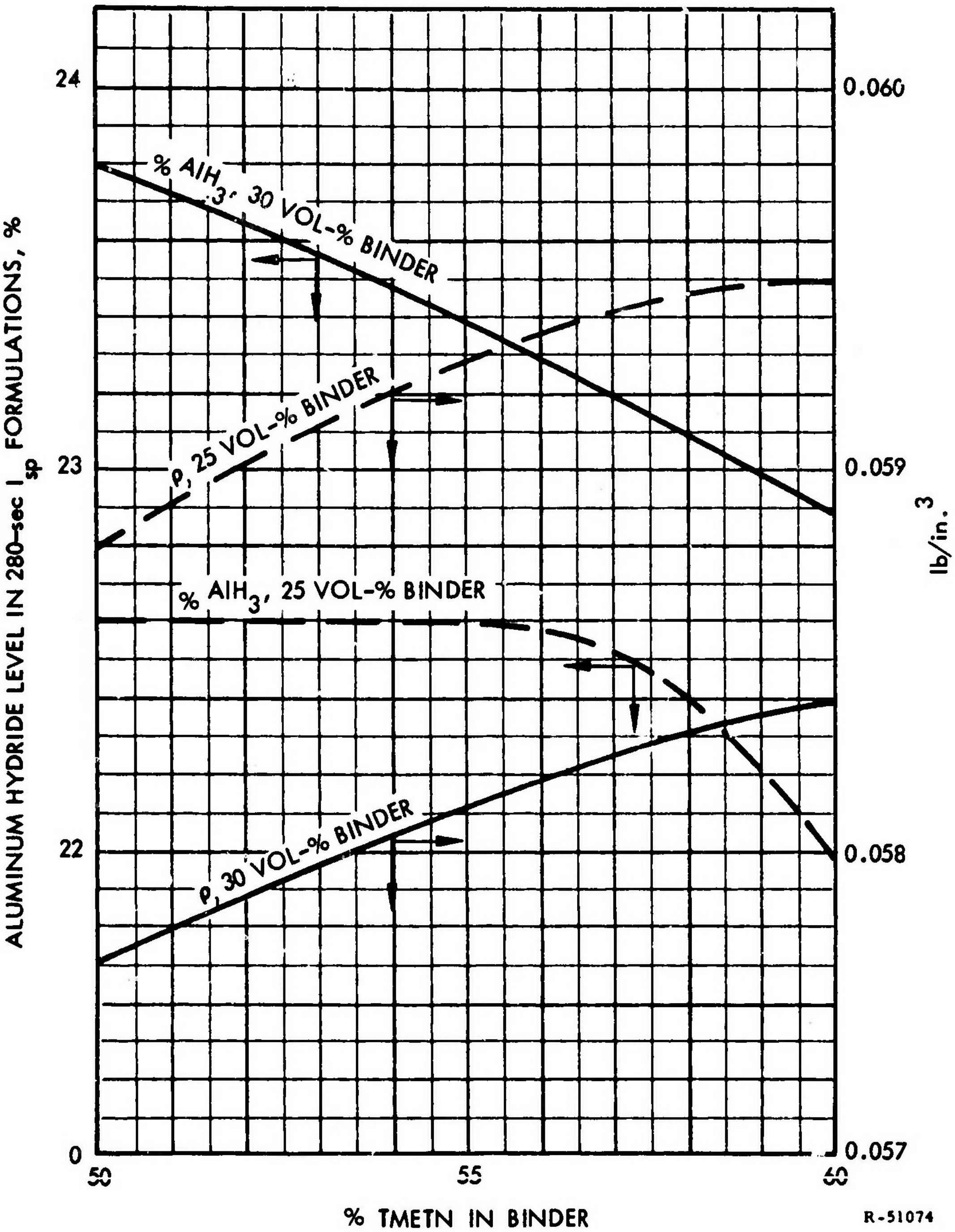


Figure 7. (C) Hydride Level and Density in I_{sp} 280-sec Formulations for Various Binder TMETN Levels

(C) The significant conclusion to be derived from figures 6 and 7 is that performance advantages appear more rapidly in the PEP-100 systems at plasticizer levels above 55 wt-%, and that the performance gain at the 25 wt-% hydride level between 50 wt-% and 55 wt-% TMETN is only 1 sec. The decline in physical properties observed in this binder in that region suggests that some performance and density should be sacrificed in the interest of better tensile properties.

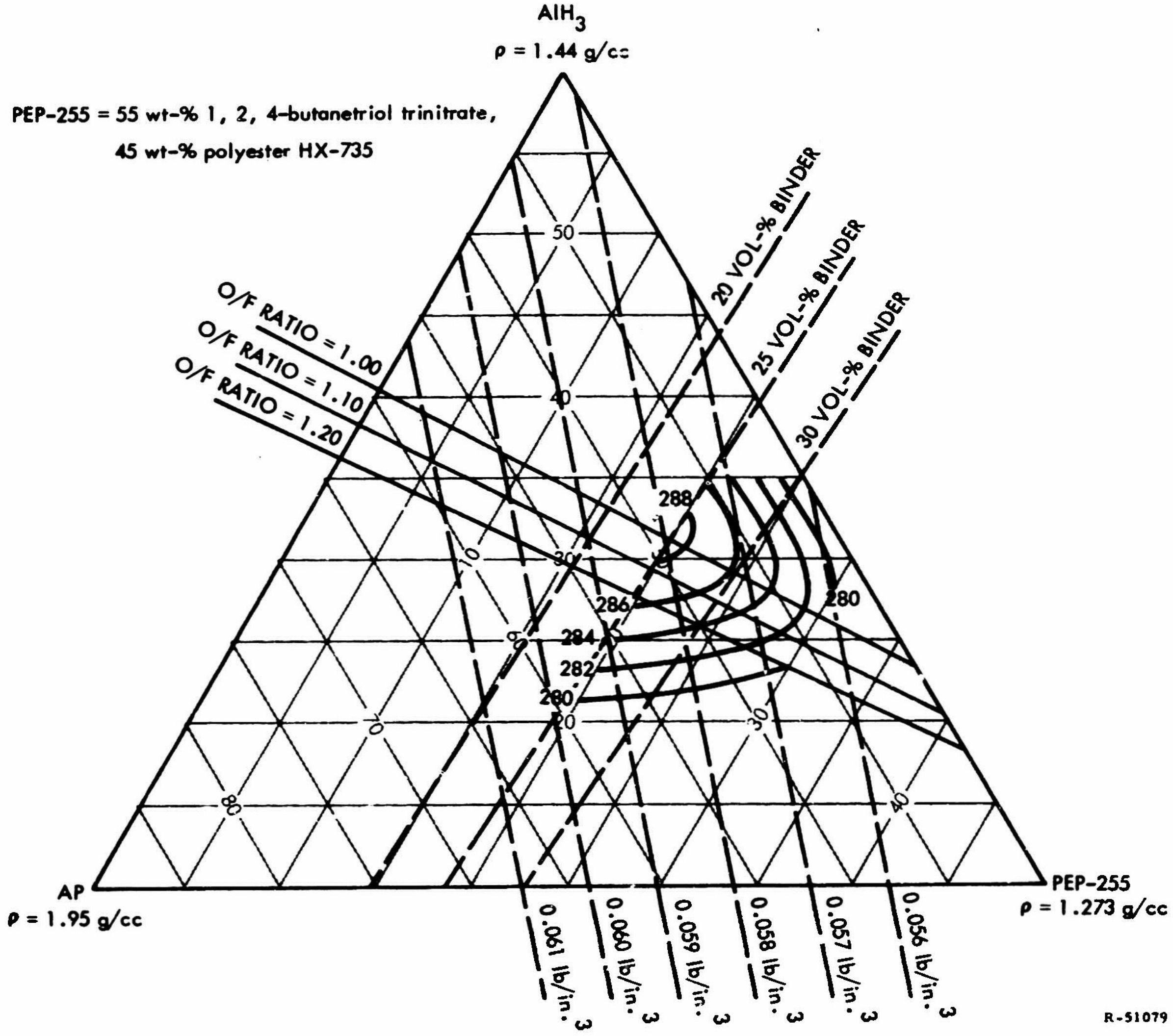
(b) Effect of Other Nitrato Ester Plasticizers

(C) The effects of other nitrate plasticizers on the performance of the AP-oxidized AlH_3 system have been studied at the 55 wt-% plasticizer level. The plasticizers investigated have been 1, 2, 4 butanetriol trinitrate (BTTN) in the PEP-255 binder, 2, 2 dinitropropylnitrate (DNPN) in the PEP-355 binder, and trimethylolnitromethane trinitrate (TMNTN, NIB-nitroglycerine) in the PEP-455 binder. The ternary performance diagrams of these systems are shown in figures 8, 9, and 10, respectively.

(C) For purposes of comparing the effects of the three new plasticizers, the intersection of the 25 vol-% binder line with the 25 wt-% AlH_3 line is selected. In the PEP-155 system, the composition at this point (25. 0% AlH_3/50. 5% AP/19. 5% PEP-155) has a theoretical I_{sp} of 283 sec with a density of 0. 0589 lb/in.^{3} in an O/F ratio of 1. 35.

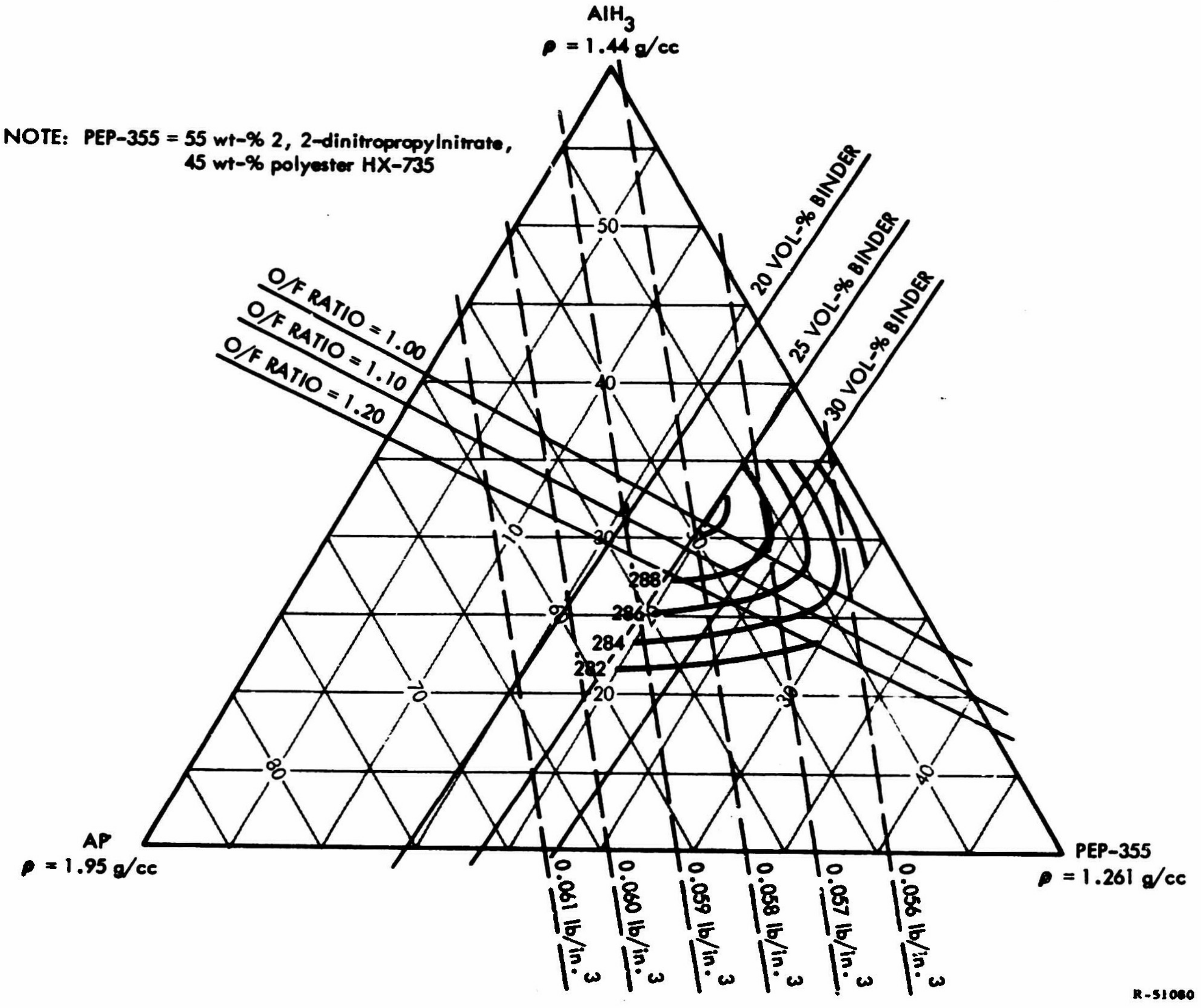
(C) In the PEP-255 system containing 1, 2, 4 butanetriol trinitrate as a plasticizer, the same intersection of the 25 vol-% binder line and the 25 wt-% AlH_3 line (composition: 25. 0% AlH_3/50. 5% AP/19. 5% PEP-255) has a theoretical I_{sp} of 284 sec, a gain of 1 sec over the PEP-155 system. This composition in the PEP-255 system has a density of slightly better than 0. 059 lb/in.^{3}, a small gain over the PEP-155 system, and an O/F ratio of 1. 40, a gain of 0. 05 over the PEP-155 system.

(C) The PEP-355 system with the plasticizer 2,2 dinitropropylnitrate shows essentially the same I_{sp} and O/F ratic relationships as the PEP-255 system and the 25 vol-% binder-25 wt-% AlH_3 intersection have identical I_{sp},



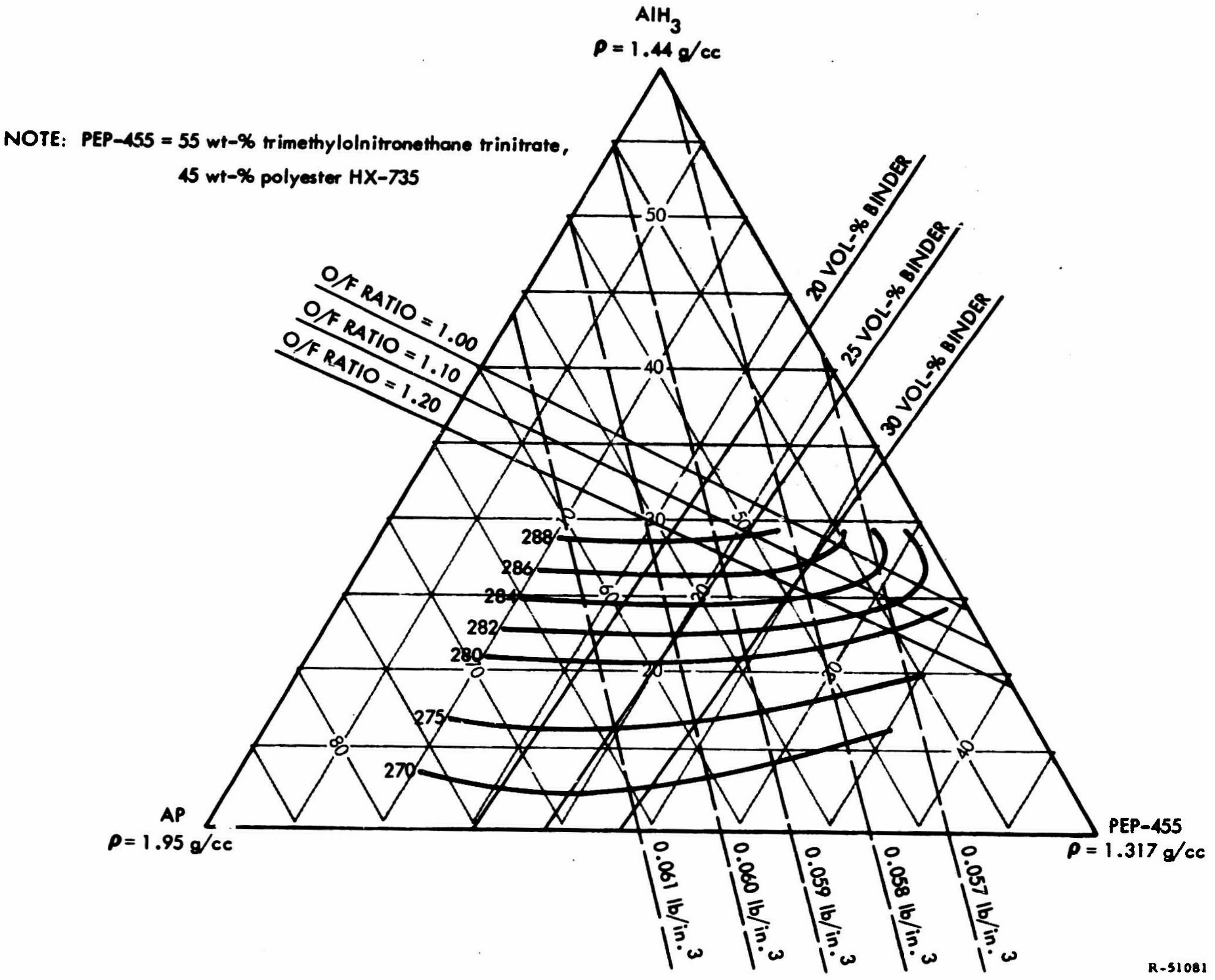
NOTE: PEP-255 = 55 wt-% 1, 2, 4-butanetriol trinitrate,
45 wt-% polyester HX-735

Figure 8. (C) Performance in the AlH_3/AP/PEP-255 System



NOTE: PEP-355 = 55 wt-% 2, 2-dinitropropylnitrate,
45 wt-% polyester HX-735

Figure 9. (C) Performance in the AlH$_3$/AP/PEP-355 System



NOTE: PEP-455 = 55 wt-% trimethylolnitronethane trinitrate, 45 wt-% polyester HX-735

Figure 10. (C) Performance in the AlH$_3$/AP/PEP-455 System

O/F ratio, and density values to that of the PEP-255 system. From the theoretical standpoint, these two plasticizers must be considered equivalent.

(C) The PEP-455 system with trimethylolnitromethane trinitrate (NIB-nitroglycerine) as a plasticizer also shows a theoretical I_{sp} of 284 sec at the 25 vol-% binder 25 wt-% AlH_3 intersection point. The O/F ratio at this point is again 1.40 as in the PEP-255 and PEP-355 systems. However, the theoretical density in this system of the intersection point is 0.0595 lb/in.^{3}, a gain of 0.85% in theoretical density over the PEP-255 and PEP-355 systems, and a gain in density of over 1.0% over the PEP-155 system.

(C) In general, theoretical calculations indicate that the use of new plasticizers in place of TMETN will allow an increase in performance of 1 sec or 0.35%, an increase in density of 0.0006 lb/in.^{3} or 1.0% and an increase in O/F ratio of about 0.05 or 3.7%. The small gains in specific impulse density and O/F ratio do not appear to warrant replacing TMETN.

(c) Effect of Advanced Oxidizers

(C) Another approach to the improvement of performance in the AlH_3 system is the substitution for AP of a more energetic or more oxygenated oxidizer. In the initial theoretical studies HMX and HAP were examined with TMETN and TMNTN.

(C) The performance diagram for the AlH_3-PEP-155 system, oxidized with HMX, is shown in figure 11. Because of the low oxygen content of this oxidizer, the performances are disappointingly poor as are the O/F ratios in the regions of interest. The best performance at 25 vol-% binder is 276 sec. However, the addition of oxygen to this system should result in some improvement in performance.

(C) The performances calculated for AlH_3/PEP-155 and PEP-455 with HAP are more encouraging. In the PEP-155 system in figure 12, the intersection of the

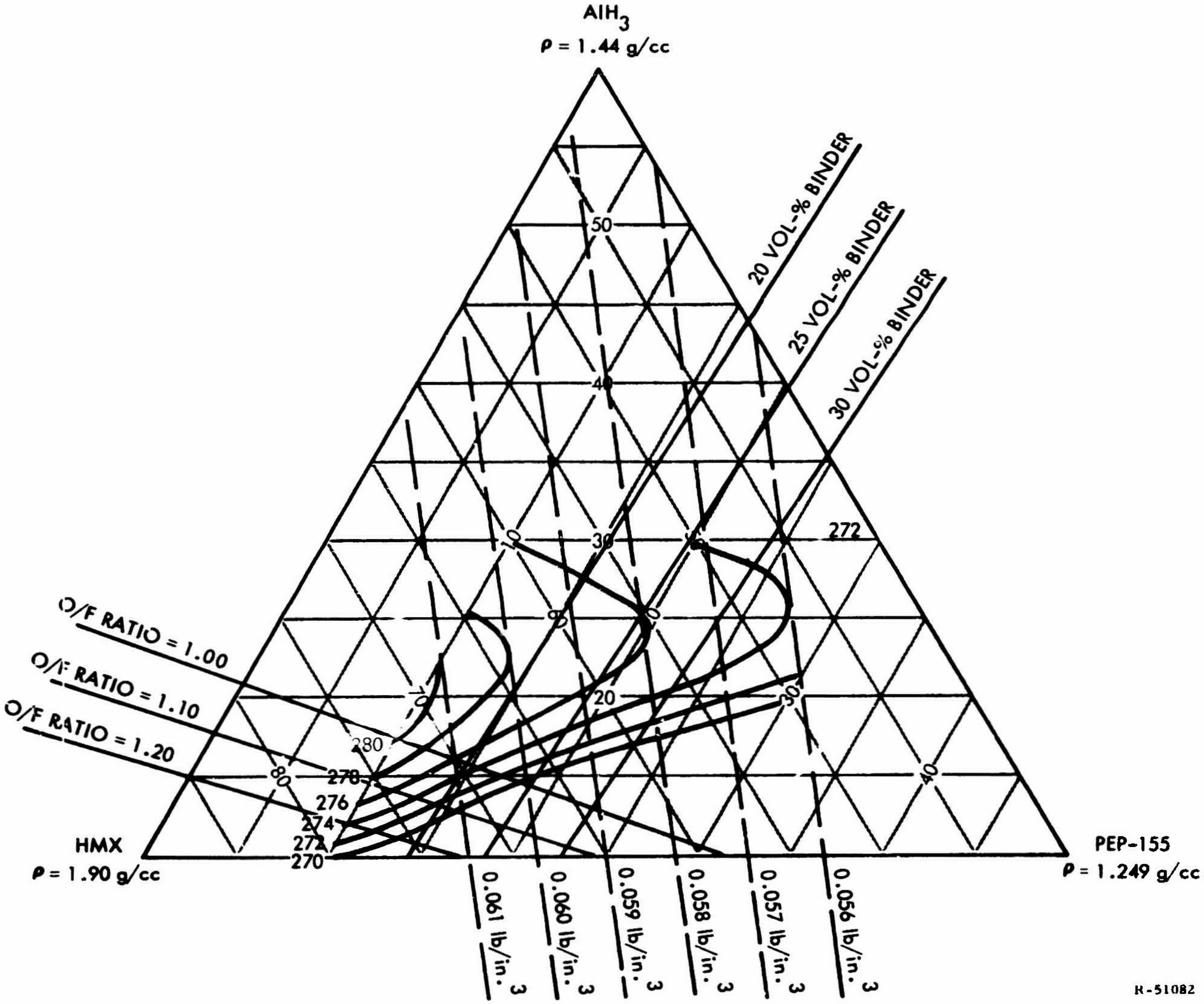


Figure 11. (C) Performance in the AlH$_3$/HMX/PEP-155 System

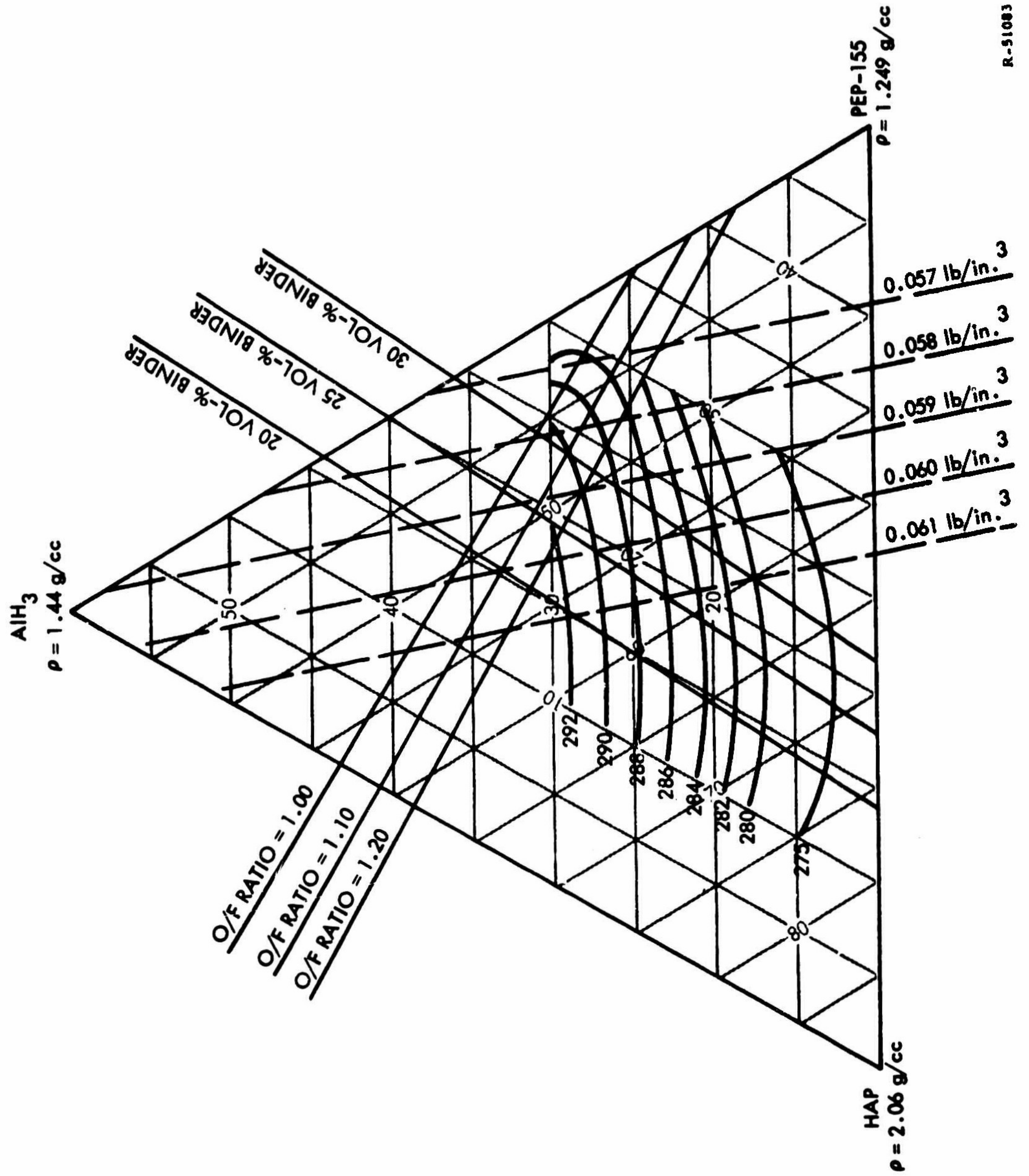


Figure 12. (C) Performance in the AlH$_3$/HAP/PEP-155 System

25 vol-% binder line with the 25 wt-% AlH_3 line gives a composition (25.0% AlH_3/56.2% HAP/18.8% PEP-155) with a theoretical I_{sp} of 288 sec and a theoretical density of 0.065 lb/in.^{3} The O/F ratio in this composition is over 1.5, favoring excellent combustion efficiency.

(C) In figure 13, the ternary for the AlH_3/HAP/PEP-455 system, the 25 vol-% binder/25 wt-% AlH_3 intersection point (composition: 25.0% AlH_3/56.0% HAP/19.0% PEP-455) has a theoretical performance of 289 sec and a theoretical density of 0.0611 lb/in.^{3} The O/F ratio of this composition is approximately 1.65.

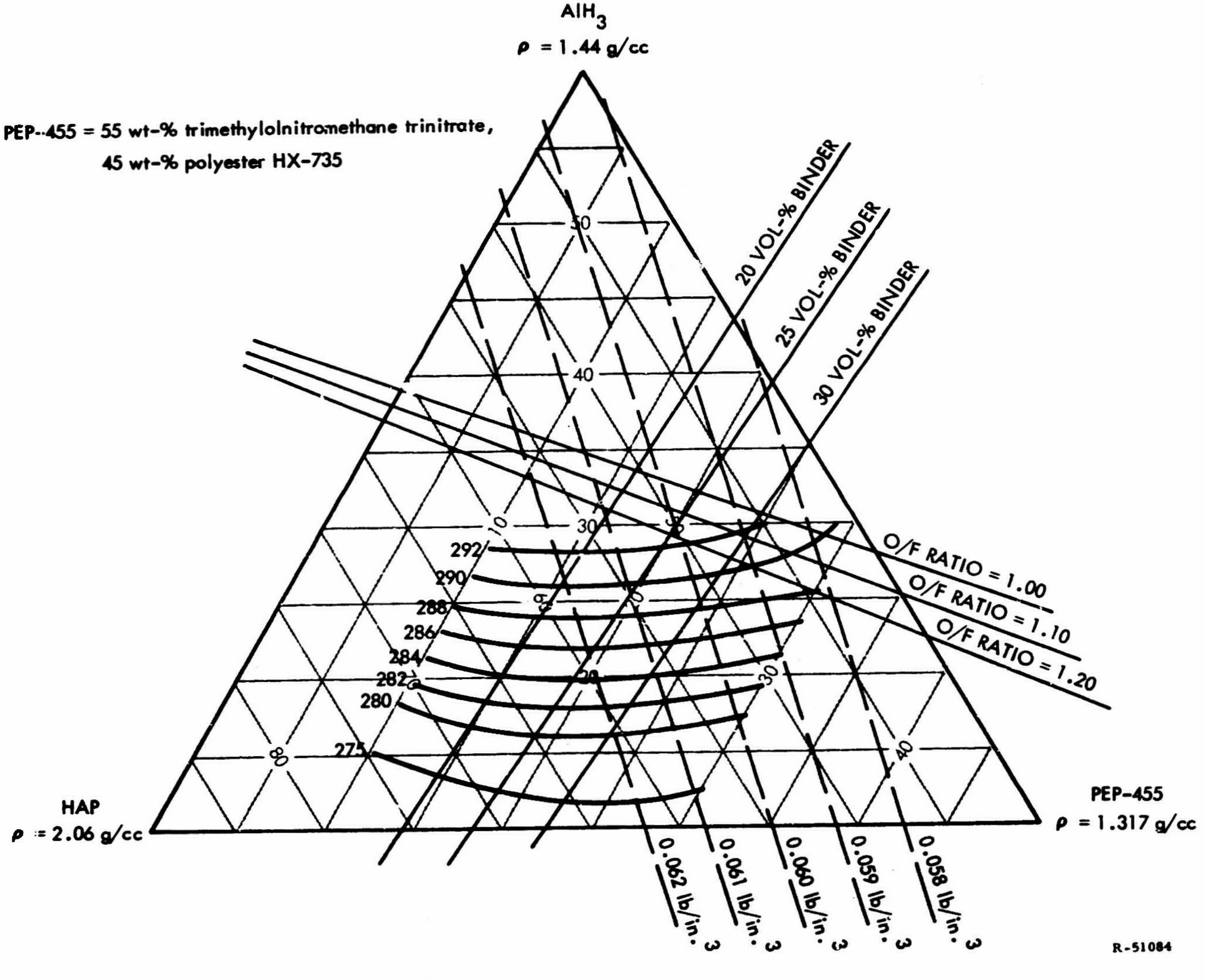
(C) From these latter calculations, it becomes obvious that the use of HAP in an AlH_3-PEP-455 system offers the best possibility for density and performance improvement in the PEP systems. The overall improvement over the AP/PEP-155 system is 6 sec or 2.1% in I_{sp} and 0.0022 lb/in.^{3} or 3.7% in density.

(d) Effect of Mixed Metal-AlH_3 Fuels

(C) The tradeoffs between increasing density and decreasing performance were examined in systems containing mixtures of AlH_3 and aluminum metal and in systems containing mixtures of AlH_3 and zirconium metal. Generally, performance losses with increasing density were greater in the zirconium than in the aluminum-containing systems.

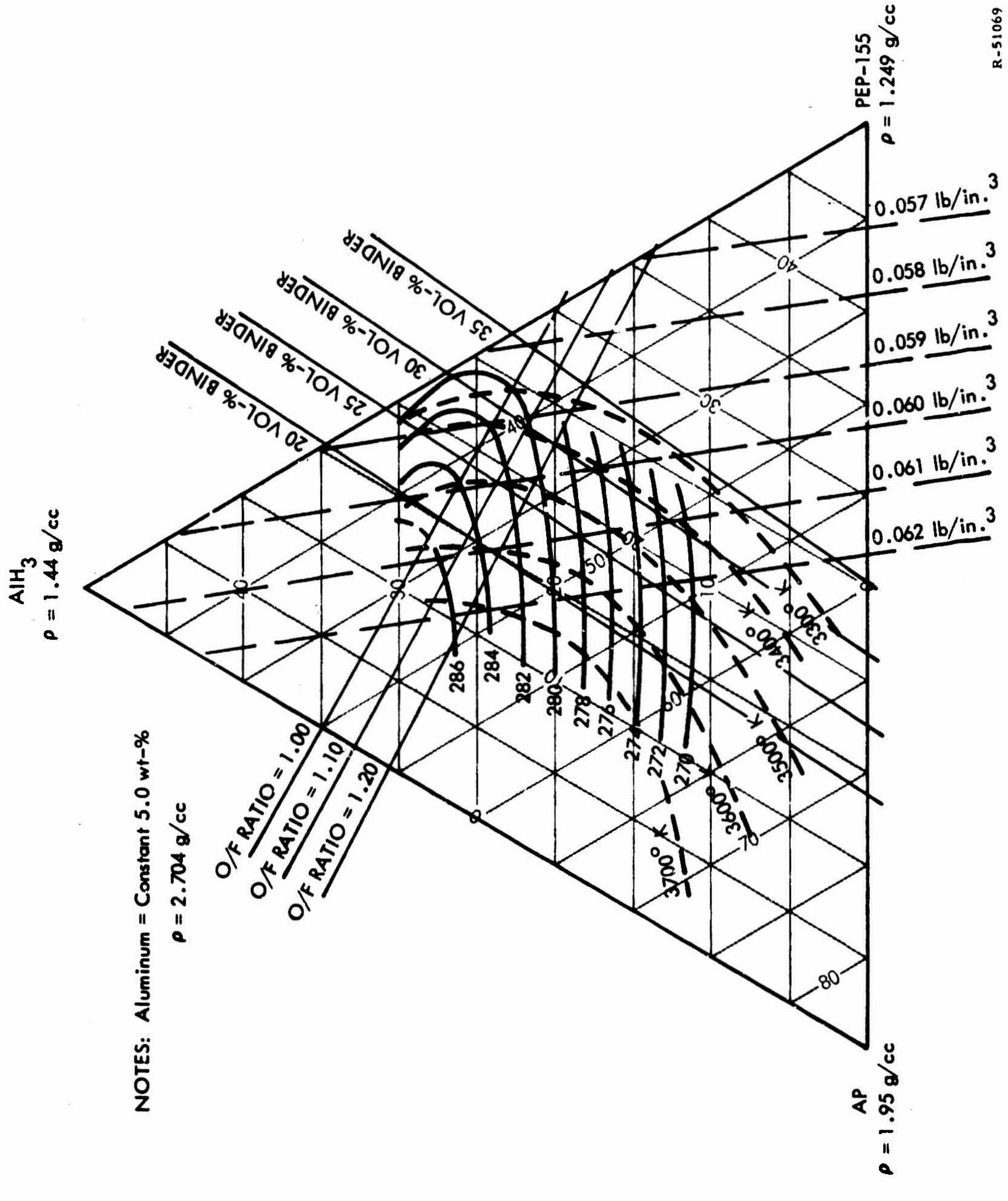
(C) Ternary diagrams of performance and other parameters in systems containing a constant 5%, 10%, and 15% aluminum metal are shown in figures 14, 15, and 16, respectively. A progressive loss in I_{sp} and in O/F ratio is accompanied by increasing density and increasing flame temperature. In compositions containing constant 22.5% metal analysis, either as metal or as hydride, the flame temperature at the 25 vol-% binder level increases from 3,300° K in the all-hydride system through 3,530° K and 3,620° K in the 5% and 10% aluminum metal systems to 3,830° K in the system containing 15% aluminum.

(C) The effect of aluminum addition on I_{sp} and density is seen in figures 17 and 18 where aluminum analysis in the composition is held to 18.0% and to 22.5%, respectively.



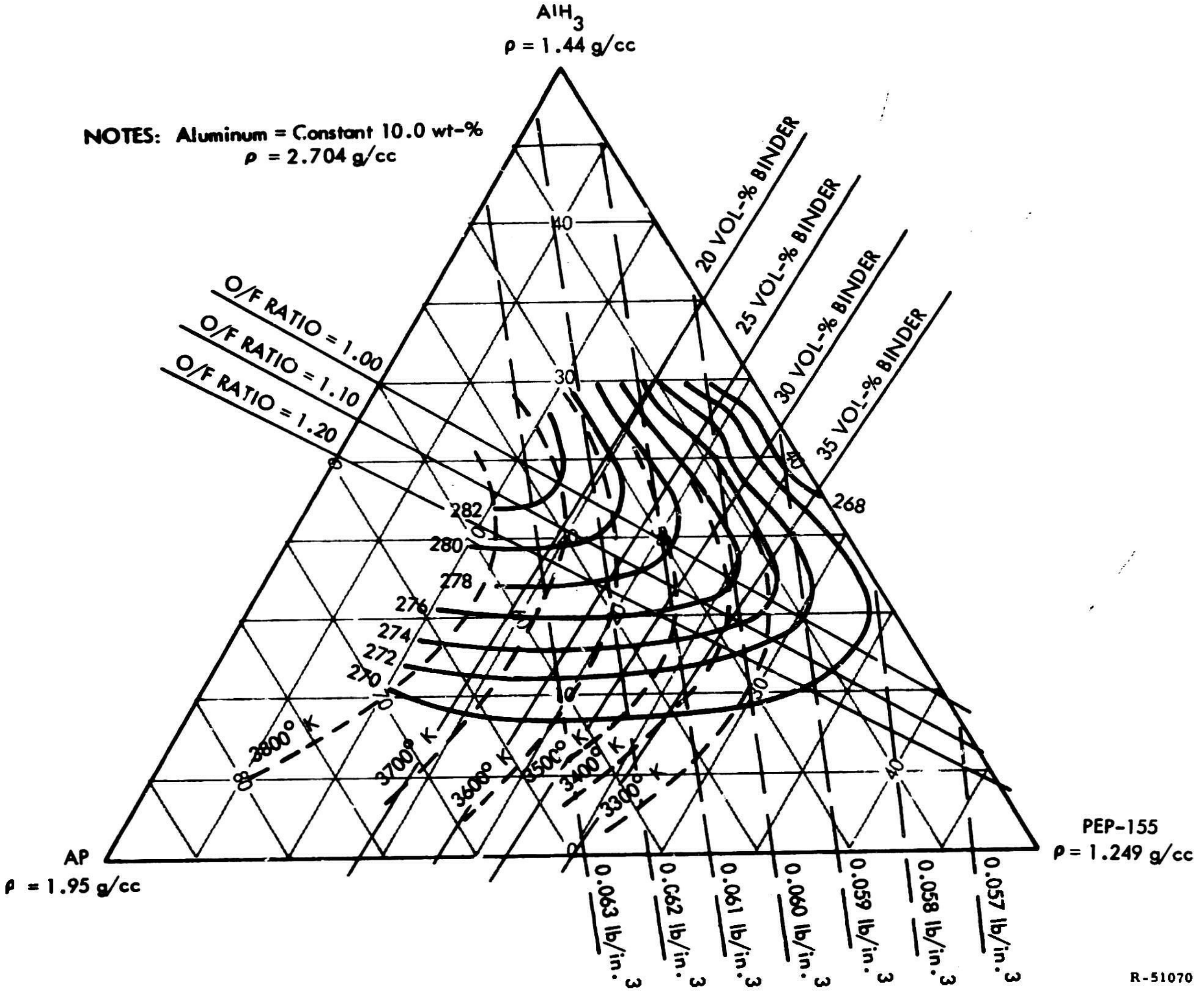
NOTE: PEP-455 = 55 wt-% trimethylolnitromethane trinitrate, 45 wt-% polyester HX-735

Figure 13. (C) Performance in the AlH_3/HAP/PEP-455 System



NOTES: Aluminum = Constant 5.0 wt-%
ρ = 2.704 g/cc

Figure 14. (C) Performance in the AlH$_3$/AP/PEP-155 System with a Constant 5% Aluminum Metal



NOTES: Aluminum = Constant 10.0 wt-%
ρ = 2.704 g/cc

Figure 15. (C) Performance in the AlH_3/AP/PEP-155 System with a Constant 10% Aluminum Metal

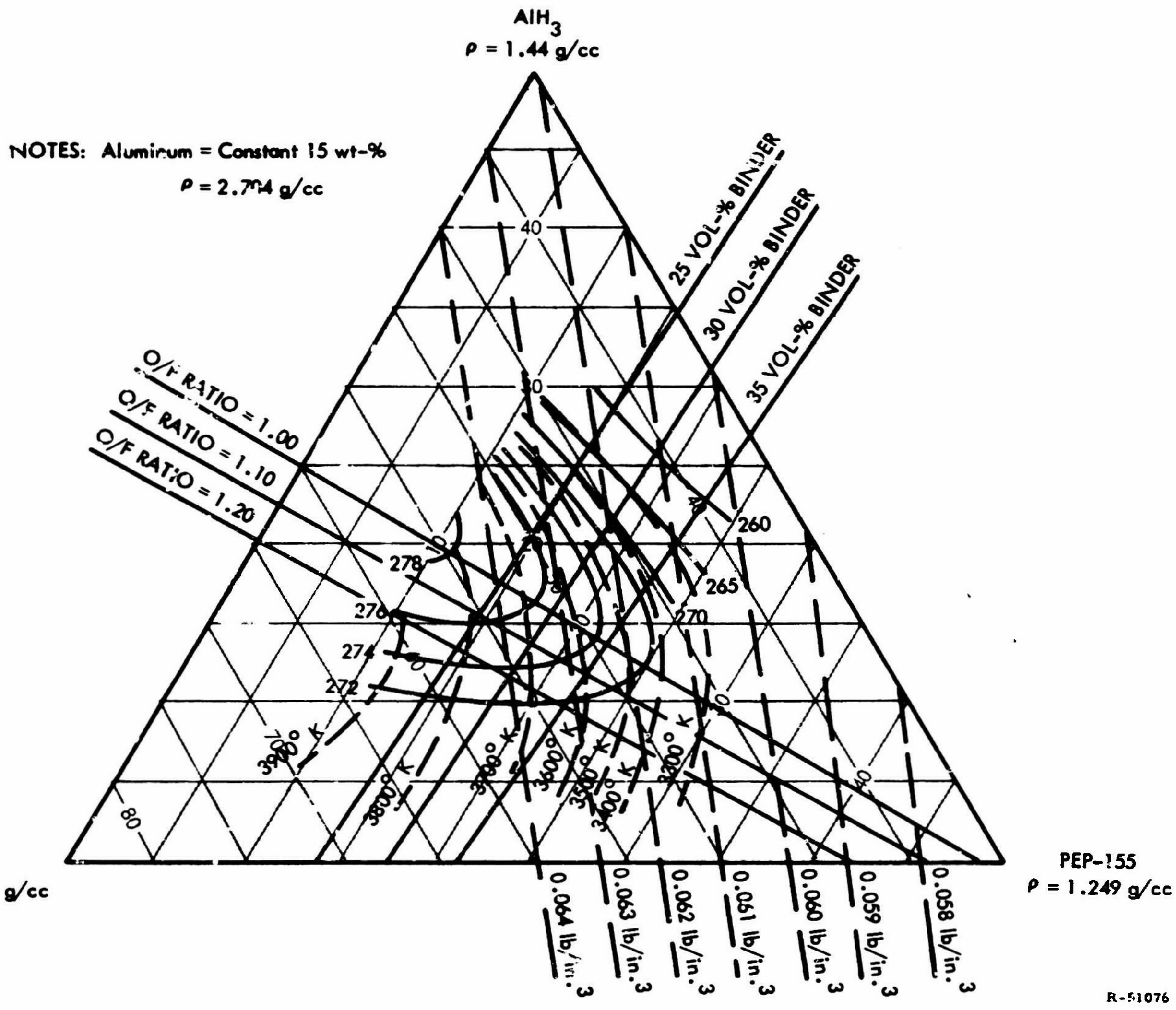


Figure 16. (C) Performance in the AlH_3/AP/PEP-155 System with a Constant 15% Aluminum Metal

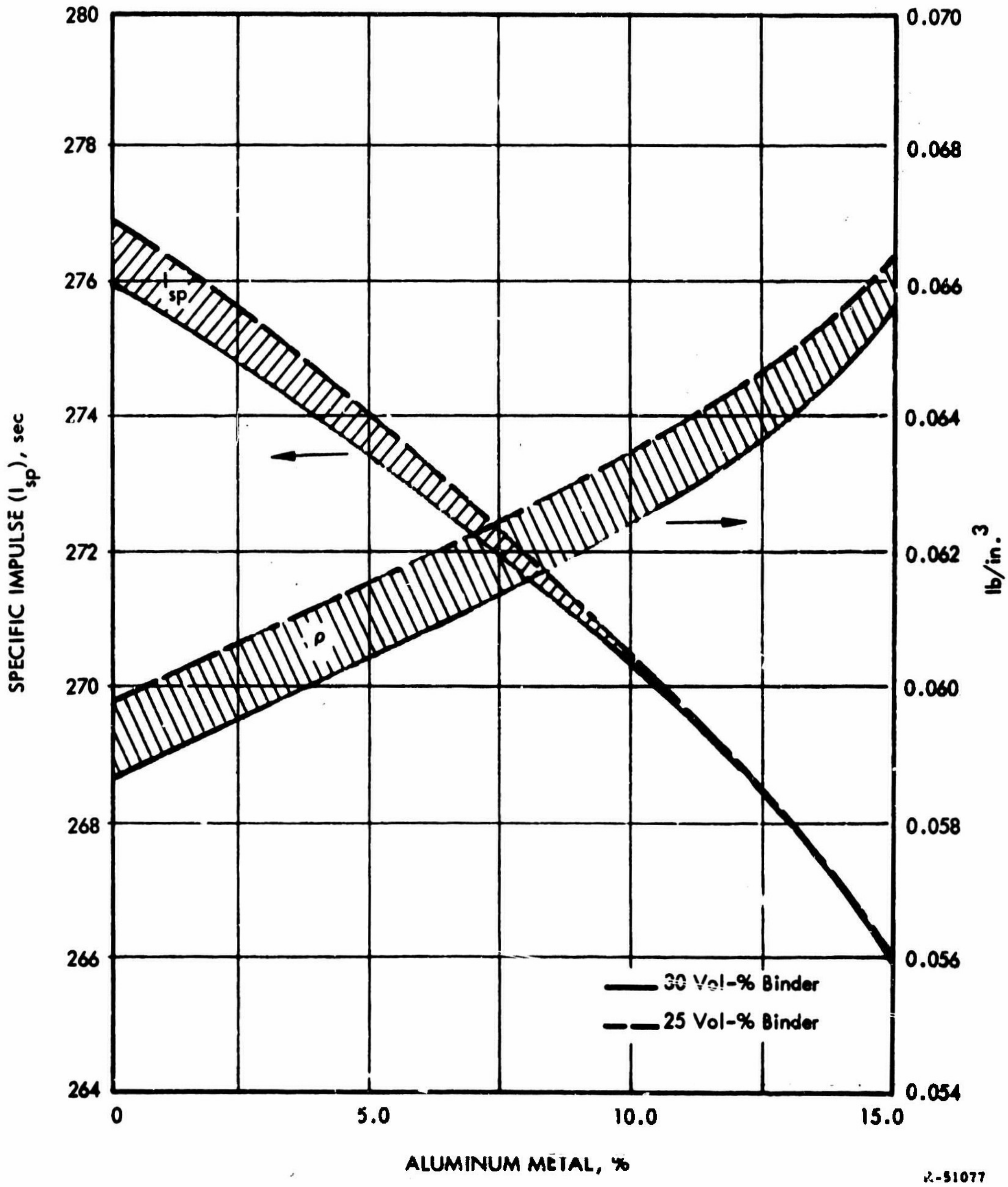


Figure 17. (U) Density-Performance Tradeoffs at Various Aluminum Metal Loadings for a Constant 18.0% Aluminum Analysis

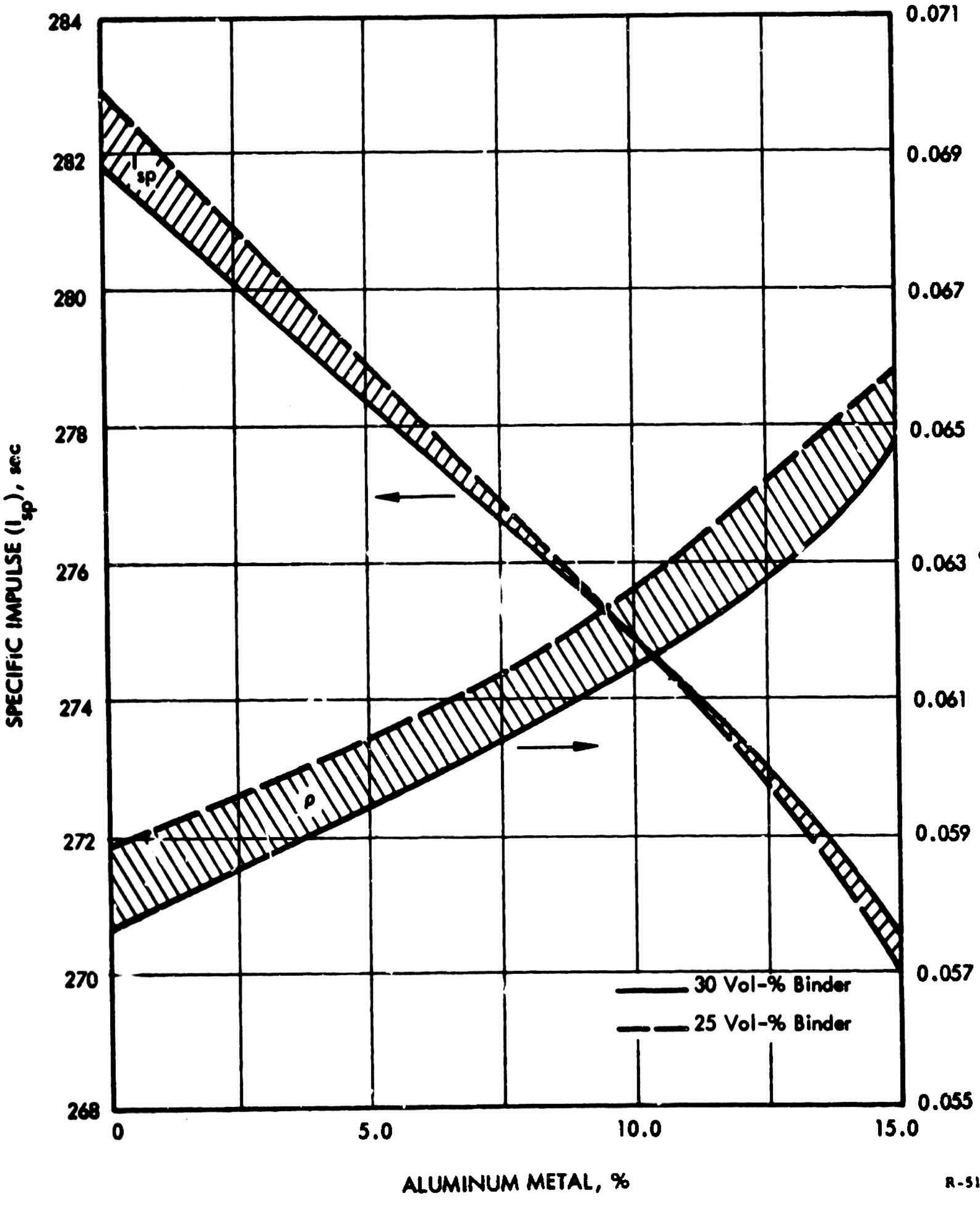


Figure 18. (U) Density-Performance Tradeoffs at Various Aluminum Metal Loadings for a Constant 22.5% Aluminum Analysis

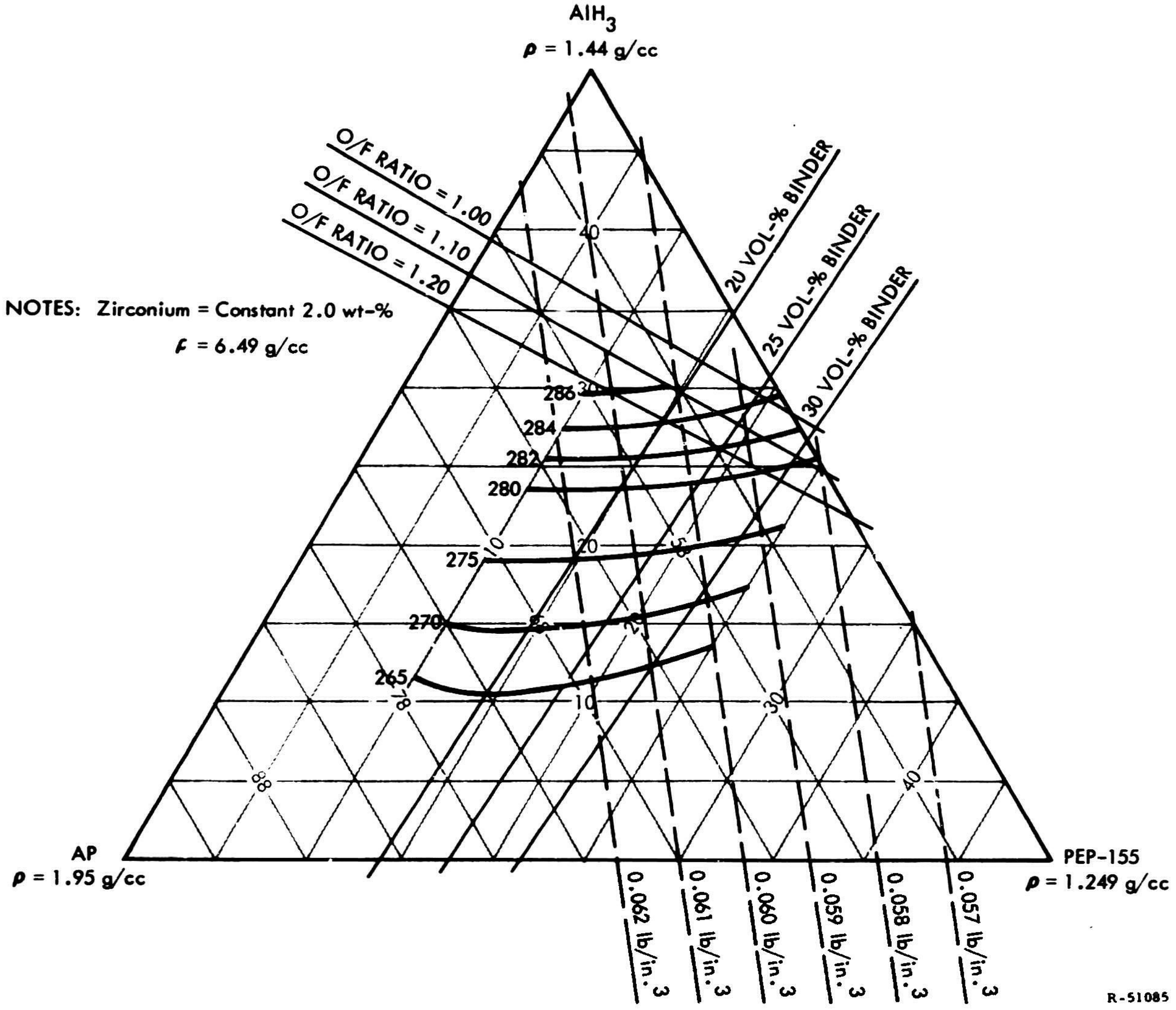
Two lines are shown for 25 vol-% binder and 30 vol-% binder to include the areas of maximum processability. In the compositions containing 18% metal, the first target impulse of 280 sec cannot be achieved. However, the second target impulse of 274 sec can be achieved at between 4.0% and 5.0% aluminum metal with a resulting density between 0.0602 and 0.0613 lb/in.^{3}

(C) In the compositions in figure 18 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 between 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 a constant 2% and 5% zirconium in the AlH_3/AP/PEP-155 system shows less favorable compositions. The ternaries are shown in figures 19 and 20. Although 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 21 in which I_{sp} and density are plotted against percent zirconium in a system whose metal analysis is 22.5%. The primary target of 280 sec is achieved at 0.9% to 1.2% 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-AlH_3 systems. For this reason, no experimental work with zirconium was undertaken.

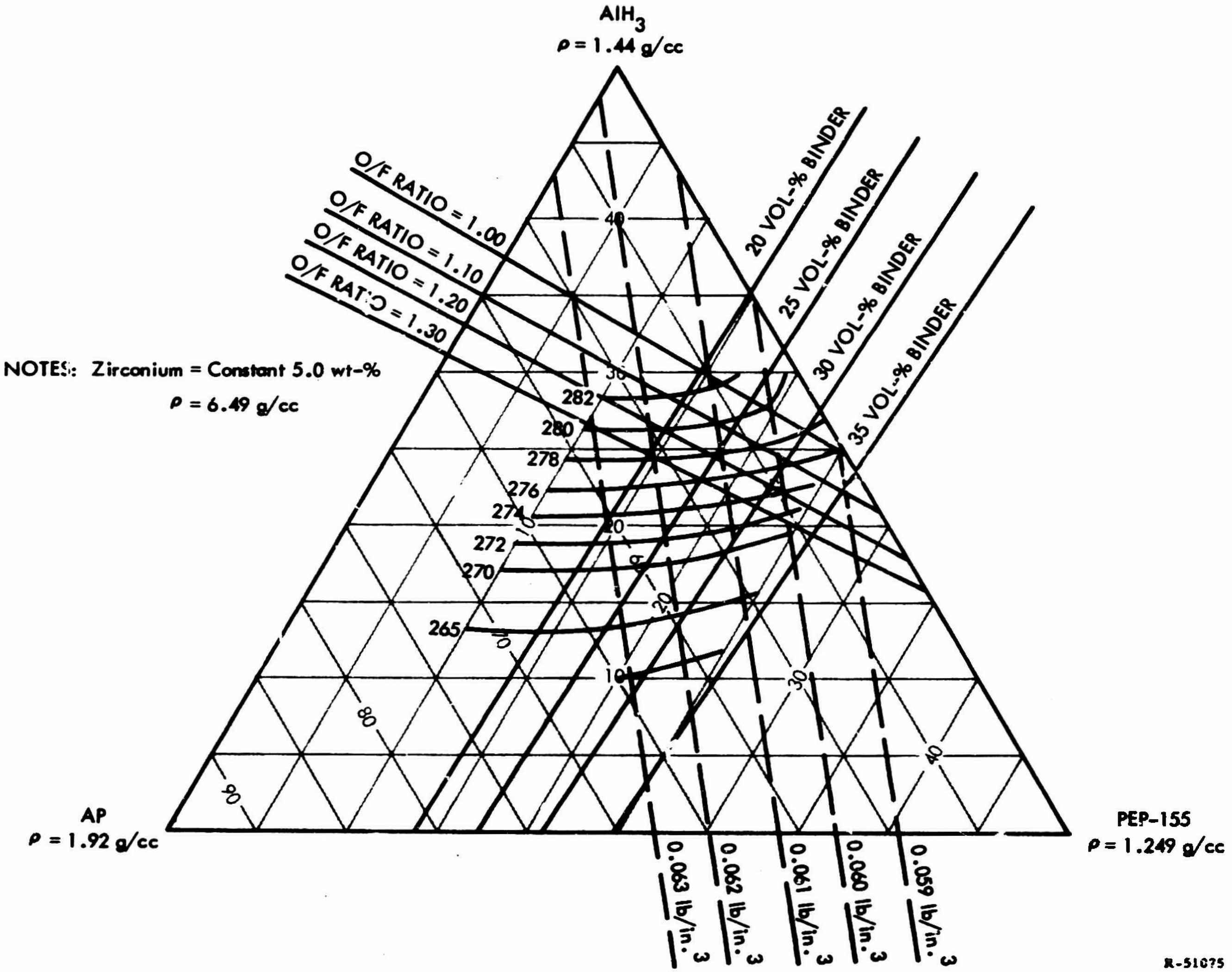
(e) Conclusions

(C) The gains in I_{sp} and density between the PEP-150 and the PEP-155 systems are sufficiently small such that physical property considerations favor the lower plasticizer level. The impulse and density gains with other nitrate plasticizers are small, but these and the



NOTES: Zirconium = Constant 2.0 wt-%
ρ = 6.49 g/cc

Figure 19. (C) Performance in the AlH$_3$/AP/PEP-155 System with a Constant 2% Zirconium Metal



NOTES: Zirconium = Constant 5.0 wt-%
ρ = 6.49 g/cc

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Figure 20. (C) Performance in the AlH_3/AP/PEP-155 System with a Constant 5% Zirconium Metal

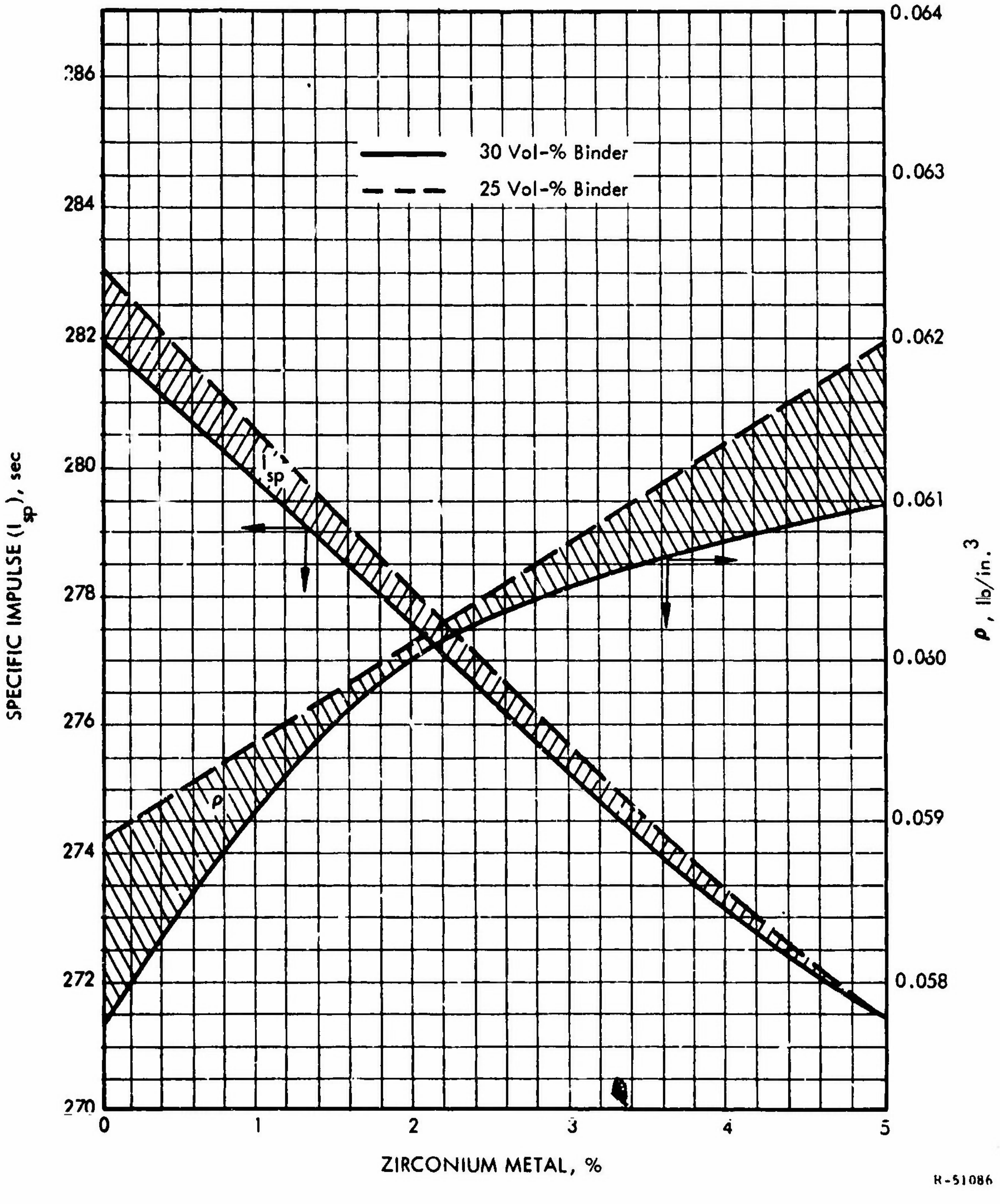

Figure 21. (C) Density-Performance Tradeoffs in Zirconium-AlH_3 Mixed Fuels

gain in O/F ratio argue for continued investigation of other plasticizers. The use of HAP in place of AP offers outstanding advantages in performance, density, and O/F ratio. Aluminum is superior to zirconium as a second fuel in achieving higher densities. It is probable that aluminum will show similar properties in the HAP-oxidized systems.

(2) Formulation and Processing Studies

(a) Evaluation of Processing Simulants for LMH-1

(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 AlH_3 was sought to decrease the cost of processing studies and to minimize the remote-handling requirements. A survey was made to find possible candidates, and outside vendors were contacted for crystalline materials of suitable particle shape, size, and density. However, only ammonium oxalate monohydrate was found to be a suitable material. Ammonium oxalate monohydrate was evaluated as a substitute for Dowane 1451 in a PEP-155 propellant system. This material has essentially the same crystal density as Dowane 1451 and is readily available at low cost. It can be ground and handled with no hazard.

(U) Screen analyses were made of two lots of Dowane 1451. Ground ammonium oxalate was classified on the same series of screens, and the various cuts were blended to duplicate the size distributions of the two lots of Dowane 1451. Two methods of grinding the oxalate were used (1) ball milling, and (2) grinding in a hammer mill (Bantam mikropulverizer). Ball milling resulted in round smooth particles, and grinding in a hammer mill yielded somewhat more jagged irregular-shaped particles. Viscosities of mixes containing the Dowane 1451 and the ammonium oxalate of comparable particle size distributions then were compared.

(C) The use of ball-milled material resulted in mixes having viscosities 50% less than the hydride mixes. The use of oxalate ground in the pulverizer also resulted in lower viscosities than the hydride mixes but somewhat closer than those obtained by ball milling. In mix UTC 6814-99, the particle size distribution was adjusted to increase the coarse fraction slightly. This adjustment resulted in essentially the same viscosity as the hydride control mix. Table I presents the screen analysis of the two lots of Dowane 1451 used in this study. Table II summarizes the data obtained on mix viscosities using ground ammonium oxalate.

TABLE I

(U) DOWANE 1451 PARTICLE SIZE DISTRIBUTION

	Wt-% Dowane 1451 Retained on Screen	
Screen Size	DL-458	Blend 27
246	0.0	0.0
147	77.69	3.10
104	17.32	43.90
74	4.69	40.96
61	0.30	8.55
43	---	3.23
0	---	0.26

TABLE II

(U) EFFECT OF FUEL SIMULANT TREATMENT ON MIX VISCOSITY

UTX-6814	Fuels or Simulant	Grinder	Viscosity Poises
59	DL-458		1,250
60	BL 27		2,000
85	DL-458 simulant	Ball mill	675
86	BL 27 simulant	Ball mill	925
97	DL-458 simulant	Mikropulverizer	1,650
98	BL 27 simulant	Mikropulverizer	1,100
99	DL-458 83%>147a 17%>104a	Mikropulverizer	1,150

(U) A brief evaluation of sugar crystals also was conducted as the crystal shape is basically the same as that of Dowane 1451. However, the resulting mix viscosities were substantially higher. No further work was conducted with this material.

(b) Particle Size Distribution Studies

(U) An experimental processing program was conducted to optimize particle size distribution. The primary objective of this study was the attainment of maximum solids loading in the propellant formulation.

(C) To reduce the number of possible particle size combinations, it was decided to use the AlH_3 in the particle sizes in which it is received and to vary the ratio of "coarse" oxidizer to "fine" oxidizer. The AlH_3 particles generally are considered to have an average particle size of about 100μ, although there is considerable variability from lot to lot. However, because larger mixes require blends of several lots, the mix-to-mix variation of AlH_3 particle size distributions is minimized. The AP size fractions which have been found to result in minimum mix viscosities in other propellant systems at UTC are the 300μ (+48 mesh) size and the "fine" 8μ to 12μ size.

(U) A series of mixes were processed using ammonium oxalate as a simulant for Dowane 1451. The propellant used 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. x 3.00-in. orifice at given rheometer pressures is shown at the various oxidizer size ratios in figure 22. These data are also presented as a function of coarse/fine particles by volume in which both the oxidizer and hydride are included. The simulated hydride is treated as all "coarse particles." This relationship is shown in figure 23.

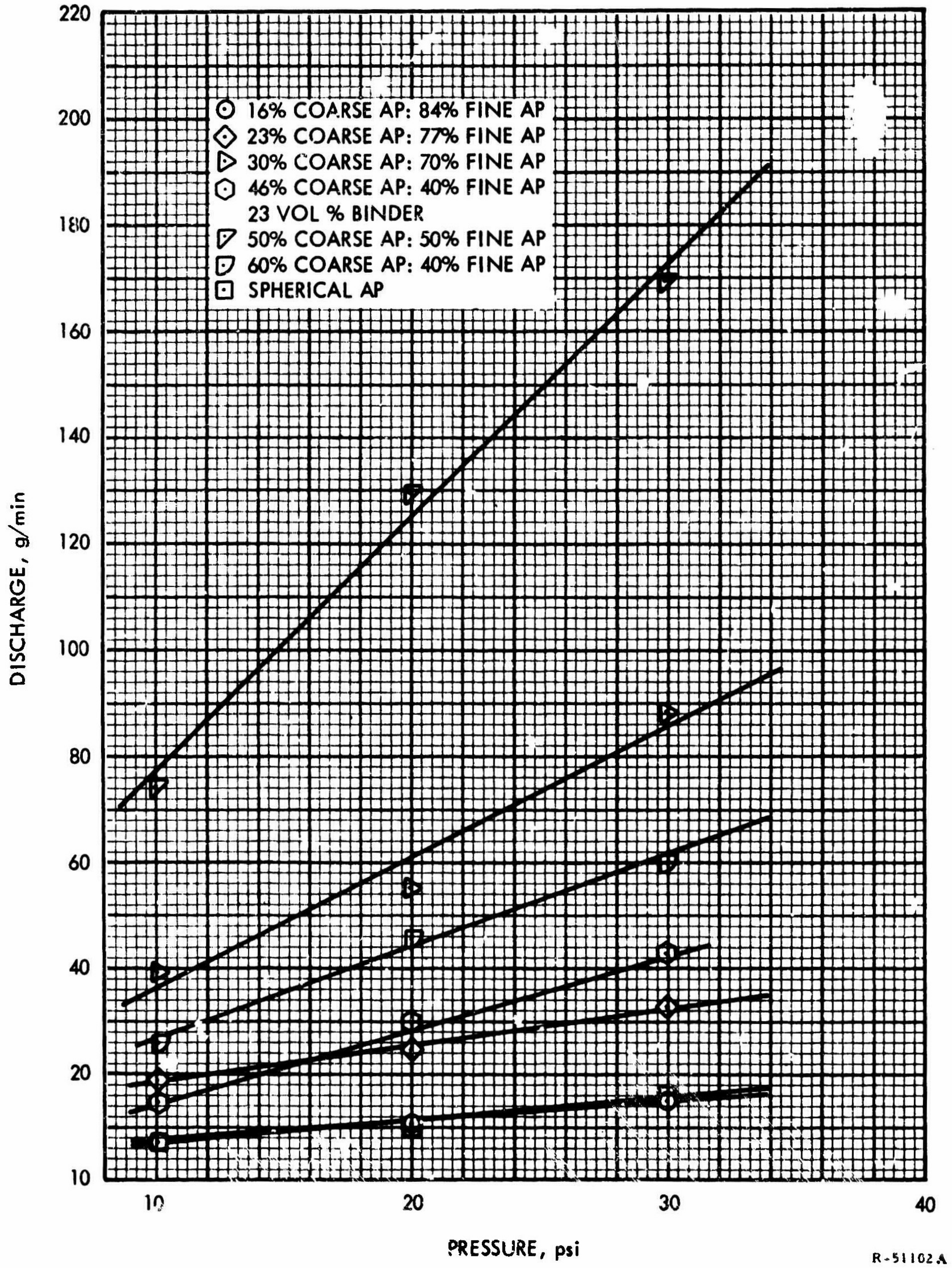

Figure 22. (U) Effect of Coarse/Fine Oxidizer Ratio on Castability of Simulated UTP 6822

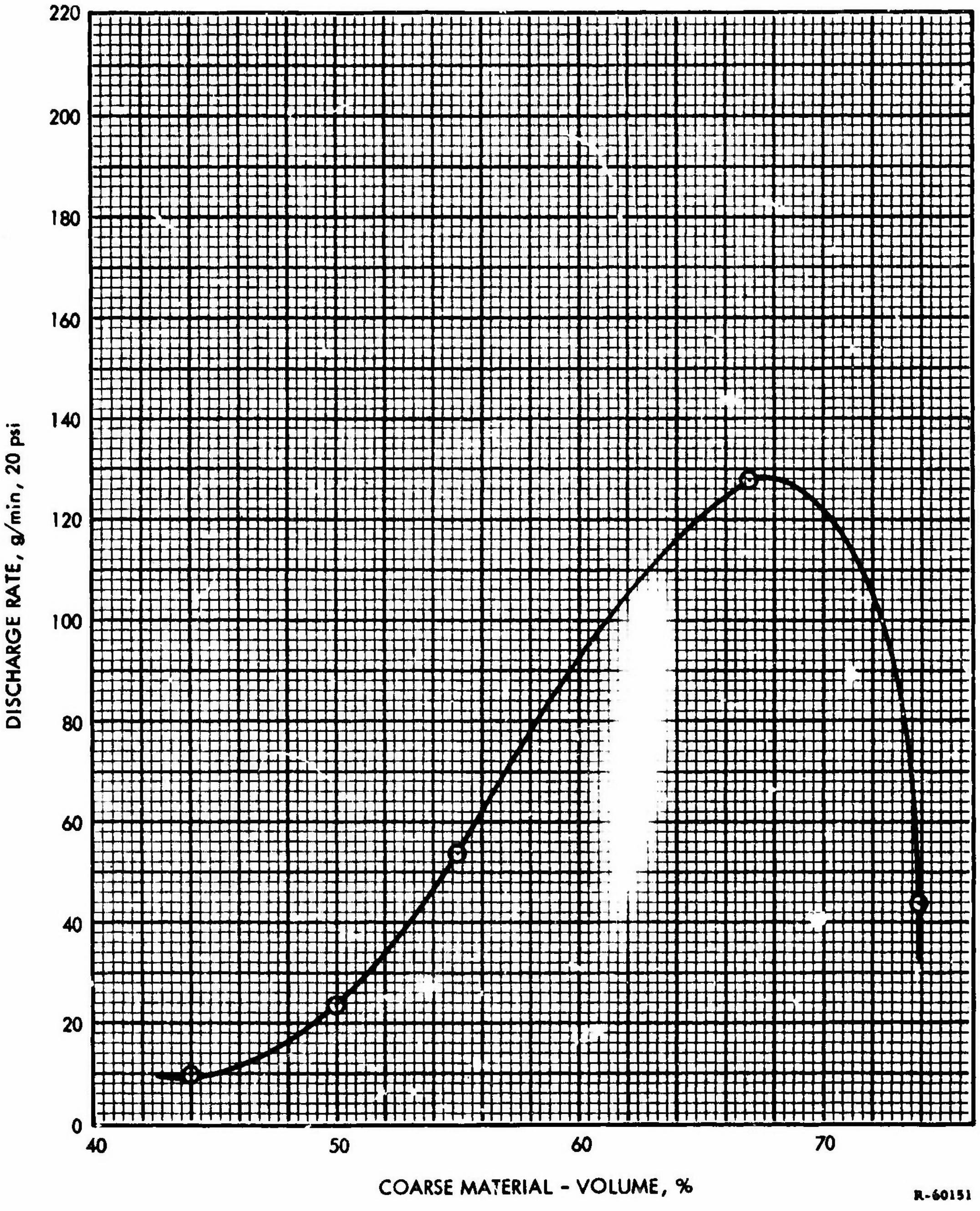

Figure 23. (U) Effect of Overall Coarse/Fine Ratio on Castability of Simulated UTP 6822

(U) An additional variable which is considered significant to propellant processability is the particle shape of the oxidizer. The fine oxidizer fractions used on Contract 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 22.

(c) Evaluation of Surfactants and Processing Aids

(U) In addition to the particle size distribution studies described in (b), a series of surfactants were evaluated for their effect on propellant mixing and casting viscosity to further increase propellant solids loadings.

1. Studies in HX-735 Binder System

(U) A number of different surfactants were evaluated with UTP-6814 premix at a concentration of 1%. The results are summarized in table III.

(U) Preliminary studies were based on the hydrophile-lipophile balance (HLB) of the surfactant, which is a system used by Atlas Chemical Co. to classify their surfactants. In this balance system, surfactants are classified according to the size and strength of the hydrophilic (water loving) and lipophilic (oil loving) groups in a molecule. An emulsifier that is lipophilic in nature is assigned a low HLB number (0 — 10), and an emulsifier that is hydrophilic in character is assigned a high number (10 — 20). If two or more emulsifiers are blended, the HLB values are intermediate.

TABLE III

(U) SURFACTANT STUDY DATA

Surfactant Name	Chemical Composition	HLB No. ±1	Age of Premix Days	Viscosity* Poise	Comments
UTX 6814 Premix (Control)			1	475	
Yelkins TSS	Lecithin derivative		1	500	Poor castability
UTX 6814 Premix (Control)				750	
Uteflo L & A-2 (50/50)	—			900	
UTX 6814 Premix (Control)			2	900	
Uteflo E-1	—		2	450	No cure
Atlas G-2410	—		2	350	
Atlas G-2421	—		2	325	
Atlas G-3300	Alkyl aryl-sulfonate	11.7	2	500	
Atlas G-3570	High molecular weight fatty amine blend	4.5	2	275	Poor cure
UTX 6814 Premix (Control)				550	
Yelkins TTS	Lecithin derivative		1	900	Poor castability
Span 20	Sorbitan monolaurate	8.6	1	538	
Tween 21	Polyoxyethylene (4) sorbitan monolaurate	13.3	1	275	
Tween 85	Polyoxyethylene (20) sorbitan trioleate	11.0	1	400	
Atlas G-2406	—		1	550	
Atlas G-2684	Blend sorbitan monooleate polyoxyethylene ester mixed acids	7.8	1	275	
Atlas G-3335	Alkyl aryl-sulfonate blended with polyoxyethylene sorbitan esters of mixed acids	13.0	1	400	
Tween 80	Polyoxyethylene (20) sorbitan monooleate	1[illegible].0	2	375	
UTX 6814 Premix (Control)			1	400	
Atlas G-1288	Polyoxyethylene fatty glyceride	16.0	1	460	
Atlas 8916T	Polyoxyethylene sorbitan esters of mixed acids	15.4	1	390	
Tween 40		6.7	1	425	
UTX 6814 Premix (Control)			1	1,400	
Tween 20	Sorbitan monolaurate	8.6	1	1,000	

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* Brookfield viscometer, T-F spindle, 120° F, 20 rpm.

(U) The data obtained in this study indicate that the chemical composition of the surfactant is more important than HLB value in lowering premix viscosities. For example, Tween 80 (polyoxyethylene [20] sorbitan monooleate), reduced the viscosity more than Tween 85 (polyoxyethylene [20] sorbitan trioleate). It appears that the number of long-chained alkyl groups present in ethylene oxide-derived mono-ionic surfactants should be kept at a minimum. The number of ethoxy groups on a molecule also appears important. Tween 21, (polyoxyethylene [4], sorbitan monolaureate) lowered the premix viscosity more than Span 20 (sorbitan monolaureate).

(U) In some instances, it was noticed that the addition of surfactants adversely altered the castability and cure characteristics of UTX-6814 premix. For example, both Yelkins TTS and TSS reduced the premix castability. Some premixes have been observed to cure more than others to yield final cured propellants which were quite hard and brittle. A very soft cure was obtained with Shell El surfactant.

(U) Results of the laboratory investigation indicated that the following surfactants yielded significant processing improvements and were evaluated 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
Dowane 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. x 3.00-in. rheometer orifice for mixes with the candidate surfactants are shown in figure 24. Atlas G-2684 is considered the standard surfactant with HX-735 at a concentration of 0.1 wt-% based on the total formulation.

2. Studies in the Uteflex Binder System

(U) As a result of the extensive evaluation of polymers and curatives conducted for the purpose of achieving improved mechanical properties (see section e), the binder system was changed to a UTC-proprietary polymer (a carboxyl-terminated polyether) designated Uteflex. The curative system is based on HX-874 (a highly reactive aziridine) and Epon 812. Preliminary processing studies indicated that this system could not be processed at the 25 vol-% binder level, although the same solids loading was readily processed in formulations based on the HX-735/MAPO/Epon 812 binder system.

(U) A number of candidate additives, selected on the basis of previous experience in other systems or because of their solubility, were evaluated for their ability to reduce mix viscosity in the Uteflex system. Materials were added to a Uteflex/TMETN/AP mix at a concentration of 1%. Rheometer flow rates were obtained as follows:

Additive 1%	Rheometer g/min	Additive 1%	Rheometer g/min
Phenyl glycidal ether	85.5	MAPO	25.9
Dioctyl adipate	77.6	Phenyl MAPO	21.5
Tricresyl phosphate	69.0	Control (no additive)	19.2
Pluronic L-64	58.4	TBM	5.0
Pluronic L-35	53.0		

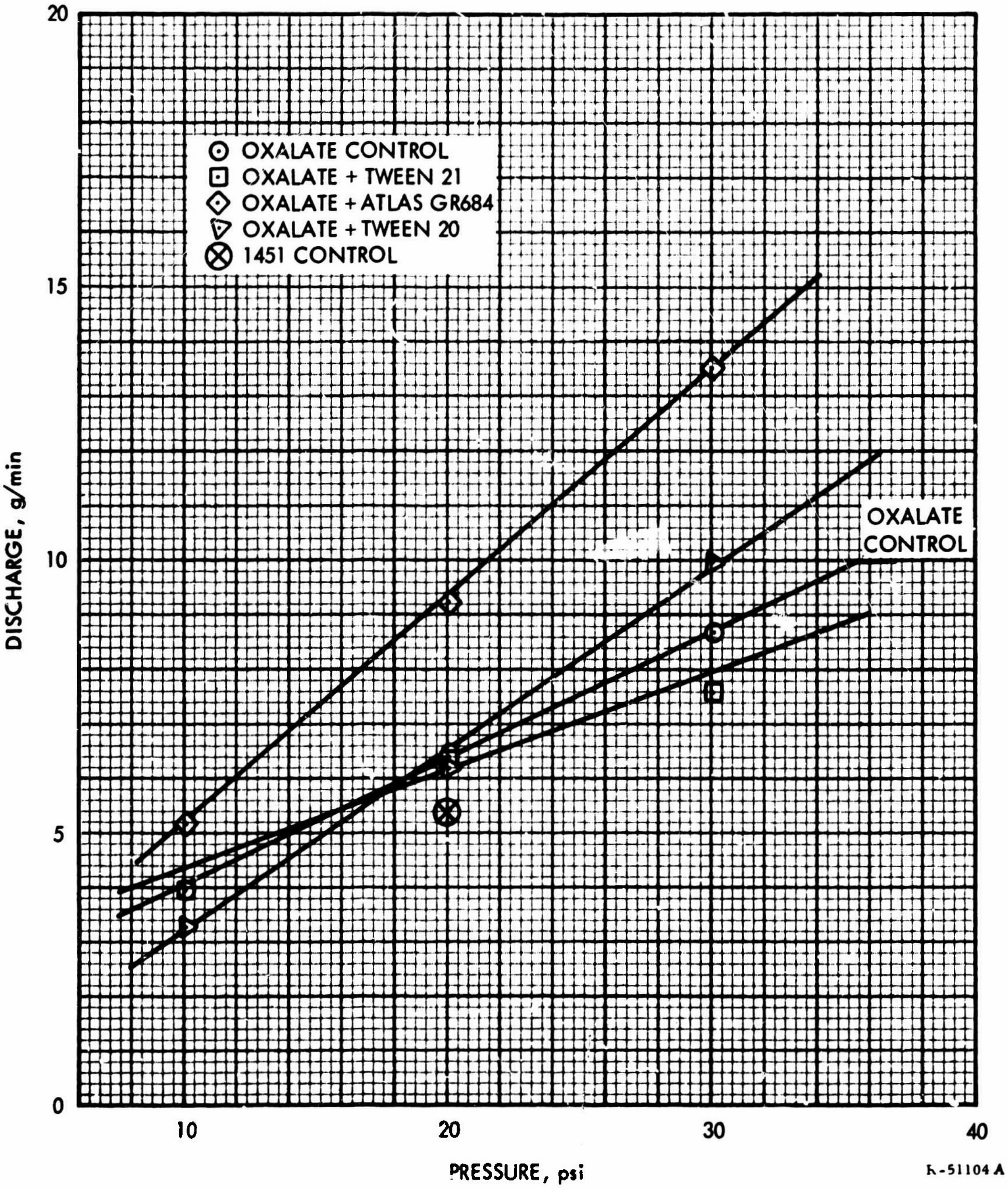

Figure 24. (U) Effect of Surfactants on Viscosity of Simulated UTP 6822

(U) A decision was made to use DOA at a concentration of 1/2% to achieve the required reduction in propellant mix viscosity. Phenyl glycidal ether was not used because of the possible effects of the monofunctional epoxides on propellant mechanical properties.

(3) Evaluation of Nitrato-Ester Plasticizers

(C) An extensive theoretical treatment of the effect of other nitrate plasticizers on the performance of the AP-oxidized AlH_3 system is presented in section (1), Theoretical Studies, for propellants formulated at the 55 wt-% plasticizer level. The plasticizers investigated as replacements for trimethylolethane trinitrate (TMETN) are 1, 2, 4-butanetriol trinitrate (BBTN), 2, 2-dinitro-propylnitrate (DNPN), and trimethylolnitromethane trinitrate (TMNTN or NIB-nitroglycerine). The ternary performance diagrams of these systems are shown in figures 8, 9, and 10.

(C) For purposes of comparing the effects of the three new plasticizers, the intersection of the 25 vol-% binder life with the 25 wt-% AlH_3 life was selected on each of the appropriate ternary diagrams. The results are summarized in table IV.

(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 was decided to maintain a plasticizer level of 50 wt-% rather than 55 wt-%. Further processing studies 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-% AlH_3. The results are compared in table V. As an additional point of comparison, the effect of nitroglycerin is also presented.

(U) Laboratory evaluation tests have been conducted with all of the candidate nitrato-ester plasticizers.

TABLE IV

(C) EFFECT OF NITRATO ESTER PLASTICIZERS ON PERFORMANCE

(55% Plasticizer in Binder, 25 vol-% Binder, 25 wt-% AlH_3)

Plasticizer	Theoretical I_{sp}	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 V

(C) EFFECT OF NITRATO ESTER PLASTICIZERS ON PERFORMANCE

(50% Plasticizer in Binder, 23 vol-% Binder, 25 wt-% AlH_3)

Plasticizer	Theoretical I_{sp}	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

(C) Butane triol trinitrate (BTTN) is shipped [illegible] cept for 1 wt-% of NDPA stabilizer. Tables VI and [illegible] ent the impact and DTA data for BTTN. Butane triol trinitrate had a lower impact value than TMETN but decomposed thermally at the same point as the other nitrate esters, TMETN, TGDN, or DEGDN. Butane triol trinitrate was found to be miscible with HX-735 at the 50 wt-% level, but at 60 wt-% plasticizer the BTTN was immiscible. Because of the greater impact sensitivity of BTTN and its marginal miscibility in the range of interest, compared with its very slight improvement in theoretical performance, butane triol trinitrate does not appear attractive as a substitute for TMETN.

TABLE VI

(U) BTTN IMPACT TEST DATA

Sample	Test Container	Positive	Negative
99% TMETN/1% ethyl centralite	Liquid	3. 5 kg-cm	3
99% BTTN/1% 2NDPA	Liquid	2. 5	2
50% BTTN/50% HX-735	Solid	Impact >250	---
23% BTTN/77% 12μ AP	Solid	16	15
50% BTTN/50% Epon 812	---	Impact >250	---

(C) A DTA made on DNPN exhibited an exotherm peak 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 MAPO in a polyethylene beaker. The mixture ignited shortly after mixing. No further tests were made with DNPN.

(C) Initial studies with TMNTN (or NIB-TN) were made with a 10-g sample obtained from Propellex Chemical Division of the Chromalloy Corporation. The plasticizer was miscible with HX-735 polymer in all proportions.

TABLE VII

(U) BTTN DTA DATA*

Run No.	Sample	Exotherm, C Onset	Exotherm, C Peak
816	99% BTTN/1% 2NDPA	148	174
817	30% BTTN/50% HX-735	143	178 boils
818	Same as above, only greater sample weight	136	172 boils
820	23% BTTN/77% AP	136	162 sharp exotherm
821	94% BTTN/6% Epon 812	131	172 boils
83	TEDGN, neat	---	171
111	TMETN, neat	---	161
340	DEGDN, neat	---	167

* Al_2O_3 reference material, 4.6° C/min heating rate, iron-constantan thermocouple, normal atmosphere, aluminum block.

However, several small-scale mixes formulated with TMNTN at the 50 wt-% plasticizer level did not cure. Because this sample of TMNTN had been synthesized approximately 2 years previously and a sufficient quantity was not available for more than a cursory evaluation, a freshly manufactured lot was obtained from Trojan Powder Company.

(C) The TMNTN is shipped with 20 wt-% acetone and 2 wt-% ethyl centralite. No attempts were made to purify the as-received TMNTN. The impact value for the diluted plasticizer was 7.0 kg-cm and the stabilized sample, TMNTN 98% + ethyl centralite 2%, was 3.0 kg-cm. The TMNTN was found to be soluble in Uteflex up to approximately 70 wt-%. The TMNTN content was corrected for 2.5% of ethyl centralite.

(C) In addition to the ethyl centralite which was contained in the TMETN, resorcinol was added in half of the cure studies made. Table VIII lists the results of various formulations which were studied. All of the Uteflex/TMNTN formulations turned dark brown and did not cure. A comparison Uteflex/TMETN sample gelled, but was yellow-green in color. A nonplasticized Uteflex formulation cured into a tan colored elastic sample. TBM and HX-874, the same curative from two different vendors, were both used in these tests, but no differences in results were observed. All samples were cured at 120° F for 24 hr.

(U) A gel time measurement made on a Sunshine Scientific Instrument gel time meter at 90° C on formulation LA37-1 in table VIII resulted in bursting of the sample tube at 166.8 min. The splattered sample material appeared to be a well cured elastic.

(U) A series of compatibility tests was made in which the samples were exposed to 120° F for 24 hr. Methyl violet paper was placed at the top of the test tube for detection of possible nitric oxide or nitric acid degradation products. The obvious color changes and/or burning of the samples indicated that TMNTN was incompatible with any tetrabutylene melamine, MAPO, Uteflex, and possible with resorcinol and HX-735. The results are summarized in table IX.

(U) The results of a series of DTA runs of TMNTN with TBM, Uteflex and MAPO, presented in table X, indicated some incompatibility between MAPO and TMNTN.

(U) As a result of the preliminary laboratory evaluation of the nitrato-ester plasticizers and the theoretical performances of these materials in the systems considered, it is evident that the development work required to make the use of any of these materials practical is beyond the scope of this program. However, because of the favorable solubility properties of TMNTN as well as its density and theoretical performance, it should still be considered as a possible candidate in a nitrato-ester plasticized system.

TABLE VIII

(U) CURE FORMULATIONS (120° F for 24 hr)

Formulation No.	LA36-1	LA36-2	LA36-3	LA36-4	LA36-5	LA36-6	LA36-7	LA36-8	LA36-9	LA38-2
Uteflex	44.42	35.54	53.04	44.29	35.43	53.15	43.83	35.07	52.60	41.90
TBM	3.72	2.97	4.46	---	---	---	---	---	---	3.51
HX-874	---	---	---	3.85	3.08	4.62	4.33	3.46	5.20	---
Epon 812	1.86	1.49	2.23	1.86	1.49	2.23	1.84	1.47	2.20	1.75
Ethyl Centralite	1.25	1.50	1.00	1.25	1.50	1.00	1.25	1.50	1.00	1.25
Resorcinol	---	---	---	---	---	---	---	---	---	2.84
TMNTN	48.75	58.50	39.00	48.75	58.50	39.00	48.75	58.50	39.00	48.75
TMETN	---	---	---	---	---	---	---	---	---	---
Melamine/Uteflex Equivalents Ratio	1.50	1.50	1.50	1.50	1.50	1.50	1.70	1.70	1.70	1.50
Epon 812/Uteflex Equivalents Ratio	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
Remarks:										
Color	Dark Brown	Dark Brown	Dark Brown	Dark Brown	Dark Brown	Dark Brown	Dark Brown	Dark Brown	Dark Brown	Dark Brown
Cure	No	No	No	No	No	No	No	No	No	No

Formulation No.	LA38-3	LA38-4	LA38-5	LA38-6	LA38-7	LA38-8	LA38-9	LA38-10	LA38-11	LA37-1
Uteflex	33.52	50.28	41.90	83.80	41.35	33.08	49.62	41.35	82.70	88.58
TBM	2.81	4.21	3.51	7.02	---	---	---	---	8.16	---
HX-874	---	---	---	---	4.08	3.27	4.90	4.08	---	7.71
Epon 812	1.40	2.10	1.75	3.50	1.73	1.38	2.07	1.73	3.46	3.71
Ethyl Centralite	1.50	1.00	0.50	---	1.25	1.50	1.00	0.50	---	---
Resorcinol	2.27	3.41	2.84	5.68	2.84	2.27	3.41	2.84	5.68	---
TMNTN	58.50	39.00	---	---	48.75	58.50	39.00	---	---	---
TMETN	---	---	49.50	---	---	---	---	49.50	---	---
Melamine/Uteflex Equivalents Ratio	1.50	1.50	1.50	1.50	1.70	1.70	1.70	1.70	1.70	1.50
Epon 812/Uteflex Equivalents Ratio	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
Remarks:										
Color	Dark Brown	Dark Brown	Yellow Green	Tan	Dark Brown	Dark Brown	Dark Brown	Yellow Green	Tan	Tan
Cure	[illegible]	No	Gel	Yes	No	No	No	Gel	Yes	Yes 166.8 min

TABLE IX

(U) COMPATIBILITY MIXTURES

Mixtures	Results	Methyl Violet Paper
TMNTN + 2.5% E. C.*	No change observed	Negative result
TMNTN + HX-874	Ignited, blew out cork Color of residue – dark brown	Negative result
TMNTN + TBM	Ignited, blew out cork Color of residue – dark brown	Negative result
TMNTN + Epon 812	No change observed	Negative result
TMNTN + MAPO	MAPO gelled into dark brown mess	Brownish on edges
TMNTN + Uteflex	Dark green color	Negative
TMNTN + HX-735	Yellow-green cast	Negative
TMNTN + Resorcinol	Red	Negative
TMETN, Neat	No change observed	Negative
TMETN + HX-874	Yellow	Negative
TMETN + TBM	Yellow	Negative
TMETN + MAPO	No change observed	Brownish on edges
TMETN + Uteflex	Deep yellow	Negative
TMETN + HX-735	No change observed	Negative

---

* All TMNTN and TMETN samples contain 2.5 and 1.0% Ethyl Centralite.

TABLE X

(U) DTA RESULTS

	Exotherm Onset °C	First Exotherm	Second Exotherm	Third Exotherm
TMNTN + 2.5% ethyl centralite	122	149	165	180,186
TMNTN + ethyl centralite + TBM(35%)	(Small exotherm at 82°)	139	146	160,171,179
TMNTN + ethyl centralite + Uteflex	123	141	159	174
TMNTN + ethyl centralite + MAPO	89	106	107.5	151

(4) Characterization of Processing Parameters

(U) To obtain satisfactory processing of the propellant formulations, it was necessary to characterize the operating variables. Operating variables may be classed as follows:

- (a) **Raw material preparation**
- (b) **Mixing**
  - Time
  - Temperature
  - Order of addition
  - Pressure
- (c) **Casting**
  - Temperature
  - Pressure
- (d) **Precure conditioning**
  - Vibration
  - Pressure
- (e) **Cure**
  - Time
  - Temperature

(a) Raw Material Preparation

(U) All materials are rigorously dried. The polymer is stripped of low molecular weight impurities. Oxidizer is sized (300μ material is procured to specification; 7μ material is ground). Weighup of materials is carried out under conditions which prevent contamination and pickup of water.

(b) Mixing

(U) The mixing operation is designed not only to combine the various ingredients, but to result in a castable material of maximum density. It has been found that the order of addition of the materials has a significant effect, particularly on the porosity of the resulting grains. The primary factor appears to be a degassing of the LMH-1. To achieve this, the polymer, Epon, plasticizers, and stabilizers are mixed at ambient temperature under vacuum for 15 min, the LMH-1 is added and the mix continued for 30 min (temperature raised to 120° F). This mixture is held overnight under vacuum, the temperature set at 90° F. The following day, after a 15-min premix, the pressure is raised to ambient and the curative added. The temperature is then raised to 120° F, vacuum again applied, and the batch mixed for 5 min. The blended oxidizer is then added in a number of increments (incremental addition prevents overloading the mixer) over about a 15-min period, the system is maintained under vacuum. A final 30-min mix at 120° F under vacuum completes the mixing cycle.

(c) Casting

(U) The propellant is cast directly from the mixer to a casting cart which contains the molds. The casting hardware is preheated, but no provision is made for heating during casting. Casting is accomplished under vacuum.

(d) Precure Conditioning

(U) The cast material is vibrated under vacuum for a period of 4 to 6 hr. The casting cart is heated to 120° F. The period of vibration and vacuum depends upon the degree of swelling shown by the cast material.

(e) Cure

(U) Cure is for 24 hr at 120° F. Although some post-cure has been observed in samples with higher equivalence ratios, the normal cure time is restricted to 24 hr in the interest of achieving higher density propellant. Experiments with two temperature cycles appear promising, but no definitive statement can be made at this time.

(5) Results of Motor Testing

(U) The initial processing studies in the PEP-150 propellant system (based on HX-735 polymer) used ammonium oxalate monohydrate as a fuel simulant to study the effects of surfactants and the effects of particle size distribution on processing characteristics. As a result of these studies it was concluded that a propellant system formulated at 23 vol-% binder was feasible.

(C) The AlH_3 propellant system (UTP-6814) scaled up in the previous program, AF 04(611)-9570, was formulated at 32 vol-% binder. The reduction in binder content is significant as both density and theoretical specific impulse increase with solids loading. In order to achieve maximum delivered impulse at this loading, a series of mixes was processed to evaluate the tradeoff between O/F ratio, AlH_3 content, and theoretical specific impulse in the following formulations:

Formulation No.	O/F Ratio	AlH_3 %	Theoretical I_{sp}, sec	Theoretical Density lb/in.^{3}
UTP-6833	1.2	27.5	285.6	0.0587
UTP-6834	1.3	26.0	284.0	0.0590
UTP-6835	1.4	24.8	282.8	0.0592

The mix containing the highest level of AlH_3, UTP-6833, was not sufficiently fluid to cast into 4-lb motors (0.6-in. web). The other two formulations were evaluated in 4-lb motors (2 each) and 10-lb motors (3 each). Results are presented in table XI. The correction of specific impulse values from I_{sp} (P_c, 0°) to I_{sp} (1,000, 0°) was carried out by two methods. The values resulting from both methods are presented. Method A is the classical C_f correction using an assumed γ value of 1.18. In method B $I_{sp}\,(\mathrm{cor})_{1,000/14.7,\,0^\circ} = I_{sp}\,\mathrm{eff} \times I_{sp}\,(\mathrm{theo})_{1,000/14.7,\,0^\circ}$ where

$$I_{sp}\ \mathrm{eff} = \frac{\mathrm{meas}\ I_{sp}}{\mathrm{theo}\ I_{sp}\ \mathrm{at\ measured}\ P_c\ \mathrm{and}\ \epsilon}$$

TABLE XI

(U) PROJECT 2146 – PHASE I, TASK I, MA

Test No.	Formulation No.	T_c ° K	r_b in./sec	P_{c_a} psia	P_{c_b} psia	c* ft/sec	Theoretical c* ft/sec	c* Efficiency %	D
								4-lb Motors	
248	6834	3,389	0.473	585.6	612.3	5,343	5,576	95.8	
250	6834	3,431	0.505	971.4	1,021.1	5,542	5,590	99.1	
						5,543		97.5	
252	6835	3,462	0.800	1,222.2	1,353.4	5,493	5,577	98.4	
254	6835	3,467	1.021	1,298	1,457.2	5,327	5,579	95.5	
						5,410		97.0	
247	3001	3,404	0.401	1,154.7	1,183.8	5,128	5,170	99.2	
249	3001	3,413	0.389	1,046.9	1,073.2	5,073	5,181	97.9	
251	3001	3,410	0.387	1,010.4	1,043.1	4,972	5,179	96.0	
253	3001	3,409	0.387	996.8	1,037.2	4,992	5,179	96.4	
						5,041		97.4	
								10-lb Motors	
270	6834	3,425	0.477	904.9	948.6	5,419	5,588	97.0	
272	6834	3,427	0.453	926.9	959.8	5,425	5,589	97.1	
274	6834	3,430	0.469	963.5	999.5	5,326	5,590	95.3	
						5,390		96.5	
265	6835	3,398	0.429	564.4	597.5	5,393	5,555	97.1	
267	6835	3,423	0.443	754.6	795.1	5,417	5,565	97.3	
268	6835	←					Malfunction of test sta		
						5,405		97.2	
266	3001	3,390	0.378	956.5	990.2	5,086	5,165	98.5	
269	3001	3,390	0.379	956.8	980.5	5,026	5,165	97.3	
271	3001	3,388	0.377	934.0	969.4	5,048	5,164	97.8	
273	3001	3,385	0.373	894.6	940.4	5,020	5,163	97.2	
						5,045		97.7	

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* Long tailoff

TABLE XI

PHASE I, TASK I, MAXIMUM I_{sp} STUDIES

oretical c* /sec	c* Efficiency %	Specific Impulse Delivered P_{c_a}	Corrected to 0° at P_c	Corrected 1,000/0° (A)	Corrected 1,000/0° (B)	Theoretical at Test Condition	Efficiency %
4-lb Motors							
, 576	95.8	232.4	236.4	248.4	250.2	268.3	88.1
, 590	99.1	245.7	249.9	250.4	250.5	283.2	88.2
	97.5			249.4	250.4		88.2
, 577	98.4	256.1	260.5	256.0	256.3	287.6	90.6
, 579	95.5	248.1	252.3	247.3	247.5	288.5	87.5*
	97.0			251.7	251.9		89.1
, 170	99.2	236.2	240.2	237.6	238.0	264.0	91.0
, 181	97.9	236.3	240.3	239.6	239.8	262.1	91.7
, 179	96.0	234.9	238.9	239.1	242.9	257.1	92.9
, 179	96.4	240.0	244.1	244.6	245.0	260.5	93.7
	97.4			240.2	241.4		92.3
10-lb Motors							
, 588	97.0	245.1	249.3	251.5	252.8	280.0	89.0
, 589	97.1	245.6	249.8	251.5	252.5	280.9	88.9
, 590	95.3	245.8	250.0	251.0	251.9	282.0	88.7
	96.5			251.3	252.4		88.9
, 555	97.1	226.6	230.5	244.8	245.8	265.3	86.9
, 565	97.3	237.8	241.8	247.8	249.5	274.3	88.2
Malfunction of test stand lock, no data →							
	97.2			246.3	247.7		87.6
, 165	98.5	228.4	232.3	233.0	233.5	260.2	89.3
, 165	97.3	231.1	235.0	235.8	236.1	260.3	90.3
, 164	97.8	231.5	235.4	236.7	236.9	259.7	90.6
, 163	97.2	229.3	233.2	235.3	235.6	258.8	90.1
	97.7			235.2	235.5		90.1

(C) As the result of an extensive evaluation of the effect of aluminum addition on specific impulse and density, two propellant formulations containing AlH_3/aluminum combinations were selected 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^3	0.0604	0.0627
$I_{sp} \cdot \rho^{0.3}$, g-sec/cc	330.2	325.6

(C) The average I_{sp} efficiency for UTP-6825 (88.2%) was slightly higher than for UTP-6826 (87.4%). Probable upperstage applications indicate that in the density-impulse parameter $I_{sp}\ \rho^n$, an exponent of 0.3 is appropriate. In this context, UTP-6825 is the more attractive choice. Therefore, three 10-lb motors of UTP-6825 were processed and tested. The results are given in table XII.

(U) A later addition to the program included an investigation of the specific impulse efficiency of low burning rate formulations. Lower burning rate formulations typically use a high concentration of coarse oxidizer which can be a cause of reduced combustion efficiency. Two relatively low burning rate formulations were chosen for evaluation in 10-lb motors, a straight LMH-1/AP/PEP-150 formulation and a mixed fuel aluminum/LMH-1/AP/PEP-150 formulation. Both formulations used an 80/20 coarse/fine oxidizer ratio and a slightly increased binder level (28 vol-%). The increased binder was needed to facilitate processing as the oxidizer size ratio was not optimum for maximum castability. The formulation containing only LMH-1 for the fuel — UTP-8819 — essentially duplicates UTP-8809, the low burning rate formulation developed under task 2, except that the sodium barbituate coating on the oxidizer was omitted.

(C) UTP-8820, the mixed fuel formulation is similar to UTP-6825 which also was tested in 10-lb motors. The test results for these two formulations are presented in table XIII. Table XIV is a summary of the formulation parameters and test results of all propellants tested in tasks 1 and 3. In addition, UTP-8812 the scaleup formulation characterized in phase II and UTP-6814 (developed under AF 04(611)-9570) are included for comparison. The measured performance of UTP 6834 and UTP-6835 and their controls in 10-lb motors is inexplicably low but intraconsistent.

(C) In the mixed fuel formulations UTP-6825 and UTP-6826 it appears that the 4-lb motor test results might be slightly understated because the control motor results are somewhat low. On the other hand, the I_{sp} efficiencies of 90.3% and 89.7% are well within the range of straight LMH-1 formulations at similar O/F ratios. On the basis of the limited tests performed there appears to be no specific impulse efficiency penalties associated with the use of mixed aluminum/AlH_3.

(C) In assessing the possibility of performance losses resulting from the use of increased levels of coarse oxidizer UTP-8820 can be compared to UTP-6825. The average I_{sp} efficiency of 91.7% obtained from UTP-8820 (containing the higher concentration of coarse oxidizer) can be compared favorably with the 90.3% efficiency obtained from UTP-6825. However, control motors tested with UTP-6825 delivered I_{sp} efficiencies 1.5% lower than did those tested with UTP-8820 (92.6% versus 94.1%). As a result it is not clear if performance losses did or did not occur as a result of the increased coarse/fine oxidizer ratio.

(C) A somewhat better comparison might be made between UTP-8819 and UTP-6814, the scaleup propellant developed under AF 04(611)-9570. Both formulations contain 25% AlH_3. Theoretical I_{sp} values are almost identical for the two formulations (281.4 versus 281.1). However, UTP-8819 has a slightly higher O/F ratio, 1.2 versus 1.15, and a higher flame temperature. The I_{sp} efficiency for UTP-8819 is 90.6% compared to 91.4% for UTP-6814 in 10-lb motors. On this basis it can be assumed that a performance penalty of approximately 1% might be incurred in motors of this size if a high ratio of coarse oxidizer is substituted for a high ratio of "fines." It seems logical that such a penalty will disappear in larger motors of longer residence time.

TABLE XII

(U) PROJECT 2146 – PHASE I,
DENSITY/I_{sp} STUDIES, 4-lb

Test No.	Formulation No.	r_b in./sec	T_c ° K	P_{c_a} psia	P_{c_b} psia	c* ft/sec	Theoretical c* ft/sec	c* Efficiency %
233	UTP 6825	---	3,517	1,231.4	---	5,280	5,529	95.5
234	UTP 6825	0.972	3,496	964.9	1,078.6	5,216	5,520	94.5
244	UTP 6825	0.899	3,496	971.2	1,056.9	5,279	5,520	95.6
246	UTP 6825	0.856	3,494	946.9	1,012.1	5,292	5,520	95.9
						5,267		95.4
231	UTP 6826	0.633	3,663	1,266.5	1,346.7	5,118	5,420	94.4
232	UTP 6826	0.592	3,641	1,021.9	1,081.8	5,162	5,412	95.4
240	UTP 6826	0.734	3,648	1,090.7	1,182.4	5,348	5,415	98.8
242	UTP 6826	0.701	3,640	1,016.9	1,089.2	5,185	5,412	95.8
						5,203		96.1
239	UTP 3001	0.338	3,403	919.2	940.1	5,045	5,178	97.4
241	UTP 3001	0.345	3,409	1,000.5	1,027.1	5,155	5,179	99.5
243	UTP 3001	0.342	3,409	997.2	1,025.9	5,101	5,179	98.5
245	UTP 3001	0.329	3,406	964.1	989.3	5,202	5,178	100.5
						5,126		98.9
								Ten-lb Motors
260	UTP 6825	0.707	3,483	833.0	885.9	5,273	5,517	95.6
262	UTP 6825	0.719	3,483	833.8	898.8	5,235	5,516	94.9
264	UTP 6825	0.700	3,484	347.5	902.1	5,177	5,516	93.9
						5,228		94.8
259	UTP 3001	0.383	3,390	949.5	986.7	4,977	5,165	96.4
261	UTP 3001	0.378	3,390	958.0	987.1	4,994	5,165	96.7
263	UTP 3001	0.376	3,390	958.7	982.3	4,975	5,165	96.3
						4,982		96.5

*** Bond failure**

TABLE XII

CT 2146 – PHASE I, TASK 3

I_{sp} STUDIES, 4-lb MOTORS

tical ec	c* Efficiency %	Specific Impulse Delivered P_{c_a}	Corrected to 0° at P_c	Corrected 1,000/0° (A)	Corrected 1,000/0° (B)	Theoretical at Test Condition	Efficiency %
29	95.5	251.5	255.8	251.7	251.9	284.4	89.9*
20	94.5	247.6	251.8	252.3	253.0	278.9	90.3
20	95.6	247.4	251.6	252.1	252.7	278.9	90.2
20	95.9	248.7	252.9	253.9	254.7	278.2	90.9
	95.4			255.5	253.1		90.3
20	94.4	251.1	255.4	251.0	251.1	279.7	91.3
12	95.4	244.6	248.8	247.2	249.2	274.7	90.6
15	98.8	242.2	246.3	244.3	244.2	277.3	88.8
12	95.8	240.8	244.9	244.4	244.5	275.5	88.9
	96.1			246.7	247.3		89.9
78	97.4	228.6	232.5	234.1	234.0	259.7	89.5
79	99.5	236.0	240.0	239.8	240.1	261.5	91.8
79	98.5	220.9	224.7	224.7	224.6	261.5	85.9
78	100.5	240.0	244.1	244.6	244.8	260.8	93.6
	98.9			235.8	235.9		90.2
Ten-lb Motors							
17	95.6	246.3	250.5	256.1	258.1	272.0	92.1
16	94.9	243.0	247.1	251.2	252.5	274.2	90.1
16	93.9	247.5	251.7	255.7	256.9	274.5	91.7
	94.8			254.3	255.8		91.3
65	96.4	234.7	238.7	239.6	240.1	260.1	91.8
65	96.7	235.4	239.4	240.1	240.6	260.3	92.0
65	96.3	240.8	244.9	245.9	245.8	260.4	94.0
	96.5			241.9	242.2		92.6

TABLE XIII

(U) PROJECT 2146 – PHA
I_{sp} EFFICIENCY STUDIES, 10-1

Test No.	Formulation No.	r_b in./sec	T_c ° K	P_{c_a} psia	P_{c_b} psia	c* ft/sec	Theoretical c* ft/sec	c* Efficiency %
336	UTP 8819	0. 326	3, 333	1, 006. 0	1, 038. 8	5, 435	5, 545	98. 0
887	UTP 8819	0. 303	3, 303	659. 9	694. 3	5, 285	5, 534	95. 5
340	UTP 8819	0. 300	3, 284	508. 4	544. 0	5, 239	5, 528	94. 8
						5, 320		96. 1
341	UTP 8820	0. 403	3, 440	1, 065. 0	1, 115. 2	5, 372	5, 496	97. 7
343	UTP 8820	0. 358	3, 405	694. 8	730. 8	5, 257	5, 484	95. 9
344	UTP 8820	0. 344	3, 368	448. 8	490. 3	5, 235	5, 470	95. 7
						5, 288		96. 4
335	UTP 3001	0. 381	3, 392	986. 4	1, 013. 4	5, 099	5, 166	98. 7
338	UTP 3001	0. 381	3, 390	954. 0	989. 4	5, 112	5, 165	99. 0
339	UTP 3001	0. 414	3, 392	975. 6	1, 046. 6	5, 126	5, 165	99. 2
342	UTP 3001	0. 445	3, 393	1, 000. 7	1, 081. 6	5, 064	5, 166	98. 0
						5, 100		98. 7

TABLE XIII
ROJECT 2146 -- PHASE I
NCY STUDIES, 10-lb MOTORS

		Specific Impulse					
tical ec	c* Efficiency %	Delivered P_{c_a}	Corrected $P_{c_a}/0°$	Corrected 1,000/0° (A)	Corrected 1,000/0° (B)	Theoretical at Test Condition	Efficiency %
45	98.0	250.3	254.6	257.3	258.9	276.7	92.0
34	95.5	238.8	242.9	253.1	253.5	269.5	90.1
28	94.8	230.4	234.3	250.7	252.1	261.6	89.6
	96.1			253.7	254.8		90.6
96	97.7	249.1	253.3	256.7	258.7	273.2	92.7
84	95.9	240.6	244.7	252.8	255.1	267.6	91.4
70	95.7	225.4	229.2	253.6	254.3	251.6	91.1
	96.4			254.4	256.0		91.7
66	98.7	240.9	245.0	245.3	245.5	261.0	93.9
65	99.0	240.4	244.5	245.4	245.8	260.2	94.0
65	99.2	241.8	245.9	246.4	246.6	260.7	94.3
66	98.0	241.4	245.5	245.5	245.8	261.2	94.0
	98.7			245.7	245.9		94.1

TABLE XIV

(U) SUMMARY OF FORMULATIONS TESTED ON TASKS 1 AND 3

	UTP 6825	UTP 6826	UTP 6834	UTP 6835	UTP 8819	UTP 8820	UTP 8812*	UTP 6814†
PEP-150, wt %	---	---	17.50	17.20	21.70	20.80	19.50	---
PEP-155, wt %	18.72	18.00	---	---	---	---	---	25.47
LMH-1, wt %	21.00	14.25	26.00	24.80	25.00	21.00	24.00	25.00
Aluminum, wt %	3.65	9.75	---	---	---	3.60	---	---
NH_4ClO_4, wt %	56.63	58.00	56.50	58.00	53.30	54.60	56.50	49.53
NH_4ClO_4 coarse/fines	27/73	39/61	46/54	52/48	80/20	80/20	54/46	16/84
Volume % binder	25	25	23	23	28	28	25	32
r_b at 1,000 psia	0.80	0.60	0.47	0.5	0.52	0.38	0.40	0.58
O/F	1.37	1.4	1.3	1.4	1.2	1.25	1.4	1.15
Calculated T_c, °K	3,500	3,638	3,435	3,445	3,330	3,440	3,395	3,215
Theoretical density, lb/in³	0.0604	0.0627	0.0590	0.0592	0.058	0.0596	0.059	0.0573
Theoretical I_{sp}, sec	280.1	275.0	284	282.8	281.4	279.1	280.9	281.1
I_{sp} efficiencies, %								
in 4-lb motors	90.3	89.9	88.2	89.1	---	---	---	89.4
in 10-lb motors	91.3	---	88.9	87.6	90.6	91.6	92.7	91.4
I_{sp} efficiencies of control, %								
4-lb motors	90.2	90.2	92.3	92.3	---	---	---	---
10-lb motors	92.6	---	90.1	90.1	94.1	94.1	93.2	---

* Scaleup propellant, Phase II

† Scaleup propellant on Contract AF 04(611)-9570 — delivered 93.4% I_{sp} efficiency in 300-lb motor.

b. Burning Rate Studies – Task 2

(1) Laboratory Scale Studies

(a) Parameter Studies

(C) In order to make this propellant system adaptable to a wide range of potential missile systems, a study was conducted to develop a spectrum of available burning rates. Target burning rates of 0.25 in./sec and 1.0 in./sec at 1,000 psia were established. Generally speaking, the higher burning rates in the range of about 0.4 to about 1.0 in./sec at 1,000 psia offered no serious problems as 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 AF 04(611)-9570, 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 1,000 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 AlH_3 concentration. For purposes of the burning rate investigation, the particle size distribution of the two solid ingredients, i.e., AP and AlH_3, are added parameters. Because the effect of these last two parameters (particle sizes) can generally be predicted qualitatively, it only remained to measure quantitatively the effect of AP and AlH_3 particle sizes in this particular system.

(C) The four primary formulation parameters, AP, LMH-1, 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 represents a tradeoff between AlH_3 and AP 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	4 ratios
LMH-1 size	2 fractions

Constants	Variables
30% binder (PEP-150) 50:50 AP ratio	O/F 1.0, 1.2, 1.4
30% binder, 1.2 O/F 50:50 AP ratio	Plasticizer 30, 40, 50, 60
PEP-150, 1.2 O/F 50:50 AP ratio	Binder volume 25, 30, 32.5%
30% binder, (PEP-150) 1.2 O/F	AP ratio (coarse/fine) 60/40, 50/50, 35/65, 20/80
30% binder, (PEP-150) 1.2 O/F	Screen LMH-1 into 2 fractions

(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 AlH_3 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. The micromotor test system has proved to be a reliable tool for obtaining burning rates on this program. The micromotor design is illustrated in figures 25 and 26.

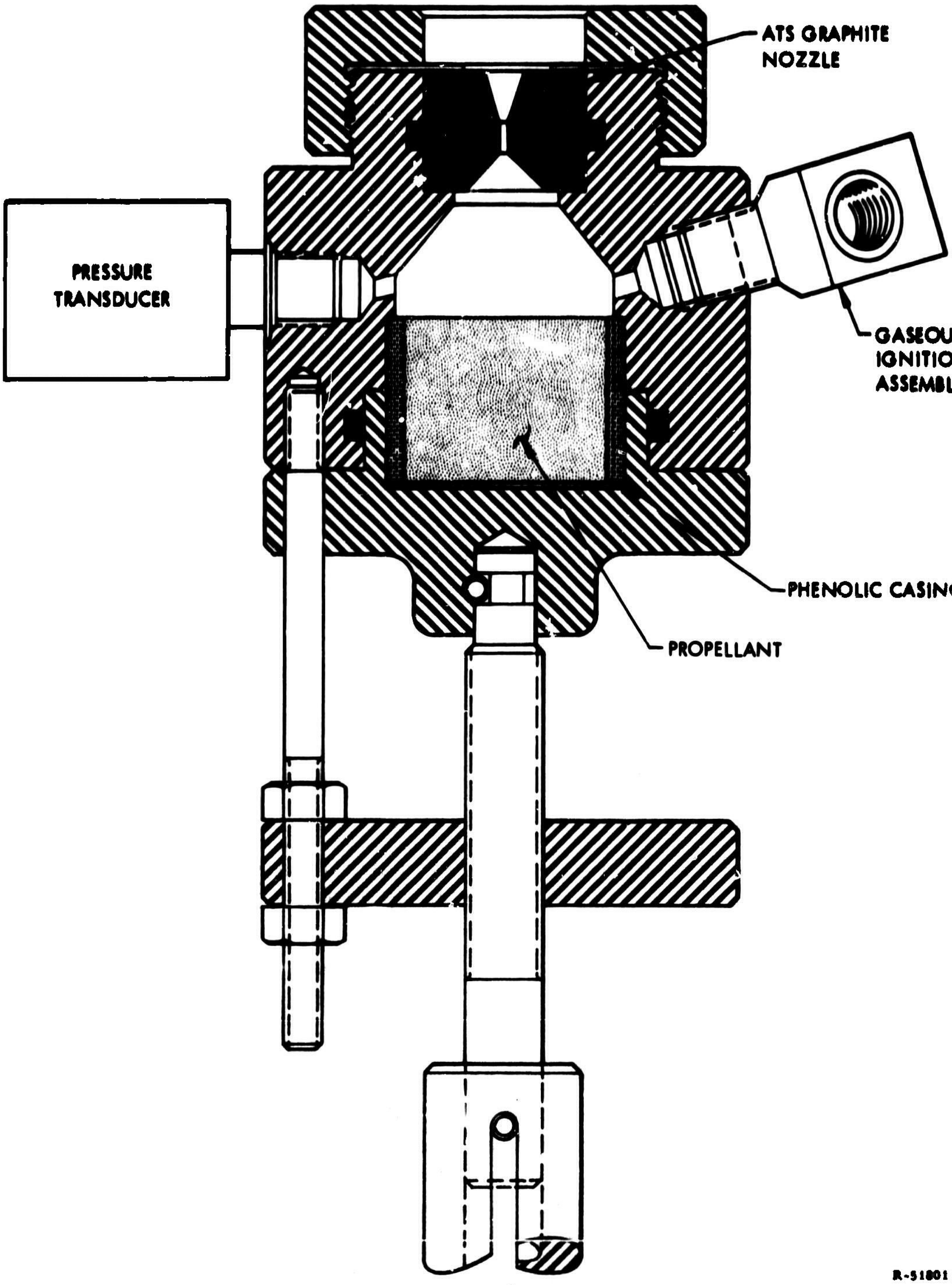


Figure 25. (U) Micromotor Chamber

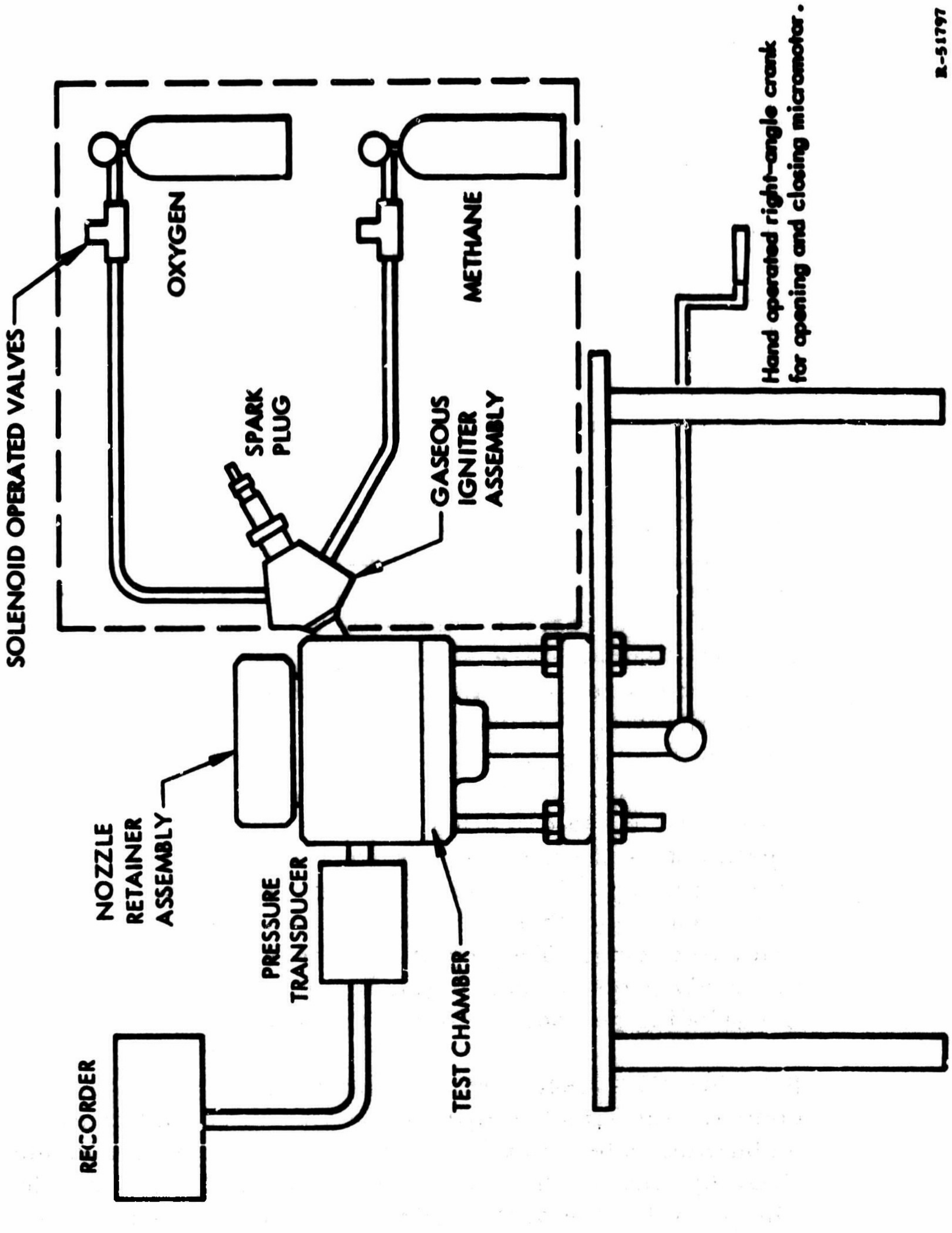


Figure 26. (U) Micromotor Test System for Burning Rate Studies

(C) The study was divided into two phases to investigate the variables which effect the burning rates of LMH-1 propellant. Phase I evaluated the effect of the formulation parameters outlined above. Phase II was an evaluation of the effect of burning rate modifiers on high burning rate and low burning rate formulations as determined from the phase I study.

(U) The initial studies were to determine the effect of grinding LMH-1. The LMH-1 was ground for different time periods in a ball mill to reduce particle sizes and then mixed in UTX-6814 formulations. The immediate goal was to determine the effect of grinding on processability and cured propellant density. The ultimate goal was to increase the amount of coarse AP and reduce the amount of fine oxidizer so that the burning rates would be lowered. Use of smaller fuel particles was expected to permit processing with more coarse AP.

(C) Table XV summarizes the experimental data on propellant viscosities and densities. The propellant viscosities were directly proportional to grinding time as longer ball milling times resulted in higher viscosities. This increase in viscosity was attributed to increased surface areas as the average particle size diameter decreased with longer mixing time. Figure 27 shows the variation in crystal sizes which were obtained after ball milling for various time periods. Propellant densities in which the LMH-1 was wet ground in acrylonitrile had densities corresponding to time of treatment as the hydride was not further surface treated after grinding. The LMH-1 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 inversely proportional to grinding time. The longer the grinding period, the lower the propellant density.

(C) Two UTX-6846 formulations were prepared to compare the effect of hydride particle size distribution on burning rate. Batch 3 contained as-received hydride (BL-30), and batch 4 contained screened BL-30 hydride. The particle size of the hydride in batch 4 was greater than 12μ as the hydride used in this formulation was

TABLE XV

(U) GRIND EFFECT, BL-28 UTC AN

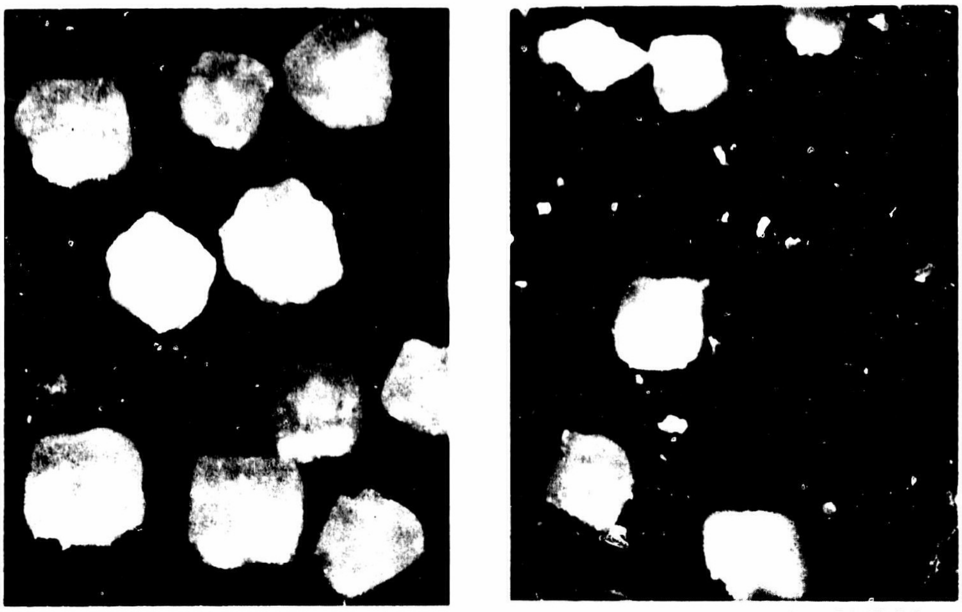
UTX 6814	Grind Time min	Viscosity Poises	Density g/cc	% Theoretical Density
116	0	1,800	1.420	89.5
117	15 in AN	1,800	1.402	88.3
118	30 in AN	5,500	1.416	89.2
119	60 in AN	9,400	1,516	95.5
120	15 dry	1,200	1.457	91.8
121	30 dry	4,200	1.440	90.7

retained on a 115-mesh Tyler Screen. The AP coarse/fine ratio was 80/20. Both batches had a burning rate of 0.27 in./sec at 1,000 psi, indicating no change in burning rate with different particle size hydrides.

(U) The effect of binder concentration on burning rate in terms of vol-% binder is illustrated in figure 28. The binder concentration within the range of interest appeared to have essentially no effect on burning rate. The effect of O/F ratio was significant as indicated by figure 28. An increase in O/F ratio from 1.0 to 1.4 resulted in an increase in burning rate of over 25%.

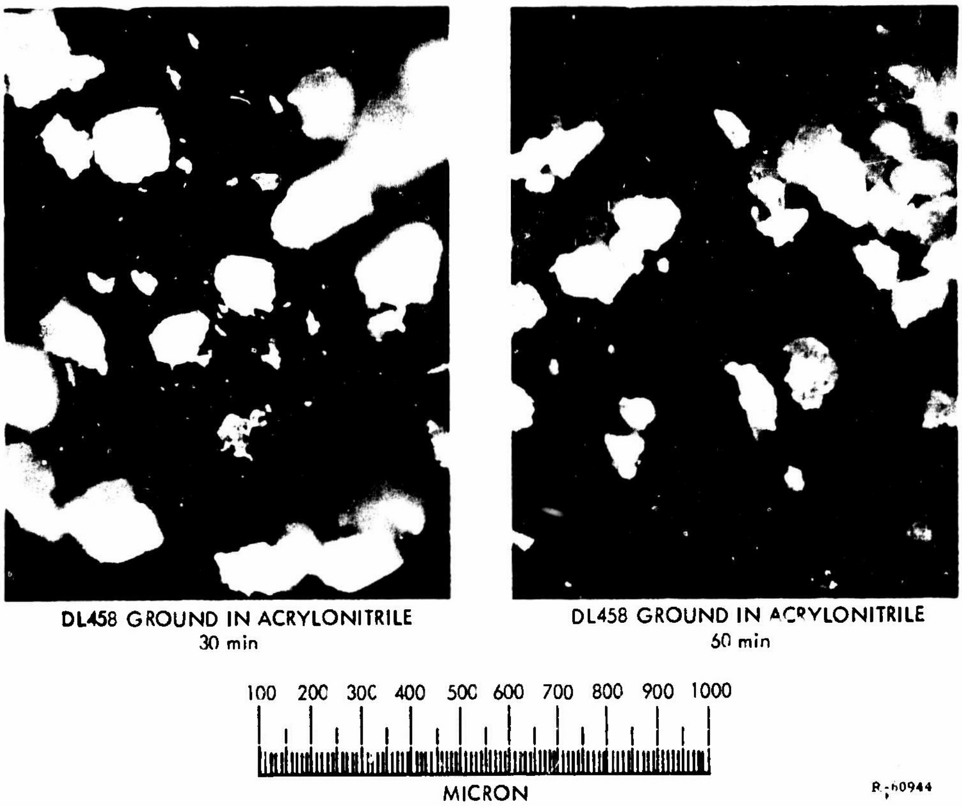
(C) Figure 29 illustrates the effect of oxidizer particle size distribution. The coarse fraction of oxidizer has an average particle diameter above 300μ. The fine fraction has an average particle diameter of 8μ to 10μ. The binder vol-%, plasticizer level, and O/F ratio were held constant in all mixes. The experimental data are summarized in table XVI. The lowest burning rate, 0.27 in./sec at 1,000 psi, was achieved with the 80/20 coarse/fine, AP grind ratio.

(C) A formulation, UTX-6846-5, was prepared using 600μ to 800μ AP in place of the 300μ AP used in mix UTX 6846-4. No change in burning rate was measured between these two formulations.



DL 458 AS RECD

DL458 GROUND IN ACRYLONITRILE
15 min



DL458 GROUND IN ACRYLONITRILE
30 min

DL458 GROUND IN ACRYLONITRILE
60 min

Figure 27. (U) Effect of Ball Milling on Dowane 1451 Particle Size (lot DL 458)

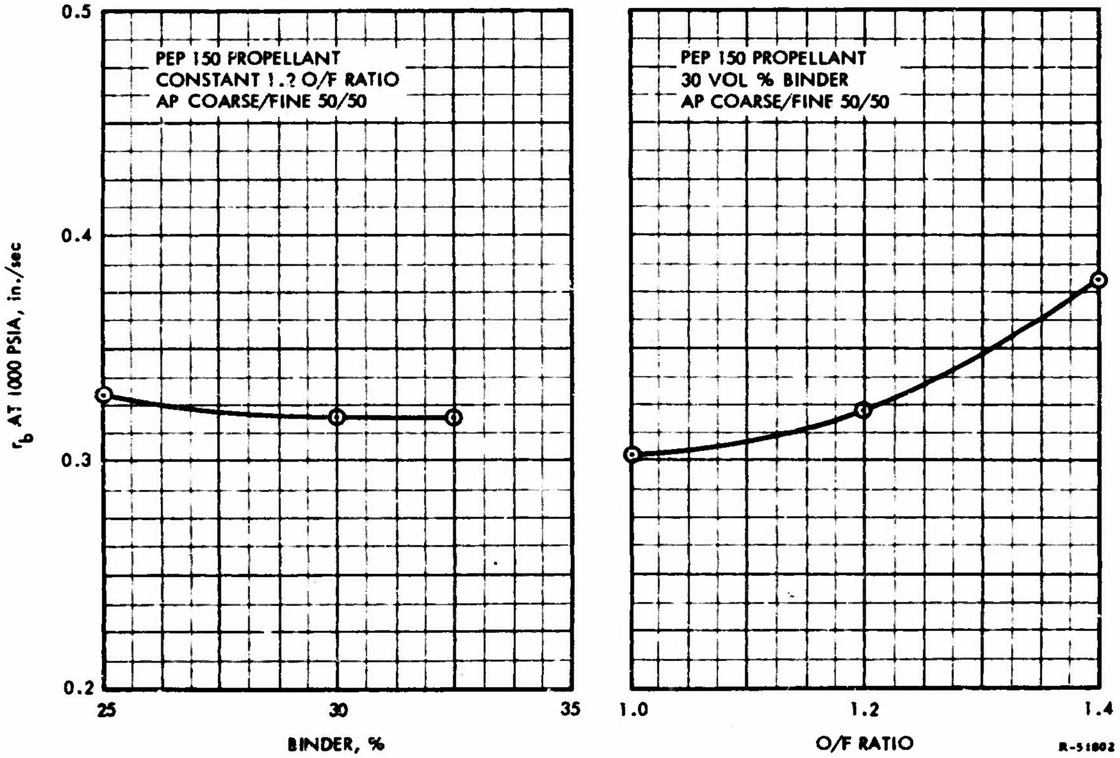


Figure 28. (U) Effect of Binder Concentration and O/F Ratio on Burning Rate

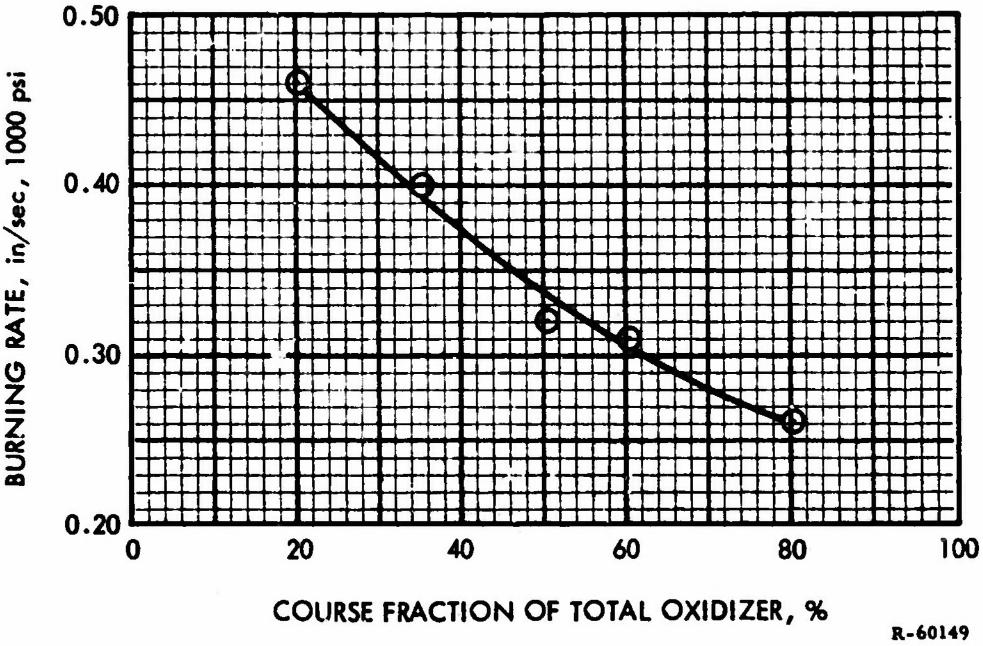


Figure 29. (U) Effect of Oxidizer Grind Ratio on Burning Rate

TABLE XVI

(U) EFFECT OF AP PARTICLE SIZE DISTRIBUTION ON BURNING RATE

UTX Formulation	Binder Vol-%	Plasticizer Level	O/F Ratio	AP Grind Ratio Coarse/Fine	in. /sec 1,000 psi
6850-3	30	50	1.2	20/80	0.46
6850-2	30	50	1.2	35/65	0.40
6843-1	30	50	1.2	50/50	0.32
6850-1	30	50	1.2	60/40	0.31
6850-7	30	50	1.2	80/20	0.26

(C) To study the effect of plasticizer concentration on burning rate, PEP-130, -140, -150, and -160 formulations were prepared with constant vol-%, O/F ratio, and AP grind ratio. Figure 30 illustrates that increasing plasticizer level results in higher burning rates.

(b) Additive Study

(U) After examination of data obtained in the formulation parameter study, the following formulations were selected to define the upper and lower limits of the burning rate spectrum that could be achieved by adding burning rate modifiers:

High Burning Rate Propellant	Formulation Variable	Low Burning Rate Propellant
1.4	O/F	1.1
160	PEP Binder	140
20/80	AP Grind Ratio Coarse/Fine	80/20

(C) The high burning rate control propellant, UTX-6885-1, had a burning rate of 0.76 in./sec at 1,000 psi. The low burning rate control formulation had a measured burning rate of 0.26 in./sec at 1,000 psi. The greatest burning rate increase was obtained in the mix containing 1 wt-% ferric oxide.

(C) The burning rate catalysts selected for the PEP-160 high burning rate formulations were iron oxide, ferrocene, and ferric ammonium sulfate. The burning rate depressants, sodium barbituate and ammonium oxalate, were evaluated with the low burning rate formulation.

(C) In several of the mixes, the burning rate modifiers were coated on the AP oxidizer using a methanol rotovap coating technique. A slurry was made of the oxidizer and coating agent with menthanol. The alcohol was then slowly evaporated by using a rotovap.

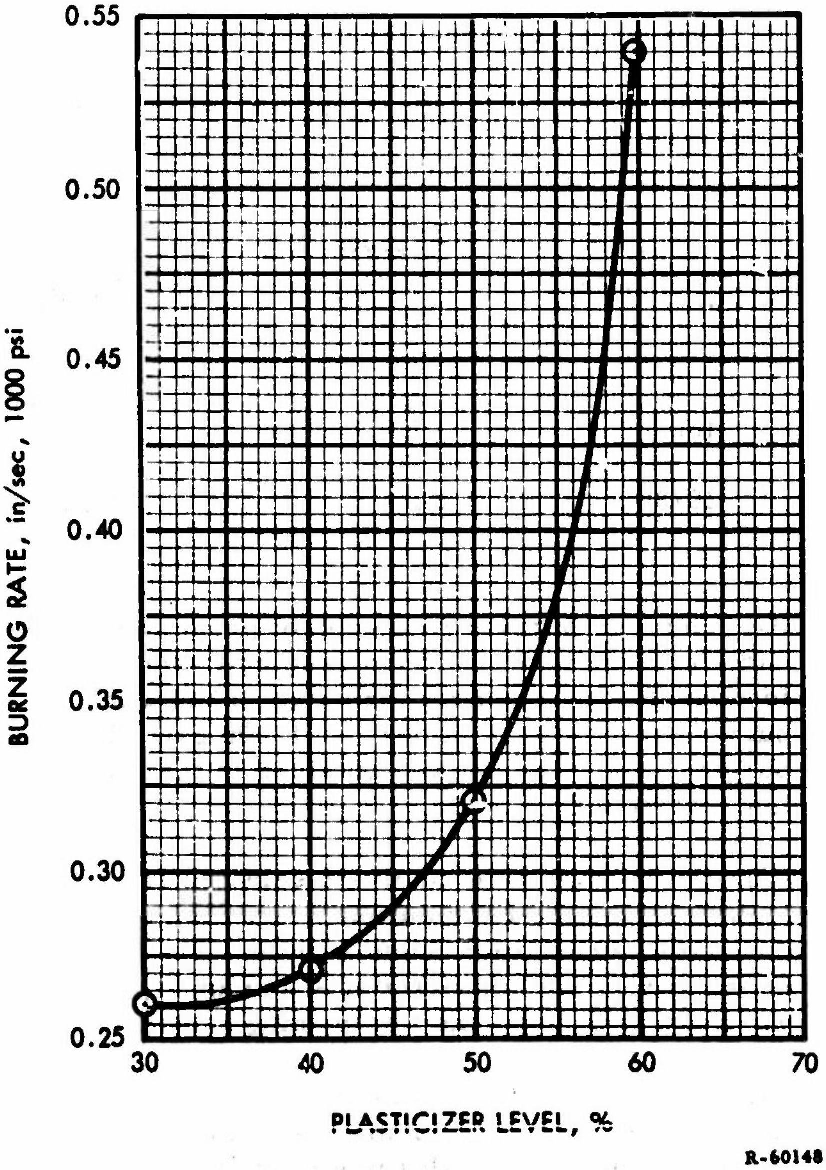


Figure 30. (U) Effect of Plasticizer Level on Burning Rate

(C) No significant lowering of burning rates was measured by mixing depressants into the PEP-140 formulation. However, the use of 1% coating of sodium barbituate on the coarse fraction of oxidizer reduced the burning rate of the low rate formulation to 0.22 in./sec at 1,000 psi. The results of the burning rate additive study are presented in table XVII.

(2) Results of Motor Testing

(U) Following the studies designed to define the effects of formulation parameters on propellant burning rate and the subsequent evaluation of burning rate additives, four formulations were selected for evaluation in 4-lb motors. These formulations and the criteria for their selection are:

UTX 8801 - fastest burning

UTX 8802 - fastest burning with good physicals

UTX 8809 - most energetic slow burning

UTX 8814 - slowest burning.

(U) The propellant formulations are listed in table XVIII. The 4-lb motor test results are presented in table XIX. For more convenient analysis of the results, the formulation parameters and test results are summarized in table XX.

(C) The control motors for the entire series of tests delivered reproducible results within the range of values expected (92.6% I_{sp} efficiency). The measured performances for the burning rate motors are therefore considered realistic. The high burning rate formulations yielded burning rates of 1.15 and 1.00 in./sec at 1,000 psia in the PEP-160 and PEP-150 systems, respectively. The resulting I_{sp} efficiency values were quite different for the two formulations, 88.8% versus 91.3%, for no apparent reason. The PEP-150 system (UTP-8802) was considered more practical as a scaleup candidate than the more highly plasticized formulation in view of the effect of plasticizer on mechanical properties.

(C) In addition, the delivered I_{sp} values (255.0 sec 1,000/14.7, 0°) in 4-lb motors were attractive. This formulation was selected for evaluation in 10-lb motors.

TABLE XVII

(U) BURNING RATE ADDITIVE STUDY

Formulation No.	Formulation Variable	r_b in/sec 1,000 psi	n*
UTX 6873-1	1/2 wt-% iron oxide	1.32	0.48
UTX 6874-1	1 wt-% iron oxide	1.58	0.50
UTX 6875-1	1/2 wt-% ferrocene	1.52	0.40
UTX 6876-1	1 wt-% ferrocene	1.45	0.48
UTX 6877-1	1/2 wt-% ferric ammonium sulfate coated on 12μ AP	0.76	0.60
UTX 6878-1	1 wt-% ferric ammonium sulfate coated on 12μ AP	1.22	0.42
UTX 6879-1	1/2 wt-% sodium barbituate coated on 300μ AP	0.25	0.50
UTX 6880-1	1 wt-% sodium barbituate coated on 300μ AP	0.22	0.46
UTX 6881-1	1 wt-% ammonium oxalate coated on 300μ AP	0.25	0.56
UTX 6882-1	1/2 wt-% sodium barbituate added directly to mix	0.26	0.40
UTX 6885-1	PEP-160 control mix	0.76	0.64
UTX 6886-1	PEP-140 control mix	0.26	0.61

* Exponent in $r_b = cP^n$

TABLE XVIII

(U) 4-lb MOTOR BURNING RATE FORMULATIONS

	UTX-8801	UTX-8802	UTX-8809	UTX-8814
HX 735	6.88	8.40	8.66	10.29
Atlas 2684	0.10	0.10	---	---
DOA	---	---	0.50	0.50
MAPO	0.71	0.87	0.96	1.05
Epon 812	0.51	0.63	0.63	0.76
TMETN	12.30	10.00	10.75	8.40
LMH-1	25.20	23.50	25.00	25.00
AP, 300μ	10.66	11.10	42.80*	43.20*
AP, 12μ	42.64	44.40	10.70*	10.80*
Fe_2O_3	1.00	1.00	---	---

* 1% coating of sodium barbituate.

(C) The low burning rate motors both yielded burning rates of 0.24 in./sec at 1,000 psia. As a result the target burning rates at both ends of the spectrum were achieved. Because the PEP-150 system (UTX-8809) yielded the higher I_{sp} efficiency 88.7% and also had the higher theoretical I_{sp} (281.7 sec), this system was chosen for evaluation in 10-lb motors.

(C) The results of the 10-lb motor tests are presented in table XXI. Inasmuch as the Uteflex/HX-874 polymer system had been selected for the phase II characterization studies it was used in the high burning rate formulation in place of the HX-735/MAPO system which had been used in the 4-lb motors. A dramatic increase in burning rate was experienced in the 10-lb motors. The Uteflex formulation (UTP-8815) delivered a burning rate of 1.48 in./sec at 1,000 psia compared to 1.0 in./sec in the HX-735/MAPO system. An I_{sp} efficiency of 93.8% was obtained in the 10-lb motors (261.5 sec 1,000/14.7, 0°) which represents the highest efficiency measured on this program for an AlH_3 formulation. The low burning rate 10-lb motors were somewhat porous and yielded inconsistent burning rate data.

[redacted]

TABLE XIX

(U) BURNING RATE STUDIES, 4-

Test No.	Formulation No.	r_b in./sec	P_{c_a} psia	P_{c_b} psia	c* ft/sec	Theoretical c* ft/sec	c* Efficiency %	De
276	UTP 8801					Bond Failure – No Data Redu		
278	UTP 8801	0.845	314.3	331.2	5,315	5,515	96.4	
280	UTP 8801	1.113	863.8	906.9	5,416	5,548	97.6	
					5,366		97.0	
282	UTP 8802	0.731	515.0	555.1	5,439	5,488	99.1	
284	UTP 8802	0.703	535.3	567.1	5,364	5,488	97.7	
286	UTP 8802	0.919	795.5	844.7	5,407	5,502	98.3	
					5,403		98.4	
314	UTP 8809	0.196	505.4	522.1	5,256	5,535	95.0	
316	UTP 8809	0.211	731.2	756.5	5,271	5,546	95.0	
319	UTP 8809	0.263	1,156.1	1,209.4	5,341	5,556	96.1	
					5,289		95.4	
321	UTP 8814	0.151	456.8	466.1	5,144	5,502	93.4	
323	UTP 8814	0.308	1,096.0	1,248.4	5,280	5,523	95.6	
326	UTP 8814	0.188	655.4	687.3	5,291	5,513	96.0	
					5,238		95.0	
275	UTP 3001	0.374	929.2	953.1	5,058	5,164	97.9	
277	UTP 3001	0.382	985.8	1,015.6	5,019	5,166	97.2	
279	UTP 3001	0.379	988.4	1,013.8	4,988	5,166	96.6	
281	UTP 3001	0.379	952.9	987.4	5,005	5,165	96.9	
283	UTP 3001	0.372	953.7	975.2	4,986	5,165	96.5	
285	UTP 3001	0.373	948.6	980.8	4,982	5,163	96.5	
313	UTP 3001	0.373	922.1	966.7	5,000	5,164	96.8	
315	UTP 3001	0.367	947.9	974.6	5,049	5,164	97.8	
317	UTP 3001	0.444	963.4	1,042.4	4,972	5,167	96.2	
318	UTP 3001	0.444	993.8	1,065.9	5,059	5,166	97.9	
320	UTP 3001	0.431	969.6	1,041.5	5,037	5,165	97.5	
322	UTP 3001	0.429	969.6	1,026.0	5,024	5,165	97.3	
324	UTP 3001	0.457	1,007.7	1,093.1	5,126	5,166	99.2	
325	UTP 3001	0.396	970.6	1,012.3	5,085	5,165	98.5	
327	UTP 3001	0.397	967.1	1,007.9	5,051	5,165	97.8	
328	UTP 3001	0.388	966.6	985.9	5,001	5,165	96.8	
					5,028		97.6	

TABLE XIX

G RATE STUDIES, 4-lb MOTORS

cal	c* Efficiency %	Specific Impulse					
		Delivered P_c	Corrected to 0° to P_c	Corrected 1,000/0° (A)	Corrected 1,000/0° (B)	Theoretical at Test Conditions	Efficiency %
ailure – No Data Reduction							
	96.4	207.5	211.0	2[illegible]2.6	246.8	240.6	87.5
	97.6	240.5	244.6	2[illegible]1.5	254.1	271.6	90.1
	97.0			247.1	250.5		88.8
	99.1	232.9	236.9	253.2	255.2	259.2	91.4
	97.7	230.4	234.3	250.5	253.5	257.9	90.8
	98.3	237.1	241.1	253.4	256.3	262.5	91.8
	98.4			252.4	255.0		91.3
	95.0	225.4	229.2	245.1	246.5	262.0	87.5
	95.0	239.4	243.5	250.7	251.3	273.1	89.2
	96.1	247.0	251.2	250.6	251.6	281.3	89.3
	95.4			248.8	249.8		88.7
	93.4	222.9	226.7	245.1	246.3	258.0	87.0
	95.6	241.9	246.0	245.5	246.3	279.8	87.9
	96.0	231.3	235.2	245.0	246.6	267.4	88.0
	95.0			245.2	246.4		87.9
	97.9	235.0	239.0	241.1	241.9	258.4	92.5
	97.2	237.1	241.1	242.1	242.9	259.6	92.9
	96.6	236.9	240.9	241.8	242.7	259.7	92.8
	96.9	235.9	239.9	241.5	242.1	259.0	92.6
	96.5	236.7	240.7	242.4	242.9	259.0	92.9
	96.5	236.1	240.1	241.7	242.4	258.9	92.7
	96.8	236.1	240.1	242.4	243.2	258.2	93.0
	97.8	236.0	240.0	241.5	241.4	260.0	92.3
	96.2	235.7	239.7	240.9	238.7	262.5	91.3
	97.9	236.3	240.3	240.7	240.6	261.3	92.0
	97.5	236.8	240.8	241.8	241.6	260.6	92.4
	97.3	237.0	241.0	242.0	241.9	260.6	92.5
	99.2	237.9	241.9	241.8	246.6	256.6	94.3
	98.5	237.4	241.4	242.4	242.1	260.6	92.6
	97.8	237.1	241.1	242.1	241.9	260.6	92.5
	96.8	237.3	241.3	242.4	242.1	260.5	92.6
	97.6			241.8	242.2		92.6

TABLE XX

(U) SUMMARY OF BURNING RATE FORMULATIONS AND TEST RESULTS

	UTP 8801	UTP 8802	UTP 8809	UTP 8814
PEP-140, wt-%	---	---	---	21.00
PEP-150, wt-%	---	20.00	21.50	---
PEP-160, wt-%	20.50	---	---	---
Aluminum hydride, wt-%	25.20	23.50	25.00	25.00
Ammonium perchlorate, wt-%	53.30	55.50	53.50*	54.00*
Coarse/fine ratio	20/80	20/80	80/20	80/20
Fe_2O_3	1.00	1.00	---	---
Volume % binder	26	26	28	28
O/F ratio	1.4	1.4	1.2	1.15
Theoretical I_{sp}	282.0	279.2	281.7	280.2
4-lb motor results, r_b at 1,000 psia, in./sec	1.15	1.00	0.24	0.24
I_{sp} efficiency, %	88.8	91.3	88.7	87.9
10-lb motor results, r_b at 1,000 psia, in./sec	---	1.48	---	---
I_{sp} efficiency, %	---	93.8†	---	---

* 1% sodium barbituate coating on oxidizer

† UTP 8815 – same as UTX 8802 except Uteflex/HX-874 substituted for HX-735/MAPO system. Theoretical I_{sp} is 278.8.

[redacted]

TABLE XXI

(U) PROJECT 2146 – PHAS
BURNING RATE STUDIES, 1

Test No.	Formulation No.	T_c ° K	r_b in./sec	P_{c_a} psia	P_{c_b} psia	c* ft/sec	Theoretical c*	c* Efficie %
330	UTP 8815	3,383	1.553	1,187.0	1,256.5	5,387.7	5,500	98.
332	UTP 8815	3,359	1.451	865.5	935.9	5,446.8	5,490	99.
334	UTP 8815	3,346	1.344	720.1	768.3	5,593	5,486	102
						5,476		99+
346	UTP 8809	3,227	0.337	529.6	615.0	5,344	5,536	96.
348	UTP 8809	3,254	0.416	806.1	972.8	5,216	5,548	94.
349	UTP 8809	3,255	0.424	817.3	996.6	5,270	5,546	95.
350	UTP 8809	3,218	0.339	464.7	539.3	5,226	5,532	94.
						5,264		95.
329	UTP 3001	3,389	0.382	935.3	976.3	5,056	5,166	97.
331	UTP 3001	3,390	0.409	958.0	1,021.2	5,078	5,165	98.
333	UTP 3001	3,388	0.378	923.9	959.8	5,098	5,164	98
345	UTP 3001	3,388	0.377	928.9	963.0	5,064	5,166	98
347	UTP 3001	3 388	0.378	925.1	960.0	5,103	5,162	98
						5,080		98

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TABLE XXI

CT 2146 – PHASE I, TASK 2

TE STUDIES, 10-lb MOTORS

		Specific Impulse					
retical *	c* Efficiency %	Delivered P_{c_a}	Corrected to 0° at P_{c_a}	Corrected 1,000/0° (A)	Corrected 1,000/0° (B)	Theoretical Test Condition	Efficiency %
500	98.0	250.3	254.6	259.5	262.1	270.7	94.0
490	99.2	235.3	239.3	255.7	259.0	257.5	92.9
486	102	232.7	236.7	259.4	263.5	250.6	94.5
	99+			258.2	261.5		93.8
, 536	96.5	230.0	233.9	248.8	251.0	262.5	89.1
, 548	94.0	238.7	242.8	247.8	249.0	274.8	88.4
, 546	95.0	238.8	242.9	247.4	248.5	275.3	88.2
, 532	94.5	223.8	227.6	245.4	248.2	258.2	88.1
	95.0			247.4	249.2		88.5
, 166	97.9	237.8	241.8	243.1	243.5	259.7	93.1
, 165	98.3	238.3	242.4	243.1	243.5	260.3	93.1
, 164	98.7	239.8	243.9	245.3	245.8	259.4	94.0
, 166	98.0	237.6	241.6	243.2	243.5	259.6	93.1
, 162	98.9	237.9	241.9	243.6	243.7	259.5	93.2
	98.4			243.7	244.0		93.3

c. Improvement of Safety Properties — Task 4

(C) Although an AlH_3 propellant has received an ICC classification of class B (unconfined), a relatively high degree of hazard is still inherent in the manufacture of such propellants. The most significant hazard appears to be in the handling of the AlH_3 itself and in the handling of in-process (uncured) propellant.

(C) One laboratory-scale mixer has been lost on this program because of a fire in the mixer. The fire was initiated while AlH_3 was being fed into the mixer from a feed hopper. Although the hydride in the feed hopper was under a nitrogen blanket, the mixer contained an oxygen-rich premix. The entire processing system was grounded, but substantial charges can build up on the nonconducting hydride surfaces as the particles slide past one another during discharge of the hopper. Studies were initiated to minimize hazards in the handling of the hydride.

(C) Modifications were made to the pilot-scale processing equipment. The fiberglass hoppers and outlet tube used in the multimix operation were treated with a graphite-based coating identified as micro-seal 100-1. This coating decreases the possibility of static charge buildup from the interior walls of the hopper when AlH_3 is loaded into the hopper or discharged into the mixer. The coating is a good electrical conductor from which static charges can be discharged from the system through a grounding strap.

(1) Conductivity Experiments

(C) A more fundamental approach also was taken through efforts to increase the electrical conductivity of the AlH_3 particles. Attempts were made to coat the surface of the particles with graphite by mechanical tumbling of AlH_3 and graphite. The first attempts did not result in successful coating. Attempts to apply 0.1% and 1.0% coatings of graphite did not result in increased electrical conductivity of the material.

(C) Two other materials were investigated for their effect in increasing the electrical conductivity of the AlH_3 particles. One material is a commercial 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 consisted of two 1-in. brass plates held 0.25 in. apart by a plexiglass spacer. The cell is shown in figure 31. A vacuum tube volt-ohmmeter (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	1,000
Dowane 1451, 1% graphite coating	1,000
Dowane 1451, 1% Statikill coating	40 to 70
Dowane 1451, 1% Catanac SN coating	750

(U) It is apparent from these results that the use of an antistatic agent such as Statikill substantially reduces the degree of static buildup on LMH-1. An antistatic agent was not used on this program because of the problems involved in scaleup of the coating operation at a relatively late date in the program schedule. The maximum benefit would result from this approach if the antistatic agent were applied during the manufacture of the LMH-1.

(2) Impact Sensitivity Testing

(C) In connection with the burning rate studies described in section b., 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 XXII. 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 concentrations 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. Also, there is no apparent effect caused by the concentrations of the individual ingredients, AP or AlH_3.

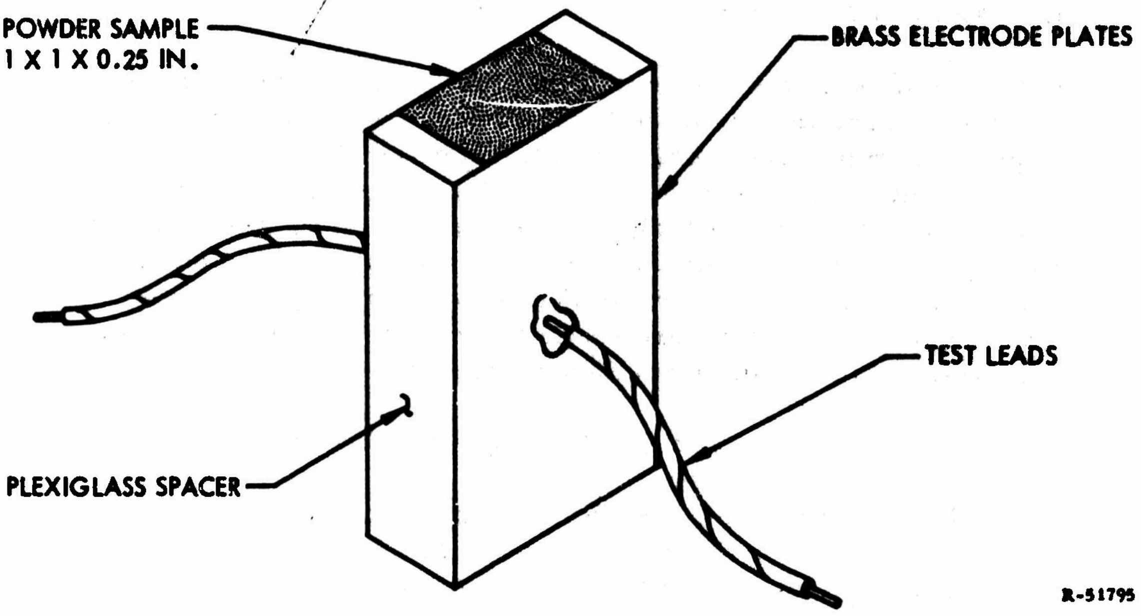

Figure 31. (U) Electrical Conductivity Cell

TABLE XXII

(U) 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
6848-1	25	50	1.2	54.1	26.7	7.3

d. Aging and Temperature Limits — Task 5

(U) This task is devoted to defining the aging characteristics and the storage temperature limitations for this system. Techniques for improving storage limits received attention. The work includes 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. Formulation and processing improvements have resulted in minimizing initial gassing which has an important influence on propellant storability.

(1) Formulation and Processing Improvements

(C) A major problem inhibiting large-scale use of AlH_3 is the frequent occurrence of low density or porous propellant caused by gassing of the AlH_3 during propellant cure. This problem is caused by relatively low thermal stability of some lots of AlH_3 and the chemical reactivity between the hydride and such ingredients or impurities in the propellant that are not completely compatible with the hydride. Efforts to improve propellant density (i. e., eliminate gas evolution) have followed three approaches on this program (1) ingredient purification, (2) aluminum hydride surface treatment, and (3) improved processing techniques.

(a) Ingredient Purification

(U) An extremely rigorous procedure of drying and purifying all propellant ingredients prior to use in propellant was developed. The technique used was to subject the HX-735 polyester to distillation in a molecular still with a wall temperature of 125° C and a vacuum of 10 to 20μ. The MAPO was vacuum distilled. The Epon 812 was diluted in methylene chloride, dried over molecular sieves for 48 hr, 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 at a dew point of -30° F or below. All weighings were done in the dry box, and any subsequent exposure to atmosphere was kept to a bare necessity as required by the processing technique.

(C) Table XXIII presents the results of the first mix series of the ingredients purification study. As expected, the mixes containing highly purified ingredients had higher cure densities compared to cure densities of as-received ingredients. Samples cured in ambient air had better densities than identical samples cured under nitrogen atmosphere. This could be explained by moisture present in the house-line nitrogen, but the dew point of the gas was not monitored. The mix which contained deliberately added water (0.1 wt-%) had the highest density. Because the binder system was weighed out one day prior to propellant mixing, the water may have reacted with the MAPO or Epon which were quite reactive as they had been purified. As expected, the final propellant density was highly dependent upon the purity of the starting ingredients. However, even with the extremely purified ingredients, densities greater than 94.8% of theoretical were not achieved. Apparently, low-stability lots of AlH_3 were used in this test series.

(U) The data of the second series of mixes is presented in table XXIV. The value of this rigorous purification treatment is shown in a comparison of batch 116 which was prepared by the Standard Procedure previously used with batch 112, where all ingredients have been purified. Batch 112 showed a density improvement from 89.5% of theory to 95.6% of theory. To determine which of the ingredients was most critical to purity, a systematic substitution of ingredients, one by one, was made starting with batch 106.

(C) One purified ingredient at a time was replaced with as-received material and the subsequent propellant density determined. In this series of mixes, a blend of several lots of AlH_3 was used to simulate the conditions existing in the scaleup facilities. This particular blended material contained lots that were of marginal stability. Under such conditions, it could be seen in batch 108 that the use of as-received Epon 812 had the most detrimental effect on the propellant density. Interestingly enough, when the Epon 812 effect was checked out with the 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 XXIV. The least effect on propellant density was shown by TMETN.

TABLE XXIII

(U) PURIFIED INGREDIENTS MIX DATA

UTX 6814	1451	Binder Condition	AP Condition	Cure Atmosphere	Top Section	Bottom Section	Average Density g/cc	% Theoretical Density
89	DL-458	Premix	Oven dried	Air	1.455	1.463	1.459	92.0
89	DL-458	↓	↓	N_2	1.431	1.440	1.436	90.5
91	BL-27	↓	↓	Air	1.462	1.459	1.461	92.0
91	BL-27	Premix	↓	N_2	1.435	1.451	1.453	91.6
94	DL-458	Purified	↓	Air	1.491	1.507	1.499	94.4
94	↓	↓	Oven dried	N_2	1.476	1.487	1.482	93.4
90	↓	↓	Vacuum oven dried	Air	1.477	1.516	1.497	94.3
90	DL-458	↓	↓	N_2	1.465	1.489	1.477	93.1
92	BL-27	↓	↓	Air	1.489	1.481	1.485	93.6
92	BL-27	Purified	↓	N_2	1.465	1.489	1.477	93.1
93	DL-458	Purified + H_2O	↓	Air	1.485	1.524	1.504	94.8
93	↓	↓	Vacuum oven dried	N_2	1.476	1.507	1.492	94.0
95	↓	↓	As received	Air	1.462	1.503	1.482	93.4
95	DL-458	Purified + H_2O	As received	N_2	1.452	1.469	1.460	92.0

TABLE XXIV

(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 as-received remainder purified	56	4	1.486	93.6
107	MAPO as received remainder purified	60	4	1.504	94.8
108	Epon 812 as received remainder purified	60	4	1.461	92.0
109	TMETN as received remainder purified	62	3	1.535	98.0
110	TMETN + molecular sieves remainder purified	52	3	1.513	95.3
111	TMETN + 1% ethyl centralite remainder purified	60	3	1.526	96.1
113	AP oven dried remainder purified	50	3	1.507	94.9
116	As received 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	As received binder, BL-28, 3 day SOX Et_2O, oven dried AP	58	4	1.492	94.0
124	Purified ingredient, (DL-482)	69	4	1.586	98.8
138	Epon 812 as received, (DL-482) remainder purified	72	4	1.574	99.2

(C) Mix 129 was made to compare the difference in LMH-1 treatment techniques. Here, the UTC technique of using ether extraction of AlH_3 in a Soxhlet extractor was again evaluated as a stabilization technique for LMH-1. A significant improvement in propellant density was obtained using as-received binder, oven-dried AP with the Soxhlet ether-treated LMH-1 as compared to the acrylonitrile treated LMH-1.

(b) Propellant Degassing Experiments

(U) It has been observed that the most severe gassing of LMH-1 propellant occurs during the first 24 hr of cure. Accordingly, a series of experiments was conducted to study the effect of curing the propellant under vacuum to remove the gas being evolved.

(U) The results of this study are summarized in table XXV. Batch 123 is the control for this study. Propellant density of 98.2% theoretical was obtained in the control. It also should be noted here that DL-482 had been acrylonitrile treated by Dow where the acrylonitrile contained approximately 0.3% water. 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, the 135A was cured under static vacuum at 120° F for 1 day followed by 3 days ambient pressure and cure. Sample 136A was under ambient temperature and static vacuum for 1 day followed by 4 days 120° F cure at ambient pressure. A slight improvement in density was obtained with the 1- and 2-day vacuum treatment at 120° F, but a deterioration of density occurred on the third and fourth day. This, of course, was anticipated because the effect of vacuum during the actual cure should result in a swelling of the propellant. The vacuum exhibited on the ambient temperature samples, however, gave reproducibly high densities. On all four samples, densities from 99.9 to 100.3% of theoretical were obtained. It was believed that the primary difference being observed in these two samples was that at ambient temperature no cure occurred in the 6814 formulation in this time period, and accordingly, when the sample was placed in the 120° F oven for cure, the propellant retained enough fluidity to reconsolidate itself after having been thoroughly degassed.

(U) Because the initial study here had been with a lot of LMH-1 that already exhibited good properties, a check was made on the effect of this technique with blend 28 in batch 140. A modification made to the

TABLE XXV

(U) VACUUM CURE DATA
DL-482 (AN Treated by Dow)

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

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. With this lot of hydride and this particular technique, no gain in density was obtained.

(c) Surface Treatments

(C) Among the many techniques for improving the stability and compatibility of AlH_3 the most commonly used is that of a surface passivation obtained by immersing the hydride in a medium such as acrylonitrile. A modification of this technique involves the addition of a fraction of a percent of water to the acrylonitrile. Another technique used 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 demonstrated that the Taliani stability of AlH_3 could be significantly improved by reacting AlH_3 directly with water. Surface oxidations of the order of 0.3% to 1% gave significant improvements in short-term thermal stability of neat material.

1. Use of Oxidizers

(C) A technique originally studied under Contract No. AF 04(611)-9570 by UTC for measuring the degree of reactivity of AlH_3 was evaluated as a surface oxidation technique for improving the stability and compatibility of AlH_3. This approach to improvement in propellant density was to react the LMH-1 surface with oxidizers under controllable laboratory conditions. The quinones, Alizarin (1, 2- dihydroxyanthroquinone) and Alizarin Red S, the sodium Alizarin sulfonate salt, were selected for the initial study. The Alizarin was dissolved in benzene, and a 1451 lot 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 known. It was assumed that an aluminum chelate was formed causing the red coloration on the LMH-1 surface. Table XXVI is

TABLE XXVI

(U) ALIZARIN DATA

UTX 6814		Density g/cc	% Theoretical Density	Shore A
142	BL-28 as received, as-received ingredient	1.388	87.5	43
116	BL-28 UTC AN, as-received ingredient	1.420	89.5	46
130	BL-28 + 1% Alizarin, as-received 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, as-received ingredient	—	92.0	—
131	DL-458 + 1% Alizarin Red S, ground 15 min, as-received ingredient	1.493	94.1	55
132	DL-458 + 1% Alizarin Red S, ground 15 min, as-received ingredient	1.555	98.0	35 soft cure

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a summary of the experimental data. The difference between batches 130 and 139 was that the excess Alizarin was removed. It was interesting to note that the hydride with no excess Alizarin had a red coated surface. This surface was not soluble in benzene and could not be removed from the hydride. It was possible, therefore, that an organic coating of the reduced Alizarin had been formed by the chelate formation of the aluminum. To obtain the maximum effect from the Alizarin treatment in batches 131 and 132, DL-458 was wet ground, i. e., ground in benezene with dissolved Alizarin or Alizarin Red S in it. This resulted in exposure of fresh surfaces for reactions with the oxidizing agents. The biggest gain in density was obtained with the Alizarin Red S treated fuel. This batch had the disadvantage of a soft cure as indicated by the low Shore A rating of 35. There was possibly some interaction with the quinone and the cure mechanism.

(C) To separate the effects of the surface oxidation obtained with the reaction of the quinone and hydride from that of the subsequent reaction of chelate formation with the oxidized aluminum another quinone, 2, 6-dibromo quinone, was evaluated. This material should react without any subsequent side reactions such as the chelate formation obtained with the Alizarin. No improvement in propellant density was obtained when the treated LMH-1 was mixed into a formulation.

(U) 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 show 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 was obtained through the Alizarin studies.

2. Use of 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 phenolthiazine (PTA). They have added the above materials during and after formation of AlH_3 and propose the following MBT LMH-1 reactions occur.

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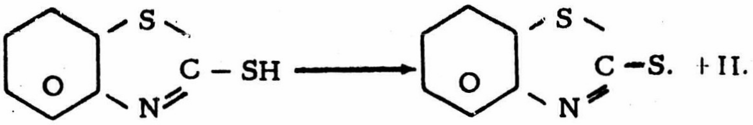
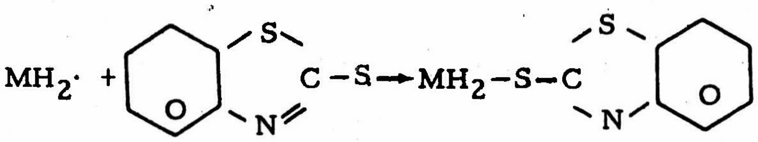
* Olin Mathieson Chemical Corporation Quarterly Report No. 95648, June 30, 1964, Contract No. DA-19-020-ORD-5648.

† Dow Chemical Company Report No. AR-3Q-64, November 1, 1964, Contract No. AF 04(611)-7554.

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



B. Termination:



(C) The termination reaction would prevent the further decomposition of a 1451 polymer chain with a minimum of hydrogen gas involved. It was hypothesized that if this mechanism were correct, the aging characteristics of AlH_3 propellant could be improved.

(C) Table XXVII summarizes the experimental data of both PTA and MBT coated LMH-1 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 densities improvements were obtained. Another part of this study was to add MBT directly to the binder instead of coating the hydride. Batch 141 contained added MBT and batch 124 contained no additional ingredients. DL-482 was acrylonitrile treated by the Dow 0.3 wt-% water acrylonitrile method. Here a definite drop in density (4%) was noticed when MBT was added to the mix.

(U) Although some improvement in density was obtained from the use of the two free radical inhibitors tested, their use was not recommended as aids to improve densities because all the propellants tested suffered from discoloration and poor cures. It is possible, of course, 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.

TABLE XXVII

(U) FREE RADICAL INHIBITOR DATA

UTX 6814	Formulation Variable	Density g/cc	% Theoretical Density
142	BL-28 as received, as-received binder	1.388	87.5
125	1/2% PTA + BL-28 as received, as-received binder	1.461	92.0
126	1% PTA + BL-28 as received, as-received binder	1.420	89.5
127	1/2% MBT + BL-28 as received, as-received binder	1.472	92.7
128	1% MBT + BL-28 as received, as-received binder	1.420	89.5
89	DL-458, as-received binder		92
133	DL-458 + 1% PTA ground 15 min, as-received binder	1.480	93.2
134	DL-458 + 1% MBT ground 15 min, as-received binder	1.384	87.1
137	DL-458 ground 15 min in molecular sieve-dried acrylonitrile	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

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3. Use of Water Treatment

(U) Numerous companies have reported that surface oxidation using a controlled hydrolysis process can be used to increase LMH-1 stability. Some of the various techniques are passing water wet gases through the hydride, reacting with liquids containing a small amount of water, and adding the LMH-1 directly to water.

(C) Two samples of LMH-1 were treated by adding the LMH-1 directly to water. The selected lots were blend 29, (BL-29), a blend of untreated LMH-1 and blend 32, (BL-32) a blend of acrylonitrile treated LMH-1. One-g samples were reacted with 50 ml of water. The decomposition rates and degrees of hydrolysis were monitored by measuring the hydrogen gas generated according to the equation:

$$AlH_3 + 3H_2O = Al(OH)_3 + 3H_2$$

(U) Figure 32 illustrates the hydrolysis rates of the two LMH-1 samples. The initial gassing reaction was rapid for BL-29 and after 50 min of reaction time, there was another sharp increase in the decomposition rate. The other sample, BL-32, displayed a slow hydrolysis rate, and no noticeable decomposition break was observed in the gassing curve. Part of the observed stability differences was a result of previous hydrolysis of BL-32 during the acrylonitrile treatment, which contained 0.3 wt-% water.

(C) In order to treat a sufficient quantity of LMH-1 to allow propellant processing, the surface oxidation process was scaled up to treat 50 g of BL-29. After reacting the fuel to the desired amount of surface hydrolysis, the reaction was terminated by vacuum drying the mixture using a coarse frit Buchner funnel. The final drying was completed in a dry box so that the LMH-1 would be under an inert gas atmosphere during the final moisture removal stage.

(U) Mixes were processed to compare the densities of propellants containing untreated and water-treated BL-29. Formulation UTX 6814-172 contained treated BL-29 (1% decomposition, estimated from figure 32), and the control formulation UTX 6814-173 contained untreated LMH-1.

(C) The treated fuel propellant had a measured density of 1.55 g/cc (96% of theoretical density)

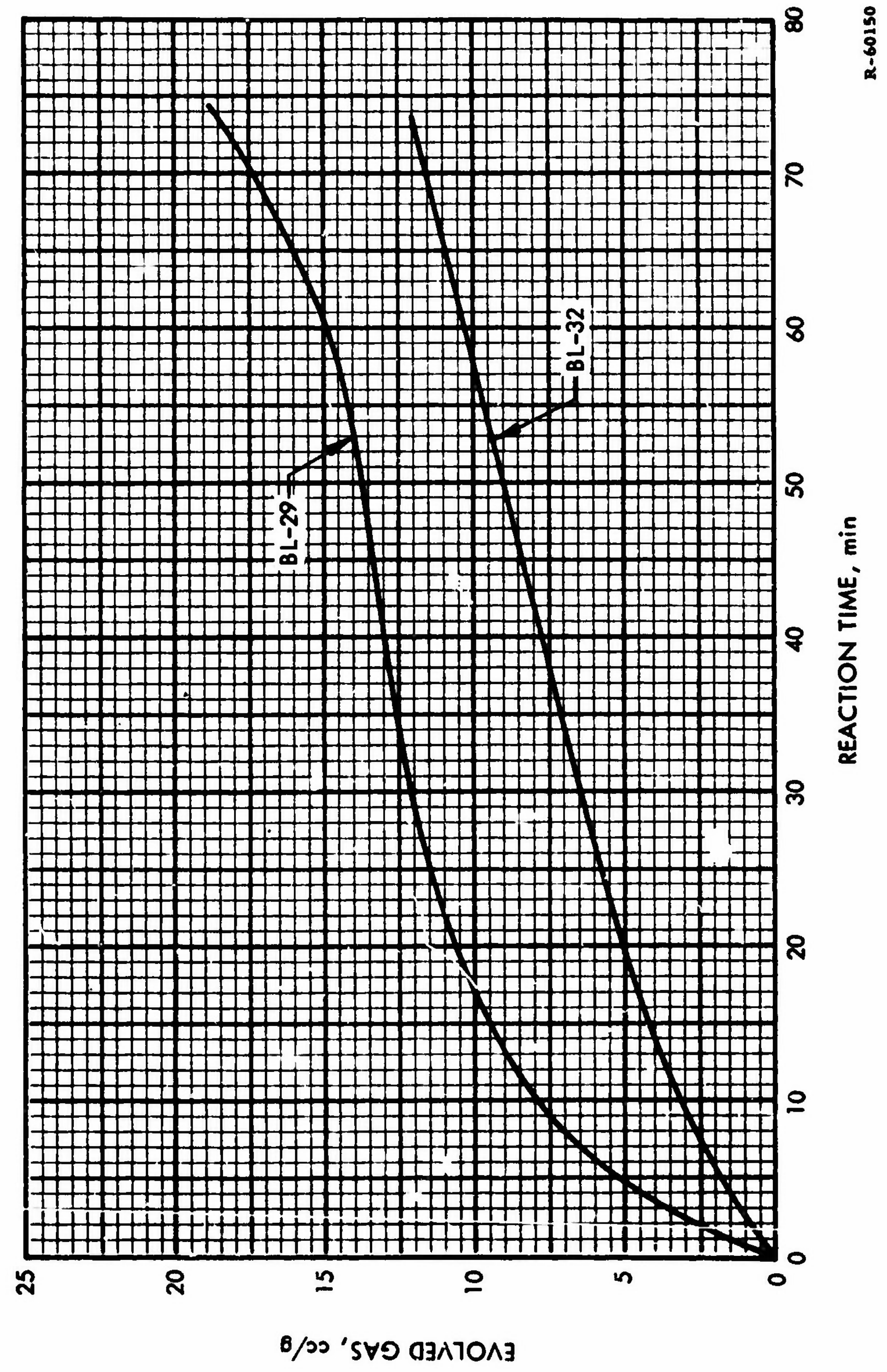

Figure 32. (U) Hydrolysis Rate of Two Lots of Dowane 1451

and the untreated LMH-1 propellant had a measured density of 1.47 g/cc (93% of theoretical density). In the investigation the controlled hydrolysis process LMH-1 propellant had a definite improvement in measured density over the untreated LMH-1. propellant.

(C) In two other tests, the hydride was water treated using quantities of hydride weighing greater than 70 g. Difficulties were encountered treating the hydride in bulk as it was impossible to dry the hydride rapidly enough to prevent excess decomposition.

(U) From the results of the preceding studies, it was apparent that a surface oxidation using a controlled hydrolysis process can improve LMH-1 stability in propellants. However, handling procedures need to be developed to rapidly and efficiently remove the water to prevent excess hydrolysis and decomposition.

(U) The most promising techniques involving surface passivation, oxidation/reduction, and free radical inhibition were evaluated in 2-in. cubes.

(U) Cubes (2-in.) of UTP 6814 formulation were made up using various treatments and additives as summarized in table XXVIII. These cubes were subjected to a screening test in which the temperature was raised 10° F each day until obvious failure occurred. One set of cubes also was placed in storage at 35° C to determine the long-term stability.

(U) The samples placed in the 10° F/day storage were placed in individual expendable ovens equipped with a remote measuring device so that the swelling could be measured daily to give a quantitative measure of the degree of decomposition. Results from a set of cubes run up to 190° F at the above rate are summarized in table XXVI. These data are considered to be the most reliable obtained in this experiment. It is evident that except for the

TABLE XXVIII

(U) THERMAL SWELLING DATA FOR EXPERIMENTAL FORMULATIONS IN PROGRESSIVE HEATING TEST

Formulation	Treatment	Thermal Swelling, cm										
		90° F	100° F	110° F	120° F	130° F	140° F	150° F	160° F	170° F	180° F	190° F
UTX 6814-165	2-6-dibromoquinone chloroimide	0	0	0	0.01	0.10	0.15	0.15	0.20	0.35	0.80	0.80
UTX 6814-166	Acrylonitrile	0	0	0.01	0.03	0.06	0.10	0.10	0.20	0.30	0.35	0.40
UTX 6814-168	1% ethyl centralite	0	0	0.04	0.10	0.13	0.15	0.15	0.20	0.35	0.50	0.75
UTX 6814-169	0.3% water	0	0.10	0.11	0.22	0.35	0.40	0.40	0.40	←Disintegrated→		
UTX 6814-170C	0.5% molecular sieve	0	0	0.02	0.02	0.08	0.10	0.10	0.20	0.22	0.65	0.70
UTX 6814-171	Ether extracted	0	0	0.01	0.05	0.10	0.10	0.10	0.10	0.20	0.55	0.55
UTX 6814-175	None	0	0	0	0.01	0.05	0.05	0.05	0.05	0.15	0.55	0.65
UTX 6814-177	Acrylonitrile	0	0	0	0.02	0.04	0.04	0.04	0.04	0.04	0.15	0.15
UTX 6626-23C	Aluminized control	0	0	0	0	0.01	0.01	0.01	0.01	0.01	0.01	0.01

aluminized control formulation, the greatest stability was obtained with the simple acrylonitrile treatment. None of the other treatments appear to have been effective.

(C) The results of the long-term storage tests are summarized in table XXIX. Here again, the best storage properties were displayed by the propellant containing acrylonitrile-treated AlH_3.

(2) Differential Thermal Analysis Experiments

(U) The static DTA experiment was designed to detect any thermal event occurring within the propellant upon prolonged storage which might lead to spontaneous cook-off in stored motors. Three micromotors of UTP 6814 composition were placed in storage at 35° C and relative humidity levels of 0%, 50%, and 90%, respectively. There was no significant thermal event recorded up to 2, 900 hr.

(C) Previously, micromotors of the UTP 6814 composition showed no thermal event up to 1, 000-hr storage at 50° C and 0%, 50%, and 90% humidity. The experiment was repeated at 35° C because the 50° C experiment was not considered to be conclusive. At the higher temperatures the decomposition of the AlH_3 had progressed to the point where visible degradation of the propellant had taken place by the time the test was terminated, and this may have overshadowed an effect of the nitrate ester. The test at the lower temperature was considered to be more realistic in this respect.

(U) As in the 50° C experiment, the samples tested were end-burning micromotors of 1 in. length and 1. 25 in grain diameter. These 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 humidity, 29 wt-% for 90% relative humidity). The sample tested under dry conditions was suspended over Drierite. An iron-constantan thermocouple was imbedded in each sample, and the temperatures were read daily at first and later semiweekly. A fourth thermocouple was inserted into an inert rubber sample to serve as a reference.

TABLE XXIX

(U) THERMAL SWELLING OF EXPERIMENTAL FORMULATIONS IN LONG-TERM STORAGE (% SWELLING)*

Temperature: 95° F
Relative Humidity: 0

		Time, days							
Formulation	Treatment	7	28	41	68	100	133	161	209
6814-164C	Alizarin	1.19	1.41	1.43	1.51	1.53	1.53	1.48	1.48
6814-165	2,6-dibromoquinone chloroimide	0.69	0.88	1.05	1.18	1.13	1.20	1.23	1.13
6814-166	Acrylonitrile	0.40	0.40	0.37	0.37	0.35	0.27	0.20	0.27
6814-167	Alizarin Red S	1.01	0.79	0.86	0.76	0.74	0.66	0.66	0.66
6814-168	1% ethyl centralite	0.94	0.91	0.91	1.09	0.84	0.79	0.74	0.74
6814-169	0.3% water	0.69	0.66	0.83	1.15	1.50	1.50	1.42	1.45
6814-170C	0.5% molecular sieve	1.39	1.25	1.27	1.20	1.22	1.20	1.12	1.15
6814-171	Ether extracted	1.11	1.36	1.43	1.58	1.58	1.53	1.51	1.01
6626-23C	Aluminized control	0.40	0.22	0.22	0.20	0.20	0.20	0.22	0.20

* Average swelling in one direction

(U) These samples were kept in storage at temperatures ranging between 32° and 35° C for 2, 900 hr. There was no significant thermal event up to that time, and the test was terminated.

(U) Dynamic DTA traces run on samples of UTP 6814 stored under various conditions have shown no significant variation over a storage period of 7 months.

(U) Five 2-in. cubes of UTP 6814 propellants were placed in storage under the following conditions of temperature and humidity:

25° C, 0% and 50% relative humidity

35° C, 0% and 50% relative humidity

45° C, 0% relative humidity

The controlled temperature environment was provided by existing propellant storage facilities: thermostated rooms at 25° and 35° C, and an oven at 45° C. Individual storage boxes within the thermostated spaces provided the controlled humidity. The boxes for storage of dry samples contained trays of Drierite, while trays of glycerine/water solutions were used to provide the controlled relative humidities. These samples were in storage for 7 months and were sampled at intervals of approximately 4 weeks. The samples were examined by conventional DTA to determine any shift in the characteristic thermal peaks which might be indicative of chemical change in the propellant. There was no detectable shift in any of the peaks which could be interpreted as an internal chemical change.

(3) Gas Evolution

(C) Gas evolution studies conducted on magnesium-doped AlH_3 show a high degree of stability both in the neat form and in UTP 6814 propellant. All the magnesium-doped samples showed more stability than a comparable untreated hydride, and the rate of gas evolution as a function of magnesium content was parallel to that reported by Dow Chemical Company. Formulations using the undoped hydride in a Uteflex binder were more stable than those based on HX-735, but were more susceptible to attack by moisture probably caused by the more hydrophilic nature of the Uteflex polymer backbone.

(U) The gas evolution studies were carried out in the improved Taliani-type apparatus shown in figure 33. Improvements over previously used apparatus include (1) the complete elimination of stopcocks and greased joints which permit very long runs to be made without interference from leaks, and (2) the use of a layer of silicone oil on the mercury in the manometers to prevent diffusion of mercury vapor into the samples. At the beginning of a run the sample was enclosed in the apparatus, as shown in figure 33, and the apparatus was swept out with inert gas. The open ends were then sealed to close off the system from the atmosphere. No attempt was made to maintain constant volume during the run, and the increase in volume caused by the manometer deflection was accounted for in the final calculations. The volumes of the various parts of the system were determined at the termination of the run when the system was disassembled.

(C) Gas evolution measurements were made to investigate three areas of interest (1) the stability of magnesium-doped AlH_3 compared to the undoped material — both neat and in propellant, (2) the relative stabilities of propellants based on Uteflex and HX-735 propellants. Results in these three areas are shown in figures 34, 35, 36, and 37, respectively.

(C) Figure 34 shows gas evolution from three batches of magnesium-doped Dowane 1451 along with data from an undoped sample. The magnesium-doped material shows considerably improved stability compared to the undoped sample. Figure 35 shows the gas evolution from the same lots of hydride incorporated in the UTP 6814 formulation. Here the contrast is even more marked with the DL-497 control sample showing decomposition approaching 10% in 400 hr, while the magnesium-doped material had still not reached this level after 3,000 hr.

(C) Figure 36 shows gas evolution from undoped AlH_3 (lot DL-497) incorporated in the Uteflex binder and in the UTP 6814 (HX-735) formulation. Data from a sample of neat hydride are included for comparison. The neat hydride shows the most rapid initial gas evolution, probably because of initial solution or trapping of gas in the propellant samples. At longer times, however, the propellant samples show the greater amount of gas evolution with the Uteflex formulation showing by far the greater stability.

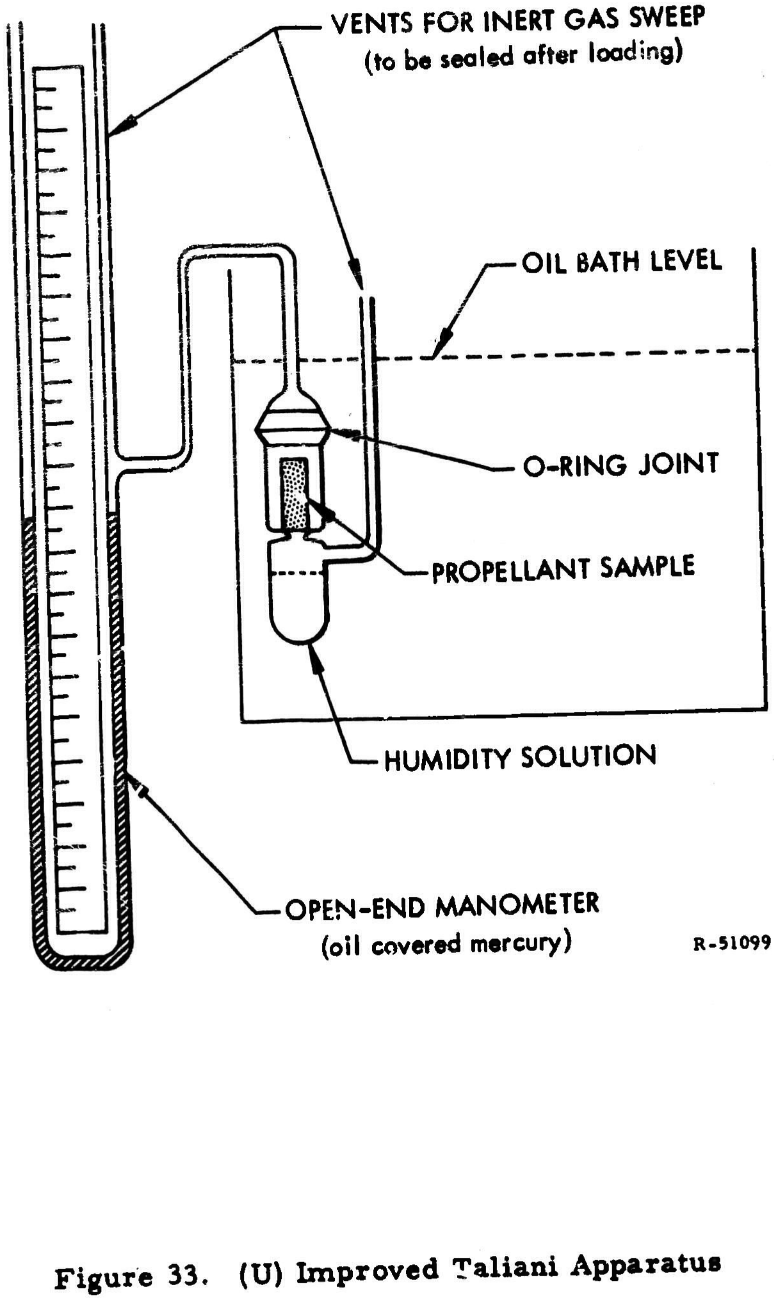

Figure 33. (U) Improved Taliani Apparatus

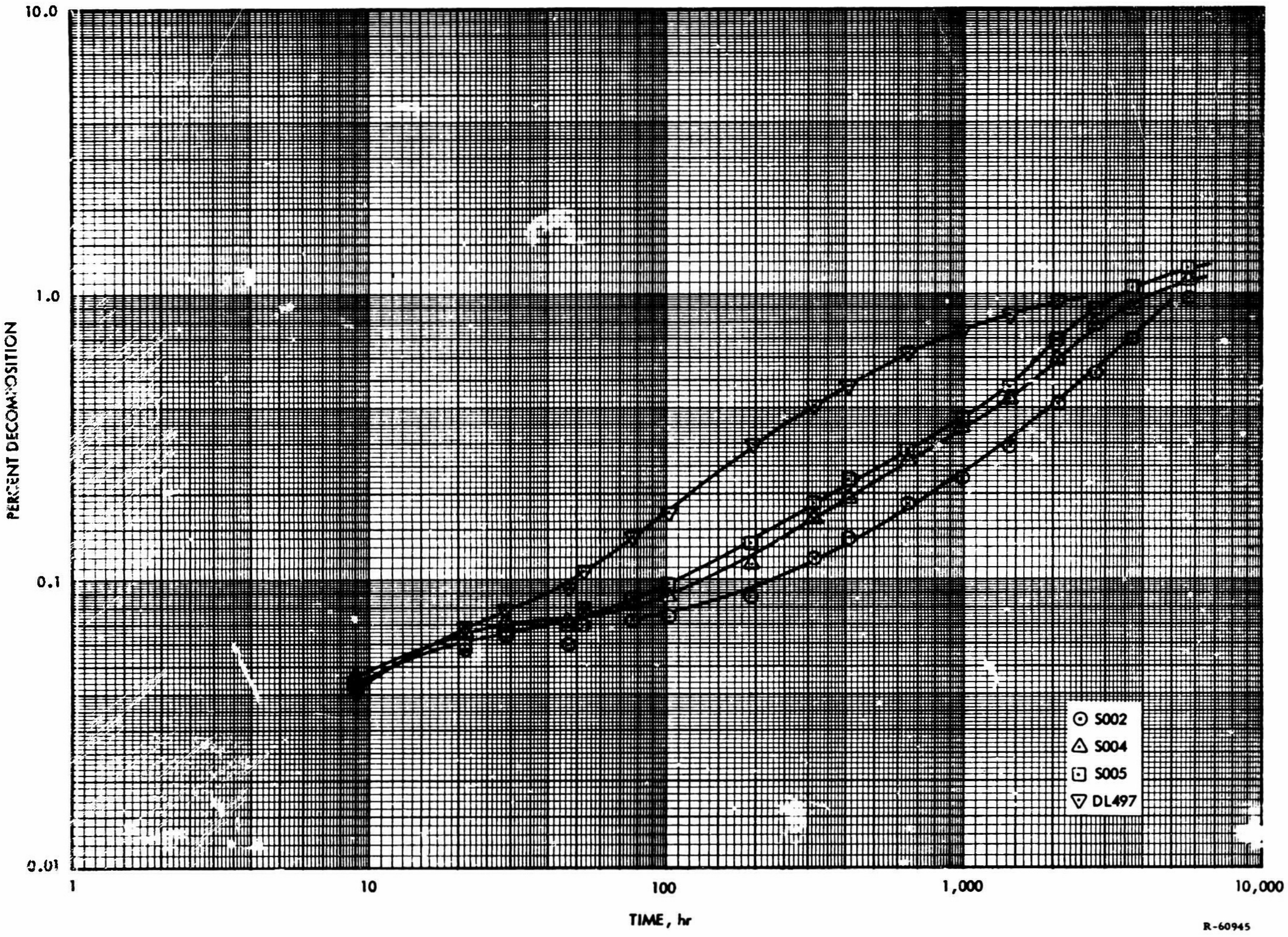

Figure 34. (C) Gas Evolution from Magnesium-Doped Dowar 1451

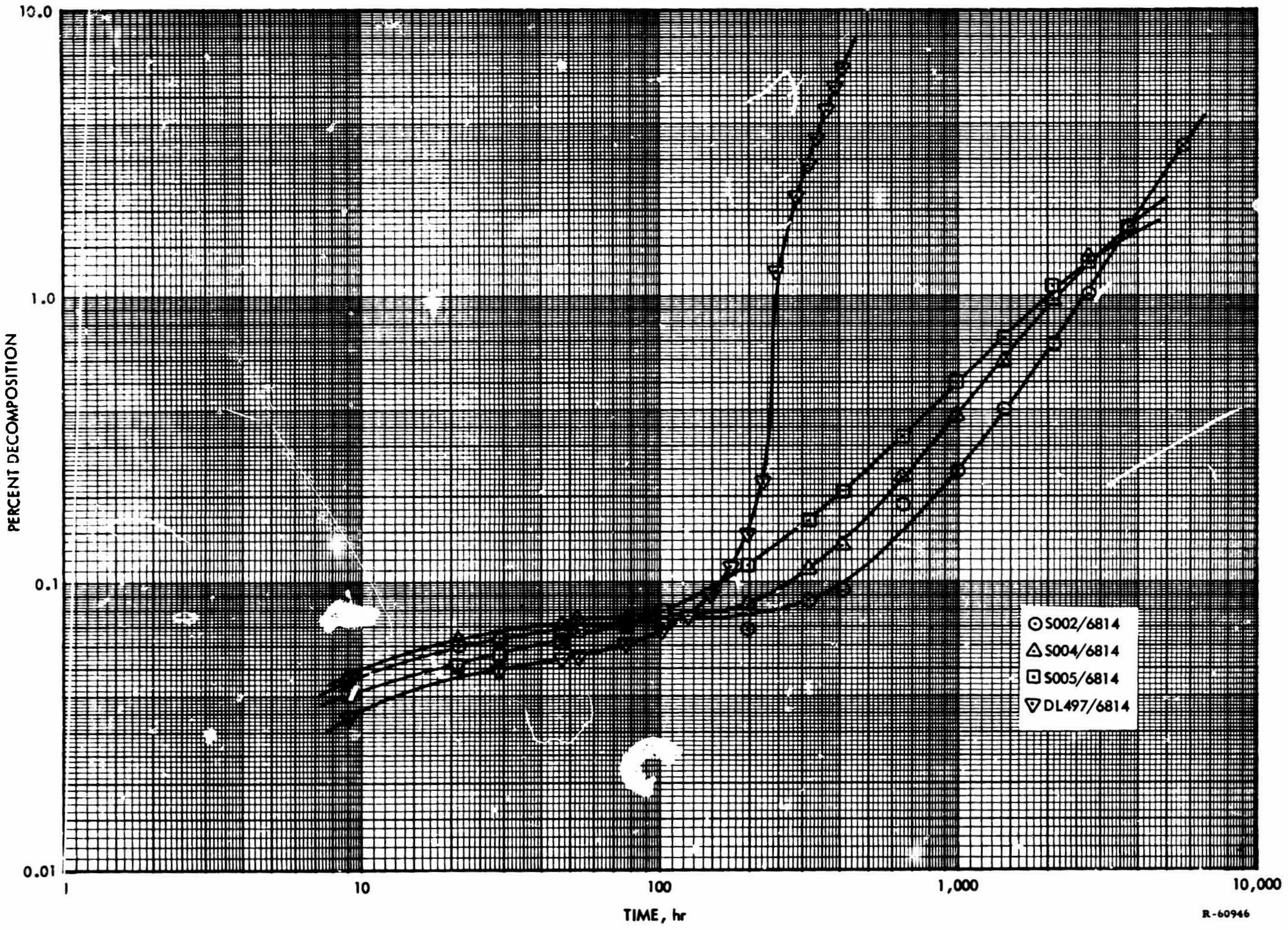

Figure 35. (C) Gas Evolution from Magnesium-Doped Dowane 1451 in UTP 6814

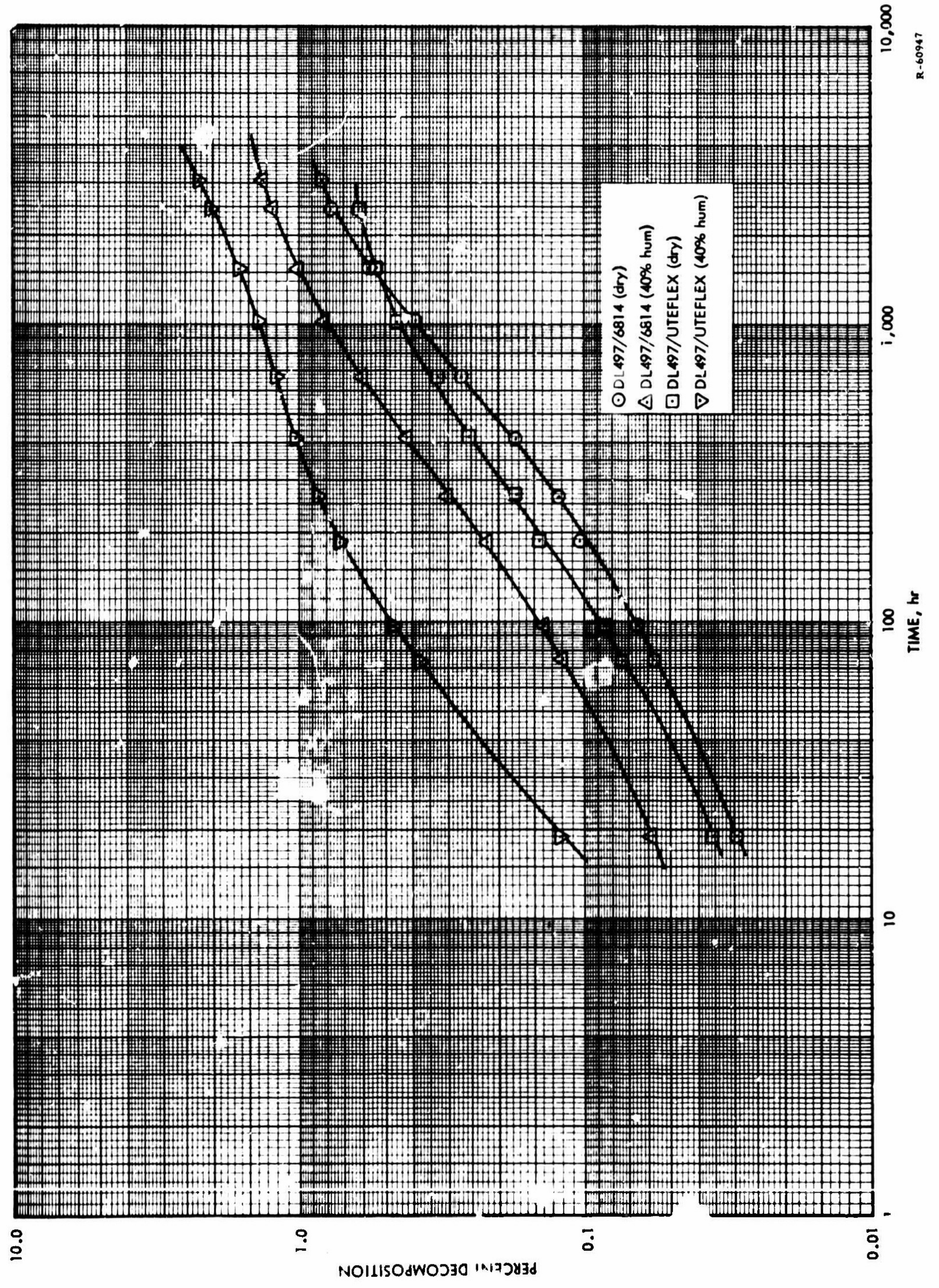

Figure 36. (U) Effect of Moisture on Gas Evolution

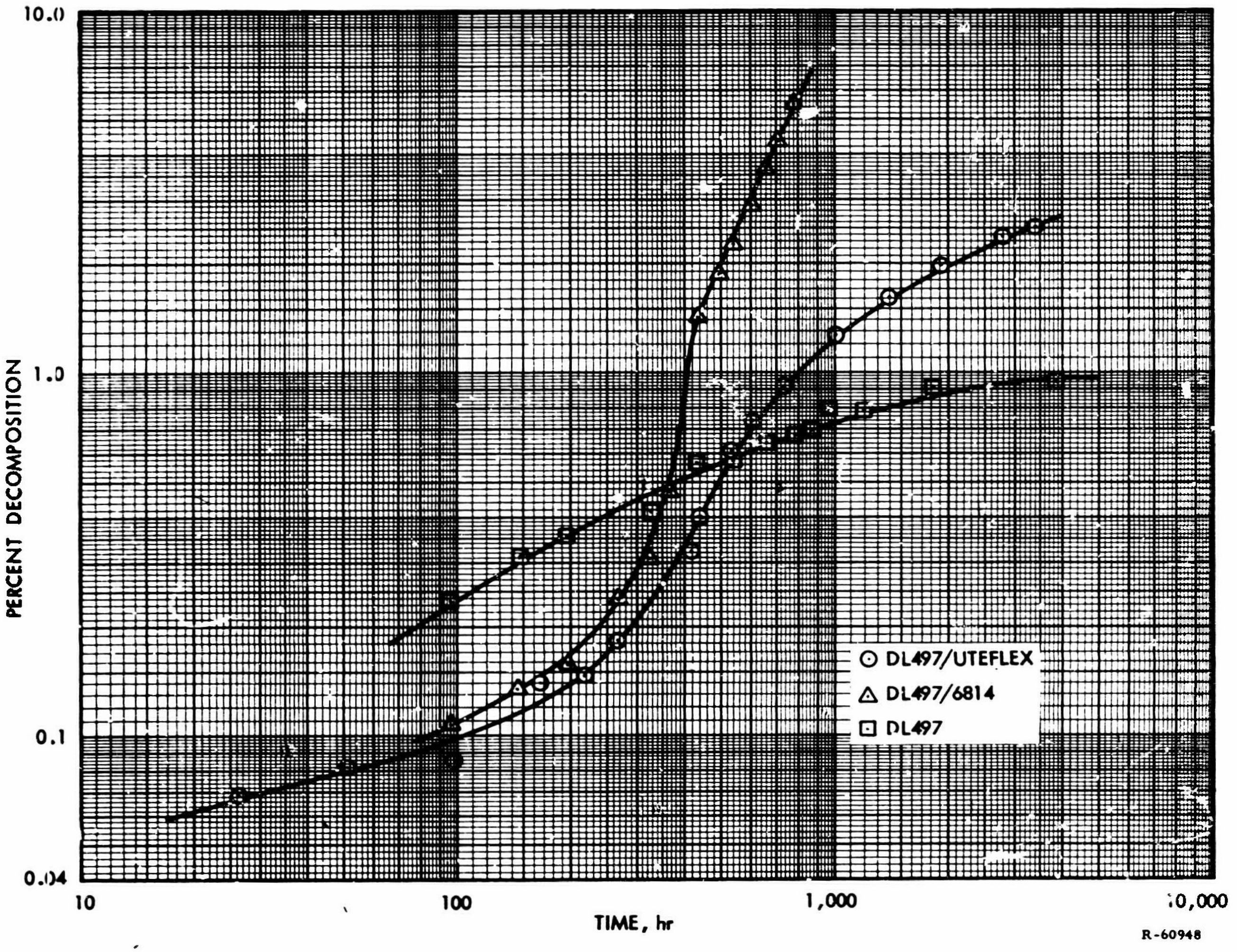

Figure 37. (U) Gas Evolution from Uteflex and UTP 6814 (HX-735) Formulations

(C) Figure 37 shows the effect of moisture on the gas evolution from the Uteflex and UTP 6814 formulations. The gas evolution is increased in both cases, but the Uteflex is more sensitive to moisture, probably because of the more hydrophilic nature of the polymer backbone which allows easier penetration by water.

(C) It should be noted that in all the above cases the percent decomposition of the samples was calculated on the basis of the weight of hydride in the samples according to the stoichiometry

$$AlH_3 \rightarrow Al + \frac{3}{2} H_2 \qquad (1)$$

In the case of reaction with moisture, the reaction

$$AlH_3 + 3\ H_2O \rightarrow Al\,(OH)_3 + 3\ H_2 \qquad (2)$$

undoubtedly takes place with the production of twice as much gas as by equation 1. A similar effect would occur in reactions involving acidic propellant ingredients. Because the degree to which this takes place is unknown, it was considered more meaningful to use equation 1 consistently.

(4) Gas Diffusion and Failure

(C) The effect of various conditions of temperature and humidity on the swelling of 2-in. cubes of UTP 6814 propellant was studied at two different propellant density levels. In general, the swelling was greatest at higher temperatures and higher humidities as expected. The lower density propellant also showed a greater sensitivity to heat and moisture.

(C) The storage conditions and corresponding swelling data are detailed in table XXX. Formulations UTP 6814-174 and -175 (high density) and UTP 6814-176 and -177 (low density) represent the primary effort under this phase of the program. Six storage conditions were chosed as follows:

25° C and 0%, 50%, and 90% relative humidity

35° C and 0% and 50% relative humidity

45° C and 0% relative humidity.

The facilities for maintaining these conditions are described in section (2), DTA experiments.

TABLE XXX

(U) THERMAL SWELLING OF CUBE SAMPLES (%)*

Temperature °C	Relative Humidity %	Time, days 7	28	41	68	100	133	161	209
		A. Formulation UTP 6814-174, density 96.1% of theoretical Formulation UTP 6814-175, density 96.6% of theoretical							
25	0	-0.05	0.32	0.15	0.30	0.37	0.47	0.45	0.5
25	50	-0.10	0.27	0.42	0.94	1.49	1.66	1.81	1.86
25	90	0	4.48†	5.96†	---	---	---	---	---
35	0	0.52	0.55	0.82	0.84	0.77	0.82	0.74	0.79
35	50	0.57	1.02	1.24	1.44	1.49	1.49	1.41	1.44
45	0	0.52	1.31‡	1.38‡	1.59‡	1.56‡	1.39‡	1.34‡	1.04‡
		B. Formulation UTP 6814-176, density 89.5% of theoretical Formulation UTP 6814-177, density 89.8% of theoretical							
25	0	-0.27	0.24	0.39	---	---	---	---	---
25	50	-0.15	1.37	2.05†	2.69†	3.03†	3.10†	3.08†	3.20†
25	90	0.15	4.19	4.44‡	---	---	---	---	---
35	0	1.22	1.28	1.30	1.32	1.25	1.20	1.23	1.23
35	50	2.31†	2.63†	2.76†	2.68†	2.73†	2.76†	2.61†	2.60†
45	0	1.32	2.97†	3.23†	3.41†	3.02†	3.11†	2.99†	2.53†

* Average swelling in one direction

† Surface blistered

‡ Bulged visibly

(U) These samples were in storage for 7 months and were measured at intervals of about 4 weeks. The cubes were measured in two directions using a fixture equipped with a dial indicator capable of being read to 0.001 in.

(U) Swelling data taken up to the present are summarized in table XXX which gives the measured swelling in percent as a function of time for the various storage conditions. The swelling was most rapid at the higher temperatures and humidities as expected. The effect of humidity was primarily noticeable at the surface of the cubes by a tendency toward blistering.

(U) It is anticipated that these data will provide an interesting comparison when corresponding data for the Uteflex formulation becomes available. Cubes of the Uteflex formulation are presently in storage. The results will be published at a later date as an addendum to this report.

(5) Strain Effects

(U) The effect of strain on the failure properties of UTX 8812 propellant is being measured by subjecting steel-case analogue motors to various time, temperature, and humidity conditions. JANAF samples stored along with the analogue motors provide zero strain reference data. All analogue motors were cast with the same mandrel size to ensure that initial strain is the same for all tests. However, the moisture and temperature effects should induce swelling, so that strain may be relieved gradually throughout the surveillance program especially for those motors stored at the highest temperature and humidity conditions. A complete report on this experiment will be issued as a part of the addendum to this final report.

e. Improvement of Mechanical Properties — Task 6

(C) 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 AF 04(611)-9570 were in the range of 72-psi maximum stress load and 16.5% maximum strain load. The stated objective of the current study is to obtain measured values of 100 psi maximum stress and 20% strain at maximum stress. However, on the basis of various mission analyses, potential applications have been identified for AlH_3 propellants which require measured strain values of 25% to 30%. Corresponding stress requirements are reduced. A further objective of these studies is to obtain a complete cure in the shortest time at 120° F or below to minimize the thermal exposure of AlH_3.

(U) The approaches toward solving these problems have been to seek new crosslinking agents, new polymers, and to identify possible stabilizers that would meet the program goals.

(1) Development of an Improved Curative System

(U) Previous problems with the degradation of physical properties over extended storage times in the nitrato-polyester (PEP 100) binder system were believed to be the result of the relative susceptability to scission of the P-N bond in the MAPO curative. Therefore, evaluation of new crosslinking agents received early emphasis. In general, more reactive materials were sought as it is also desirable to minimize the required cure time and cure temperature. Because several prospective curatives were immediately available they were evaluated with HX-735, a terminally carboxylated polyester. The candidate curatives were evaluated over a range of curative/polymer equivalent ratios. Both unplasticized and TMETN plasticized formulations were evaluated. A cure temperature of 120° F was used.

(U) The following is a list of the curatives that were in the evaluation:

	Vendor
TBM, Tris-(2 ethylaziridinyl)-s-triazine	American Cyanamid
MAPO, Tris(1,2 methyl) aziridinyl phosphine oxide	Interchemical Corp.

	Vendor
NTEB, Nitrilotriethyl-β-ethyleniminobutyrate	American Cyanamid
NTPB, Nitrilotriethyl-β-propyleniminobutyrate	American Cyanamid
MAES, Bis(2 methyl aziridinylethyl) sulfone	American Cyanamid
HMAT, Hexamethyl aziridinyl triazine	Interchemical Corp.
NC 1034, Polyethylene imine polymer	Dow Chemical Co.
NC 10262, Polyethylene imine polymer	Dow Chemical Co.
HX-858, propylene imine adduct of trimesic acid	3M Company
HX-868 butyl imine adduct of trimesic acid	3M Company
HX-874 tris-(2 ethylaziridinyl)-s-triazine	3M Company
MAM, (a proprietary imine compound)	American Cyanamid
Epon 812, (n aliphatic epoxide)	Shell Chemical Co.

(U) Initial observations were qualitative and were used to screen the available candidates. A summary of these results is presented in tables XXXI and XXXII.

(U) The best results were obtained with NTPB and blends of NTEP-NTPB, NTEB-MAPO, and NTPB-MAPO. Qualitative tests also were performed with HX-874 and TBM, but at a later date as these curatives were not available at the beginning of the program.

(U) Following the screening tests, binder ring samples were prepared to obtain quantitative values of the binders in which NTPB, NTEB, and MAPO were used as curatives. These tests were made on an Instron Tensile Tester. The data are presented in table XXXIII. The results indicated that the NTPB-NTEB blend and NTPB, unblended, were the best of the series.

TABLE XXXI

(U) BINDER CURES WITH UNPLASTICIZED HX-735
(HX-735 Polymer Equivalents = 1.0)

Formulation	Curative and Equivalents	Cure Time Hr	Results
182-88-1	NTEB (1.0)	64	Poor cure
88-2	NTEB (1.5)	64	Poor cure
88-3	NTEB (1.8)	64	Poor cure
88-4	NTEB (2.1)	64	Poor cure
182-90-1	NTPB (1.2)	64	Very good cure
90-2	NTPB (1.5)	64	Poor cure
90-3	NTPB (1.8)	64	No cure
90-4	NTPB (2.1)	64	No cure
182-92-1	NTEB (0.6) NTPB (0.6)	64	Fair cure
92-2	NTEB (0.75) NTPB (0.75)	64	Fair cure
92-3	NTEB (0.9) NTPB 0.9)	64	Good cure
92-4	NTEB (1.05) NTPB (1.05)	64	Poor cure
182-94-1	NTEB (0.3) NTPB (0.9)	64	Good cure
94-2	NTEB (0.9) NTPB (0.3)	64	Poor cure
94-3	NTEB (0.45) NTPB (1.35)	64	Poor cure
94-4	NTEB (1.35) NTPB (0.45	64	Poor cure
182-96-1	HMAT (1.75)	64	Poor cure
96-2	HMAT (1.50)	64	Poor cure
96-3	HMAT (1.50) MAPO (0.25)	64	Poor cure
96-4	HMAT (1.50) EPON 812 (0.25)	64	Poor cure
182-98-1	NTPB (1.2) NC 1034 (1.0)	90	Poor cure
98-2	NTPB (1.2) NC 1034 (0.5)	90	Poor cure
98-3	NC 1034 (1.0)	90	Poor cure
98-4	NC 1034 (1.5)	90	Poor cure

TABLE XXXI (Continued)

Formulation	Curative and Equivalents	Cure Time Hr	Results
182-99-1	NTPB (1.2) NC 1026L (1.0)	120	No cure
99-2	NTPB (1.2) NC 1026L (0.5)	120	No cure
99-3	NC 1026L (1.0)	120	No cure
99-4	NC 1026L (1.5)	120	No cure
182-74-1	MAPO (1.0) NTEB (0.2)	168	Good cure
74-2	MAPO (1.0) NTEB (0.4)	168	Fair cure
74-3	MAPO (1.0) NTEB (0.6)	168	Poor cure
74-4	MAPO (1.0) NTEB (0.8)	168	Poor cure
182-75-1	MAPO (1.0) NTPB (0.2)	168	Poor cure
75-2	MAPO (1.0) NTPB (0.4)	168	Good cure
75-3	MAPO (1.0) NTPB (0.6)	168	Fair cure
75-4	MAPO (1.0) NTPB (0.8)	168	Poor cure
182-76-1	MAPO (1.0) MAM (0.2)	168	Fair cure
76-2	MAPO (1.0) MAM (0.4)	168	Poor cure
76-3	MAPO (1.0) MAM (0.6)	168	Poor cure
76-4	MAPO (1.0) MAM (0.8)	168	Poor cure
182-77-1	MAPO (1.0) MAES (0.2)	168	Good cure
77-2	MAPO (1.0) MAES (0.4)	168	Good cure
77-3	MAPO (1.0) MAES (0.6)	168	Good cure
77-4	MAPO (1.0) MAES (0.8)	168	Good cure
182-84-1	NTEB (1.0) MAM (0.5)	64	Poor cure
84-2	NTEB (1.0) HMAT (0.5)	64	Poor cure
84-3	NTEB (1.0) MAM (0.8)	64	Very poor cure
84-4	NTEB (1.0) HMAT (0.8)	64	Very poor cure
182-86-1	NTPB (1.0) MAM (0.5)	64	Fair cure
86-2	NTPB (1.0) HMAT (0.5)	64	Fair cure
86-3	NTPB (1.0) MAM (0.8)	64	Poor cure
86-4	NTPB (1.0) HMAT (0.8)	64	Poor cure

TABLE XXXII

(U) BINDER CURES WITH TMETN PLASTICIZED HX-735
(HX-735 Polymer Equivalents = 1.0)

Formulation	Curative and Equivalents	Cure Time Hr	Results
182-78-1	MAPO (1.0) NTEB (0.2)	168	Poor cure
78-2	MAPO (1.0) NTEB (0.4)	168	Poor cure
78-3	MAPO (1.0) NTEB (0.6)	168	Poor cure
78-4	MAPO (1.0) NTEB (0.2)	168	Poor cure
182-79-1	MAPO (1.0) NTEB (0.2)	168	No cure
79-2	MAPO (1.0) NTEB (0.4)	168	Poor cure
79-3	MAPO (1.0) NTEB (0.4)	168	Poor cure
79-4	MAPO (1.0) NTEB (0.4)	168	Fair cure
182-80-1	MAPO (1.0) MAM (0.2)	168	Poor cure
80-2	MAPO (1.0) MAM (0.4)	168	Poor cure
80-3	MAPO (1.0) MAM (0.6)	168	Poor cure
80-4	MAPO (1.0) MAM (0.8)	168	Poor cure
182-81-1	MAPO (1.0) MAES (0.2)	168	No cure
81-2	MAPO (1.0) MAES (0.4)	168	No cure
81-3	MAPO (1.0) MAES (0.4)	168	No cure
81-4	MAPO (1.0) MAES (0.4)	168	No cure
182-85-1	NTEB (1.0) MAM (0.5)	64	Poor cure
85-2	NTEB (1.0) HMAT (0.5)	64	Poor cure
85-3	NTEB (1.0) MAM (0.8)	64	Poor cure
85-4	NTEB (1.0) HMAT (0.8)	64	Poor cure
182-87-1	NTPB (1.0) MAM (0.5)	64	Poor cure
87-2	NTPB (1.0) HMAT (0.5)	64	Poor cure
87-3	NTPB (1.0) MAM (0.8)	64	Very poor cure
87-4	NTPB (1.0) HMAT (0.8)	64	Very poor cure
182-89-1	NTEB (1.2)	64	Very poor cure
89-2	NTEB (1.5)	64	Very poor cure
89-3	NTEB (1.8)	64	Very poor cure
89-4	NTEB (2.1)	64	Very poor cure

TABLE XXXII

(U) BINDER CURES WITH TMETN PLASTICIZED HX-735
(HX-735 Polymer Equivalents = 1.0) (Continued)

Formulation	Curative and Equivalents	Cure Time Hr	Results
182-91-1	NTPB (1.2)	64	Very good cure
91-2	NTPB (1.5)	64	Poor cure
91-3	NTPB (1.8)	64	No cure
91-4	NTPB (2.1)	64	No cure
182-93-1	NTEB (0.6) NTPB (0.6)	64	Good cure
93-2	NTEB (0.75) NTPB (0.75)	64	Fair cure
93-3	NTEB (0.90) NTPB (0.90)	64	Poor cure
93-4	NTEB (1.05) NTPB (1.05)	64	Poor cure
182-95-1	NTEB (0.3) NTPB (0.9)	64	Good cure
95-2	NTEB (0.9) NTPB (0.3)	64	Fair cure
95-3	NTEB (0.45) NTPB (1.35)	64	Poor cure
95-4	NTEB (1.35) NTPB (0.45)	64	Poor cure
182-97-1	HMAT (1.75)	64	Very poor cure
97-2	HMAT (1.50)	64	Very poor cure
97-3	HMAT (1.50) MAPO (0.25)	64	Very poor cure
97-4	HMAT (1.50) Epon 812 (0.25)	64	Very poor cure
182-106-1	HX-868 (1.2)	168	No cure
106-2	HX-868 (1.4)	168	No cure
106-3	HX-868 (1.6)	168	No cure
182-107-1	HX-858 (1.2)	168	No cure
107-2	HX-858 (1.4)	168	No cure
107-3	HX-858 (1.6)	168	No cure
182-108-1	HX-874 (1.2)	144	Very good cure
108-2	HX-874 (1.4)	144	Very good cure
108-3	HX-874 (1.6)	144	Very good cure

TABLE XXXIII

(U) STRESS-STRAIN PROPERTIES OF UNFILLED HX-735 BINDERS

	Formulation No.						
	182-90-1	182-91-1	182-92-3	182-83-1	182-95-1	182-78-4	182-79-4
Equivalents							
HX-735	1.0	1.0	1.0	1.0	1.0	1.0	1.0
NTPB	1.2	1.2	0.9	0.6	0.9	---	0.8
NTEB	---	---	0.9	0.6	0.3	0.8	---
MAPO	---	---	---	---	---	1.0	1.0
Wt % (of binder)							
TMETN	---	55	---	55	55	55	55
Stress, psi							
5° F	102	24	62	---	28	---	8
40° F	---	18	---	14	---	---	---
72° F	45	18	---	14	---	---	6
120° F	---	---	10	14	11	---	---
Strain, %							
5° F	56	127	176	---	23	---	236
40° F	---	72	---	72	---	---	---
72° F	209	61	---	54	---	---	185
120° F	---	---	21	45	73	---	---

* Did not cure

(U) Absent from the quantitative evaluation were cure tests in which TBM and HX-874 were used. Qualitatively, these curatives appeared promising; but because of the late introduction into the program, no ring tests were made. The TBM cured binder properties were found to be similar to those found with HX-874.

(U) The most promising binder systems were selected from the NTPB, NTEB, MAPO binder tests for further tests in propellant formulations. These formulations contained aluminum and AP as the solid phase. The binder was HX-735 polymer, TMETN, and the curative.

(U) Table XXXIV presents mechanical property data obtained from propellants processed with these curatives. The only propellant is this series that appeared of value was the control, UTX 7902. It was this cure system that had been used previously, and although mechanical properties were acceptable it had exhibited poor stability in elevated temperature storage.

(U) In view of these findings, tests continued with UTX 7902 propellant but with calcium hydroxide as a cure catalyst. Although the stress values increased with the addition of calcium hydroxide, the strain values decreased. A castability problem also was encountered when calcium hydroxide was used. The data are presented in table XXXV and show that the control formulation, UTX 7902, had the highest strain value. However, 0.05 wt-% calcium hydroxide did improve the stress properties with only slight decrease of the strain value, and the addition of calcium hydroxide could be of some value in this system.

(U) Another approach to improving the cured mechanical properties of the UTX 7902 propellant was the addition of NTPB to the MAPO-Epon cure system. This approach was the result of quantitative binder tests presented in table XXXVI which indicated improved mechanical properties when NTPB was the only curative used. Table XXXVII presents the data obtained on these propellants. The stress values were increased to a greater degree than with the use of calcium hydroxide. The propellant also was more easily processed.

TABLE XXXIV

(U) PROPELLANT EVALUATION OF PEP 155 BINDERS
(Cure 168 Hours at 120° F)

Formulation	Binder (Equivalents)		Stress	Stress	Remarks
	HX-735	Curative	psi	%	
UTX 7913	1.0	NTPB (1.1)	---	---	Insufficient cure
UTX 7900	1.0	NTPB (1.2)	11	65	
UTX 7910	1.0	NTPB (1.3)	26	33	Reversion evident
UTX 7911	1.0	NTPB (1.4)	28	26	Reversion evident
UTX 7914	1.0	NTPB (1.5)	24	29	Reversion evident
UTX 7915	1.0	NTEB (1.1)	---		No cure
UTX 7916	1.0	NTEP (1.2)	---		No cure
UTX 7917	1.0	NTEB (1.3)	---		No cure
UTX 7918	1.0	NTEB (1.4)	---		No cure
UTX 7919	1.0	NTEB (1.5)	---		No cure
UTX 7912	1.0	NTPB (0.825) NTEB (0.275)	---	---	Reversion (11 days at 120° F)
UTX 7901	1.0	NTPB (0.90 NTEB (0.30)	---	---	Reversion prior to test
UTX 7907	1.0	NTPB (0.975) NTEP (0.935)	---	---	Too soft to test
UTX 7908	1.0	NTPB (1.05) NTEB (0.35)	---	---	Reversion prior to test
UTX 7909	1.0	NTPB (1.125) NTEB (0.375)	24	40	---
UTX 7920	1.0	NTPB (0.825) Epon 812 (0.275)	---	---	Too soft to test
UTX 7921	1.0	NTPB (0.90) Epon 812 (0.30)	---	---	Too soft to test
UTX 7922	1.0	NTPB (0.975) Epon 812 (0.325)	31	30	---
UTX 7923	1.0	NTPB (1.05) Epon 812 (0.35)	44	28	---
UTX 7924	1.0	NTPB (1.125) Epon 812 (0.375)	---	---	No cure
UTX 7902	1.0	MAPO 1.5 Epon 812 (0.7)	62	25	---

Formulation

	Wt %
PEP 155 binder	15.9
Aluminum	16.0
NH_4ClO_4	68.0
Ethyl Centralite	0.1
	100.0

TABLE XXXV

(U)EFFECT OF CALCIUM HYDROXIDE ON THE CURE OF PEP 150 PROPELLANTS

Formulation UTX	Curative and Equivalents	Stress at Maximum psi	Strain at Maximum %
7902 (Control	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

TABLE XXXVI

(U) EFFECT OF NTPB ON THE CURE OF PEP 150 PROPELLANTS

Formulation UTX	Curative and Equivalents	Stress at Maximum psi	Strain at Maximum %
7902 (Control)	MAPO (1.5 Epon 812 (0.7)	62	25
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

TABLE XXXVII

(U) EFFECT OF HX-874 ON THE CURE OF PEP 150 PROPELLANTS

Formulation UTX	Curative and Equivalents	Stress at Maximum psi	Strain at Maximum %
7934	HX-874 (1.4)	41	8
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

(U) Two curatives that were not evaluated quantitatively in the binder ring tests were tested in propellant formulations. These were a 3M Company proprietary aziridine, HX-874, and an American Cyanamid trifunctional aziridine, TBM. These two curatives were assumed to have similar chemical structures. The initial tests with HX-874 and TBM did not show results that were indicative of any similarity of the two curatives. Comparison of tables XXXVII and XXXVIII show the data obtained on propellants cured with HX-874 and TBM. Stress values were similar, but the strain values were lower when the propellant was cured with HX-874. At the time of these tests it was assumed that TBM was the preferred curative because the strain values were higher. As a result, greater effort was expended on testing formulations that used TBM as the principal curative.

(U) The most promising curative mixture in which TBM was used was UTX-7592 (see table XXXVIII). This curative contained Epon 812 and MAPO blended with TBM. In these tests the addition of MAPO to the curative blend improved the strain values. However, there were changes in the stress values as the MAPO loading was changed in the tri-curative blend.

TABLE XXXVIII

(U) EFFECT OF TBM AND MIXTURES UPON MECHANICAL PROPERTIES OF PEP 150 PROPELLANT

Formulation	Curative and Equivalents	Stress at Maximum psi	Strain at Maximum %
UTX 7577	TBM (1.3)	46	13
UTX 7578	TBM (1.25) Epon 812 (0.7)	90	16
UTX 7579	TMB (1.0) Epon 812 (0.55)	67	26
UTX 7580	TBM (1.25) HX-874 (0.7)	77	8
UTX 7581	TBM (1.0) HX-874 (0.55)	61	10
UTX 7584	TBM (1.25) NTPB (0.70)	61	11
UTX 7585	TBM (1.0) NTPB (0.55)	44	15
UTX 7586	TBM (1.25) UNOX 201 (0.70)	34	17
UTX 7587	TBM (1.00) UNOX 201 (0.55)	18	34
UTX 7588	TBM (0.50) MAPO (0.70) Epon 812 (0.7)	58	46
UTX 7589	TBM (0.60) MAPO (0.60) Epon 812 (0.70)	74	42
UTX 7590	TBM (0.40) MAPO (0.80) Epon 812 (0.70)	43	51

TABLE XXXVIII

(U) EFFECT OF TBM AND MIXTURES UPON MECHANICAL PROPERTIES OF PEP 150 PROPELLANT (Continued)

Formulation	Curative and Equivalents	Stress at Maximum psi	Strain at Maximum %
UTX 7591	TBM (0.3) MAPO (0.9) Epon 812 (0.7)	47	52
UTX 7592	TBM (0.7) MAPO (0.5) Epon 812 (0.7)	83	41
UTX 7593	TBM (0.6) MAPO (0.6) Epon 812 (0.7)	49	45
UTX 7594	TBM (0.8) MAPO (0.4) Epon 812 (0.7)	76	32
UTX 7595	TBM (0.9) MAPO (0.3) Epon 812 (0.7)	78	31
UTX 7596	TBM (0.5) MAPO (1.0) Epon 812 (0.7)	77	37
UTX 7597	TBM (1.5) Epon 812 (0.5)	109	13
UTX 7598	TBM (1.75) Epon 812 (0.25)	112	11
UTX 7599	TBM (1.25) Epon 812 (0.75)	98	14
UTX 8200	TBM (1.50) Epon 812 (0.75)	119	11
UTX 8201	TBM (1.60) Epon 812 (0.75)	114	12

(U) Following the initial series of tests with both TBM and HX-874 it was found that storage conditions of the curative had a profound effect on its stability. Each of the vendors confirmed this. To illustrate the change in purity that does occur, the following information was obtained on two separate samples stored under various conditions:

Time	Purity, Sample A		Purity, Sample B	
Days	45° F	75-85° F	15° F	75-85° F
0	92.5%	92.5%	92.7%	92.7%
4	92.6%	91.6%	---	---
11	---	---	92.4%	88.0%
20	90.2%	80.2%	---	---
21	---	---	93.2%	74.6%

(U) As a result of these storage tests, the differences that were found in the cured mechanical properties between TBM and HX-874 cures could be explained because the storage history of the two materials could not be assured to be the same.

(U) New closely controlled lots of HX-874 and TBM were procured from the respective vendors. Shipment and storage upon arrival were similar. A comparative evaluation was made. Table XXXIX shows the excellent similarity of the cured mechanical properties.

(U) The tests made with available crosslinking agents indicated that the improvement of propellant mechanical properties could be achieved with the greatest probability by the use of either TBM or HX-874 as the primary crosslinker.

(U) Concurrent with the evaluation of potential curative systems in aluminized propellant formulations, the Advanced Formulations Group conducted an evaluation of imine curatives in LMH-1 formulations. This work was guided by preliminary results in the aluminized system and was designed to reveal potential problems that might be unique to the LMH-1.

TABLE XXXIX

(U) COMPARISON OF TBM AND HX-874 IN A UTEFLEX PROPELLANT

Test Temperature °F	UTX-8640 (TBM)		UTX-8461 (HX-874)	
	Stress psi	Strain %	Stress psi	Strain %
130	32	33	33	31
76	51	41	55	38
0	200	32	229	31

Formulation

	Equivalent	Wt %
Uteflex	1.0	8.80
TBM or HX-874	1.3	
Epon 812	0.5	
TMETN		8.80
NH_4ClO_4		66.10
Aluminum		16.00
Ethyl Centralite		0.30

(U) The following imines were evaluated as curing agents in an LMH-1 system: tris [1-(2-methyl) aziridinyl] phosphate oxide, (MAPO); diethyleneglycol bis [bis (1-aziridinyl phosphate], (DGAP); nitrilotriethyl-β-ethyleniminobutyrate, (NTEB) (failed to cure this system), and nitrilotriethyl-β-propyleniminobutyrate, (NTPB).

(C) The control formulation, UTX-6626, contained 32 vol-% binder and the same wt-% aluminum as the standard propellant, UTX-6814. Table XL is a list of the formulations and results obtained varying the cure system. These formulations were all with the PEP-155 binder system with 22.5% aluminum, 55.6% AP, and 0.5 equivalents of Epon 812.

(U) The MAPO cured system optimized at 1.55 to 1.60 equivalents of MAPO. Nitrilotriethyl-β-propylenimonobutyrate appeared to have potential as a curative for the PEP system.

(U) The data indicated that MAPO has a longer induction period and a slower rate of cure than either DGAP or NTPB. Nitrilotriethyl-β-propylenimonobutyrate at 1.2 equivalence cured completely within 24 hr at 120° F. This indicated a lower cure temperature was feasible with this system. The pot life of the NTPB appeared to be greater than 2 hr from the studies at 120° F.

(U) Another area of curative investigation was the evaluation of tris-(2-ethylaziridinyl)-s-triazine (TBM). Table XLI presents mechanical property data from the first series of experimental mixes. It was apparent that the problem of increasing the elongation could not be solved by simple substitution of TBM for MAPO.

(U) Formulations UTX 6840-1 and -6841 in table XLII using 0.7 and 0.9 equivalents TBM showed that higher TBM equivalents resulted in higher tensile values. Formulations were then made holding the TBM and Epon 812 equivalents constant at 0.8 and 0.5, respectively, while the MAPO equivalents were varied (0.6, 0.7, and 0.8 equivalents) to obtain increased strain values. The experimental data are summarized in table XLII. On the basis of the three formulations, UTX 6871 yielded the best mechanical properties (σ_m = 50 psi, ϵ_m = 37.8% crosshead).

TABLE XL

(U) EFFECT OF CURATIVE ON PEP PHYSICAL PROPERTIES

Formulation	Curative	Curative Equivalent	Shore A 24 hr	Shore A 48 hr	Shore A 72 hr	Shore A 96 hr	S_m psi	E_m %	Remarks
UTX 7321	MAPO	1.40	0	0	0	0	68.8	6.9	Shore A = 25.0 at 144 hr
UTX 7322-1	MAPO	1.50	0	20.0	21.0	33.0	63.1	6.5	Shore A = 45.0 at 142.5 hr
UTX 7323-1	MAPO	1.60	0	10.0	38.0	46.0	100.9	6.2	Shore A = 48.0 at 138.5 hr
UTX 7324-1	MAPO	1.70	0	14.0	36.0	46.0	97.4	4.9	Shore A = 60.0 at 151.5 hr
UTX 7325-1	MAPO	1.80	0	17.0	43.0	55.0	79.2	2.6	Shore A = 63.0 at 142.5 hr
UTX 7326-1	DGAP	1.40	8.0	25.0	31.0	36.0	38.7	10.1	Shore A = 40.0 at 116.0 hr
UTX 7327-1	DGAP	1.50	10.0	28.0	35.0	40.0	45.1	8.6	Shore A = 42.0 at 115.0 hr
UTX 7328-1	DGAP	1.60	28.0	40.0	42.0	43.0	64.5	7.7	Shore A = 43.0 at 114 hr
UTX 7332	NTPB	1.00	28.0	35.0	37.0	37.5	52.2	29.2	Shore A = final at 92.0 hr
UTX 7331	NTPB	1.10	35.0	40.0	41.0	42.0	55.1	19.6	Shore A = final at 94.5 hr
UTX 7333	NTPB	1.20	45.0	45.0	45.0	45.0	63.6	19.8	Shore A = final at 90.5 hr

TABLE XLI

(U) HX-735 – TBM MECHANICAL PROPERTY DATA

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 6827-1	TBM curative 1.0 HX-735/1.0 TBM/ 0.7 Epon 812	38.0	30.0	44.0	19.2	3	40	1.472	92.7
UTX 6829-1	TBM curative 1.0 HX-735/1.5 TBM/ 0.7 Epon 812	88.0	14.8	95.0	8.4	3	72	1.524	96.0
UTX 6641-1	TBM curative, Al 1.0 HX-735/1.5 TBM/ 0.7 Epon 812	92.0	13.8	100.0	8.6	3	79	1.781	96.1
UTX 6834-1	TBM curative All Purified ingredients 1.0 HX-735/0.9 TBM/ 0.6 MAPO/0.5 Epon 812	97.5	11.8	104	6.8	4	80	1.546	94.4

TABLE XLII

(U) EFFECT OF HX-723 + TBM AND MIXTURES ON MECHANICAL PROPERTIES

Formulation	HX-735	TBM	MAPO	Epon 812	Crosshead S_m psi	Crosshead E_m %	Shore A Hardness	Density g/cc	% Theoretical Density
UTX 6840-1	1.0	0.9	0.6	0.5	97	10.4	85	1.60	97.6
UTX 6841-1	1.0	0.7	0.5	0.5	61	19.4	75	1.55	94.5
UTX 6870-1	1.0	0.8	0.6	0.5	59	28.8	45	1.56	98.1
UTX 6871-1	1.0	0.6	0.7	0.5	50	37.8	---	1.55	97.5
UTX 6872-1	1.0	0.8	0.8	0.5	63	25.5	---	1.52	95.6

(2) Evaluation of Candidate Polymers

(U) One approach to the improvement of propellant mechanical properties involves the use of other polymers. The compatibility of candidate polymers with other propellant ingredients limits the type of polymers available.

(U) At the outset of the program the only polymer that was known to be compatible was HX-735, a neopentyl glycol/azelaic acid polyester with terminal carboxyl groups. This polymer had a molecular weight of approximately 1,700. An effort was made to obtain a similar polymer but with a higher molecular weight. Both the 3M Company and Emery Industries were contacted and each supplied higher molecular weight polymers.

(U) Two polymers were supplied by the 3M Company. These were identified as L-1806 and L-1807. The former had a molecular weight of 2,200 and the latter was 2,300. Each was tested in propellant formulations. The binder ratio and resulting mechanical properties are presented in table XLIII.

TABLE XLIII

(U) EVALUATION OF HIGH MOLECULAR WEIGHT 3M COMPANY POLYMERS

Formulation UTX	Equivalent	Equivalent	Equivalent	Equivalent	Equivalent	Stress psi	Strain %
8608	1.0	---	---	1.5	0.7	97	16
8610	1.0	---	---	1.3	0.5	82	19
8607	---	1.0	---	1.5	0.7	83	19
8611	---	1.0	---	1.3	0.5	60	29
3260	---	---	1.0	1.5	0.7	89	15
8262	---	---	1.0	1.3	0.5	61	31

(U) Emery Industries provided two polymers. These were also neopentyl glycol polyesters with carboxylic groups. The first of these was identified as Emery 1025-9-R and had a molecular weight of 1,900. The second was Emery 1025-94-R which had a molecular weight of 2,950. Table XLIV presents a summary of the formulations tested and the resulting mechanical properties. Each of the polymers appeared to show improvement in the mechanical properties.

TABLE XLIV

(U) EVALUATION OF HIGH MOLECULAR WEIGHT

EMERY INDUSTRIES

Formulation UTX	Binder Materials and Equivalents	Stress at Maximum psi	Strain at Maximum %
8226	Emery 1025-9-4 (1.0) TBM (1.5) Epon 812 (0.7)	91	19
8227	Emery 1025-94-R (1.0) TBM (1.5) Epon 812 (0.7)	66	32

(U) The improved elongation of the high molecular weight polymer (Emergy 1025-94) and the good tensile strength of the low molecular weight polymer (Emery 1025-9-R) were believed to offer a method of mechanical property improvement by blending the two polymers. Propellant mixes were made to evaluate the effect of blending the two Emery polymers on the resulting propellant mechanical properties. The data are presented in table XLV. There was no apparent correlation in association with the blends and the resulting cured properties. It was concluded that the best properties were obtained by use of the high molecular weight polymer, Emery 1025-94 unblended.

(U) As a result of the studies made to evaluate the effect of ingredient purification on propellant density under task 5 (see d., (1)), it was observed that treatment of the polymer

TABLE XLV

(U) EFFECT OF POLYMER MOLECULAR WEIGHT ON PROPELLANT CURED PROPERTIES

Estimated Molecular Weight	1025-9-R Equivalent	1025-94-R Equivalent	TBM Equivalent	Epon 812 Equivalent	MAPO Equivalent	Stress* psi	Strain* %
1,900	1.0	---	1.5	0.7	---	91	19
2,226	0.8	0.2	1.5	0.7	---	120	11
2,239	0.7	0.3	1.5	0.7	---	110	13
2,352	0.6	0.4	1.5	0.7	---	103	14
2,465	0.5	0.5	1.5	0.7	---	103	16
2,578	0.4	0.6	1.5	0.7	---	78	20
2,691	0.3	0.7	1.5	0.7	---	71	20
2,804	0.2	0.8	1.5	0.7	---	89	17
3,030	---	1.0	1.5	0.7	---	66	32
1,900	1.0	---	0.9	0.7	0.6	48	47
3,030	---	1.0	0.9	0.7	0.6	No cure	---

* Crosshead data

in a molecular still had an effect on propellant mechanical properties. Therefore, the effect of the various purification techniques for the HX-735 on physical properties of LMH-1 propellant was examined. JANAF specimens were prepared from different lots of the purified polymer. A summary of the effort of molecular still stripping on HX-735 equivalent weight is shown in table XLVI. Tables XLVII and XLVIII present the JANAF data for the mechanical properties specimens.

(U) An increase in tensile and Shore A hardness was obtained with the higher purity polymer. Some of this increase was a result of the improved density of the propellant.

(U) Azelaic acid is the major impurity in the as-received HX-735 polymer. The effect of adding back the azelaic acid to the stripped polymer is shown in mix UTX 6832-2. A decrease in density was obtained indicating an interaction between LMH-1 and the azelaic acid.

(U) All previous work on the PEP propellant used lot-2 HX-735. Another lot of HX-735, lot 3, was evaluated in UTX 6814-148. A lower tensile and higher elongation was obtained with this information. Subsequent tests indicated that a fast flow rate during the molecular distillation procedure resulted in poor stripping of the polymer. The poor density and physical properties were caused by the poor stripping action. This work supported the view that the azelaic acid was reacting with LMH-1 and was one of the main causes for low density propellant prepared from as-received HX-735.

(U) The UTC Organic Chemistry Group supplied a polymer for evaluation. This was a polyether with terminal carboxyl groups which was specifically designed to accept large concentrations of nitrato-plasticizer. It was identified as Uteflex 170-156. Listed below is a comparison of various properties of Uteflex and HX-735:

	Uteflex	HX-735
M_n, g/mole	1,935	1,765
E_n, g/equiv	955	872
Functionality	2.0	2.0
Viscosity, 24° C poises	17.0	223
Gel Time, hr	1.6	2.8
Density, g/cc	1.09	1.06

TABLE XLVI

(U) 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

TABLE XLVII

(U) EFFECT OF PROCESSING VARIABLES ON PROPELLANT DENSITY

Formulation UTX-6814	Variable	Shore A	Cure Temperature Days	Density g/cc	% Theoretical Density
112	All ingredients purified (control)	45	3	1.52	96.5
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
115B	Vibration under vacuum, purified ingredients	48	3	1.62	102

TABLE XLVIII

(U) MECHANICAL PROPERTY DATA

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 6814-148	Lot No. HX-735, all purified ingredients	41.5	25.0	48.0	15.8	4	49	1.461	92.0
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

(U) A series of Uteflex polymers with various molecular weights were prepared and evaluated. Table XLIX presents a summary of the cured mechanical property data. These data show that good propellant properties were obtained on all Uteflex polymers. However, it was believed that the high molecular weight polymer, Uteflex 207-77, offered the best combination of properties. A short study was then initiated further to evaluate these polymers with AlH_3.

(U) Uteflex + TBM mixes were prepared using 23 vol-% binder. However, these formulations (UTX-6851-1, 6852-1, 6868-1) were viscous and difficult to cast. A short pot life was observed. Because of the low measured densities caused by casting voids, the mechanical property data presented in table L are questionable.

(U) The effect of TBM was investigated in formulations UTX 6859-1 and 6860-1. A summary of the mechanical properties is presented in table LI. With increasing equivalents of TBM, the tensile increased and the elongation decreased.

(U) An elongation greater than 20% could not be achieved without a poor tensile value and so the use of a mixed curative system (TMB-MAPO) was investigated. In the formulations UTX 6868-1 and 6862-1, the total triazine equivalents were the same as used in the UTX 6859-1 and UTX 6860 mixes. A minor increase in tensile and a drop in elongation was noted with increasing TBM content.

(U) To increase the tensile values of the above mixes, formulations UTX 6867 and 6868 were mixed with higher TBM equivalents — 1.3 and 1.5. Table LII presents the experimental data. A slight gain in tensile was obtained but the value was below the desired value.

(U) Mixes 6863-1 and 6864 were prepared to determine the effect of MAPO concentration on physical properties. The curative equivalents are presented in table LII. No cure was obtained with either mix indicating that MAPO would not cure Uteflex alone or that higher equivalents were necessary to obtain propellant cures.

TABLE XLIX

(C) EVALUATION OF UTEFLEX TYPE PROPELLANTS

Formulation UTX	Uteflex Type	Molecular Weight	Propellant Cured Properties			
			Stress (psi)		Strain (%)	
			Crosshead	Measured	Crosshead	Measured
8289	207-70	2,014	91	---	23	---
8292	209-104	1,900	154	169	15	10
8294	207-72	1,920	150	168	21	16
8295	207-73	2,460	89	107	27	20
8297	207-78	2,980	75	96	40	28
8298	207-77	3,150	53	74	53	41

Binder Composition

	Equivalent	Wt %
Uteflex	1.0	
TBM	1.5	50.0
Epon 812	0.7	
TMETN		50.0

TABLE L

(U) EFFECT OF VARIOUS CURATIVES ON UTEFLEX MECHANICAL PROPERTIES

						Mechanical Properties Crosshead				%
Formulation	PEP System	Uteflex	TBM	MAPO	Epon 812	S_m, psi	E_m, %	Shore A Hardness	Density g/cc	Theoretical Density
UTX 6851-1	150	1.0	1.4	---	0.5	55.4	14.6	75	1.51	92.1
UTX 6852-1	150	1.0	1.6	---	0.5	34.2	18.2	60	1.42 (large voids)	86.6
UTX 6858-1	160	1.0	1.1	0.5	0.5	30.4	18.0	45	1.42 (large voids)	86.1

TABLE LI

(U) EFFECT OF TBM CONCENTRATION ON UTEFLEX MECHANICAL PROPERTIES

						Mechanical Properties Crosshead				%
Formulation	PEP System	Uteflex	TBM	MAPO	Epon 812	S_m, psi	E_m, %	Shore A Hardness	Density g/cc	Theoretical Density
UTX 6859-1	155	1.0	1.4	---	0.5	27.0	50.4	42	1.37	86.2
UTX 6860-1	155	1.0	1.6	---	0.5	72.6	14.8	50	1.48	93.1

TABLE LII

(U) EFFECT OF MIXED CURATIVE CONCENTRATION ON UTEFLEX MECHANICAL PROPERTIES

						Mechanical Properties Crosshead				%
Formulation	PEP System	Uteflex	TBM	MAPO	Epon 812	S_m, psi	E_m, %	Shore A Hardness	Density g/cc	Theoretical Density
UTX 6861	155	1.0	0.9	0.5	0.5	37.9	52.7	48	1.40 (large voids)	88.1
UTX 6862-1	155	1.0	1.1	0.5	0.5	38.8	40.0	50	1.42	89.3
UTX 6867-1	155	1.0	1.3	0.5	0.5	43	33.6	38	1.45	91.2
UTX 6868-1	155	1.0	1.5	0.5	0.5	42	41.0	48	1.45	91.2
UTX 6863-1	155	1.0	---	1.4	0.5	←No cure→		---	---	---
UTX 6864-1	155	1.0	---	1.6	0.5	←No cure→		---	---	---

(U) The other new polymer evaluated in the PEP system was Emery 1023-94-R, a neopentyl-glycol azelaic acid polyester. This polymer has the same structure as HX-735. A mix was prepared using the following equivalents:

Emery 1025-94-R	1.0
TBM	1.5
Epon 812	0.7

The propellant had low crosshead mechanical properties, $S_m = 53$ psi and $E_m = 19.8\%$.

(3) Effect of TMETN on Mechanical Properties

(C) 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 LIII and in figure 38. It is apparent that propellant stress values are quite sensitive to plasticizer level, whereas the increase in strain capability with increasing plasticizer 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 a modest performance increase.

(4) Evaluation of Stabilizers

(U) Prior to the introduction of the new polymer-curative system (Uteflex/HX-874/Epon 812) three binder systems had been selected for accelerated aging tests. The primary objective was to determine the type of curative system that would provide the greatest stability with the HX-735 polymer.

(U) Propellant blocks representing three formulations were stored at 140° F. Specimens were machined and tested weekly for 11 weeks. The formulations and test data are presented in figure 39 and table LIV. Considerable data scatter makes the results difficult to interpret. However, it appears that the UTX 7700 formulation (TBM/MAPO/Epon 812 curative system) is the most stable in terms of minimizing fluctuations in propellant stress capabilities. However, high stress values were maintained by the UTX 7702 formulation (MAPO/NTPB/Epon 812) which also had the highest total curative concentration.

TABLE LIII

(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		1.0

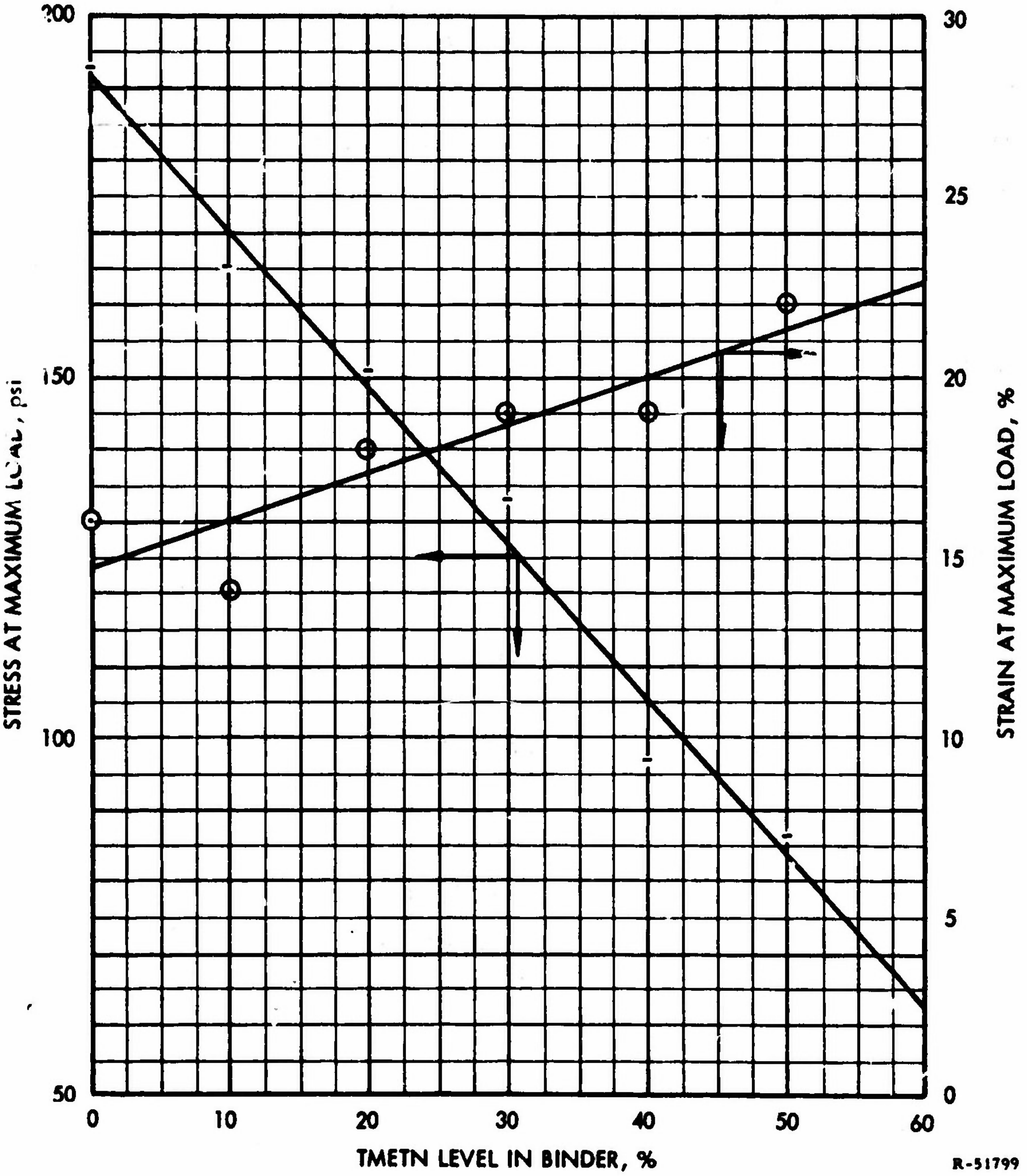

Figure 38. (U) Effect of Plasticizer Level on Propellant Mechanical Properties

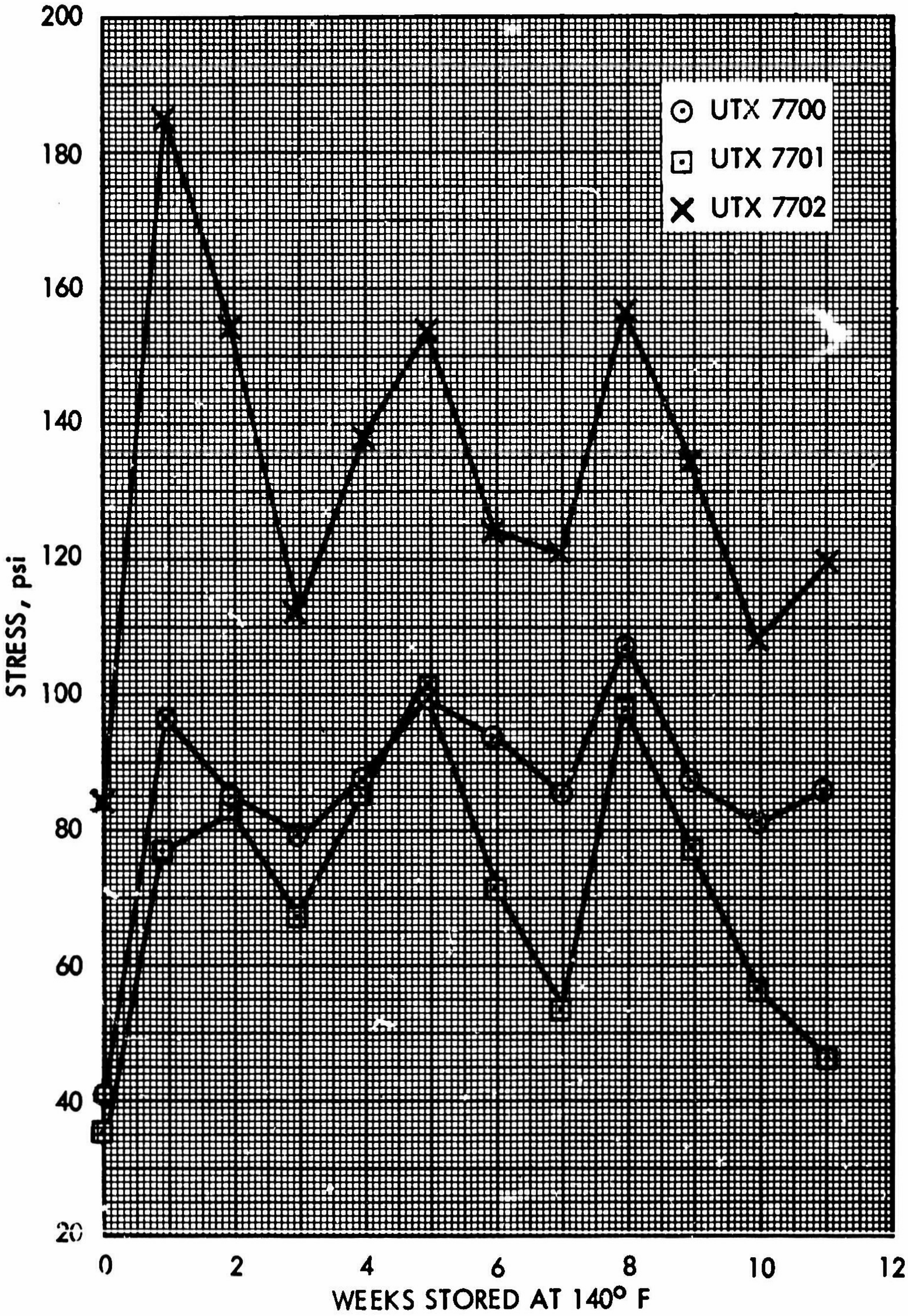

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Figure 39. (U) Effect of High Temperature Storage on Stress Properties of Propellant (HX-735 Polymer System)

TABLE LIV

(U) ACCELERATED AGING TESTS OF HX-735 TYPE PROPELLANT

	UTX-7700		UTX-7701		UTX-7702	
Formulation	**Equivalent**	**wt-%**	**Equivalent**	**wt-%**	**Equivalent**	**wt-%**
HX-735	1.0	8.80	1.0	8.80	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	---	8.80	---	8.80
Al H-322	---	16.00	---	16.00	---	16.00
NH_4ClO_4 (MS-4)	---	45.78	---	45.78	---	45.78
NH_4ClO_4 (ground)	---	19.62	---	19.62	---	19.62
Ethyl centralite	---	1.00	---	1.00	---	1.00

Storage Time Weeks	Stress psi	Strain %	Stress psi	Strain %	Stress psi	Strain %
0	41	37	36	31	[illegible]5	17
1	97	18	78	22	186	14
2	86	15	84	21	155	14
3	80	16	67	22	113	13
4	88	16	87	22	139	15
5	100	18	102	24	155	16
6	95	16	72	24	125	15
7	86	15	54	25	122	16
8	108	17	99	24	158	17
9	88	15	78	26	135	18
10	82	17	57	28	109	18
11	86	14	47	30	120	18

(U) In general, all of these formulation displayed a stability of mechanical properties which represented a substantial improvement over those obtained under the previous program, AF 04(611)-9570, with essentially the same formulation as UTX 7701. The major difference between UTX 7701 and earlier formulations is the concentration of ethyl centralite.

(U) To determine the effect of ethyl centralite on propellant aging properties, the UTX 7701 propellant was evaluated with ethyl centralite concentrations of 0.1%, 0.5%, and 1.0%. The propellant was cured 5 days at 140° F and stored at 160° F. It was anticipated that this propellant which had a history of instability would show early signs of degradation when stored at 160° F, and from this, the effect of ethyl centralite could be determined. The formulations and data are presented in table LV, and the stress values are shown as a function of storage time in figure 40.

TABLE LV

(U) EFFECT OF ETHYL CENTRALITE ON PROPELLANT AGING STABILITY

Formulation	UTX 7701		UTX 7708		UTX 7709	
Ethyl Centralite wt-%	1.00		0.50		0.10	
Storage Time Weeks	Stress psi	Strain %	Stress psi	Strain %	Stress psi	Strain %
0	67	39	76	26	93	29
1	98	29	115	20	130	19
2	75	34	115	22	133	21
3	85	33	75	25	128	24
4	70	36	90	23	102	23
5	54	35	64	22	75	24
6	48	37	70	25	52	29
7	50	22	59	24	73	18
8	27	42	---	---	---	---
9	53	22	40	24	47	26

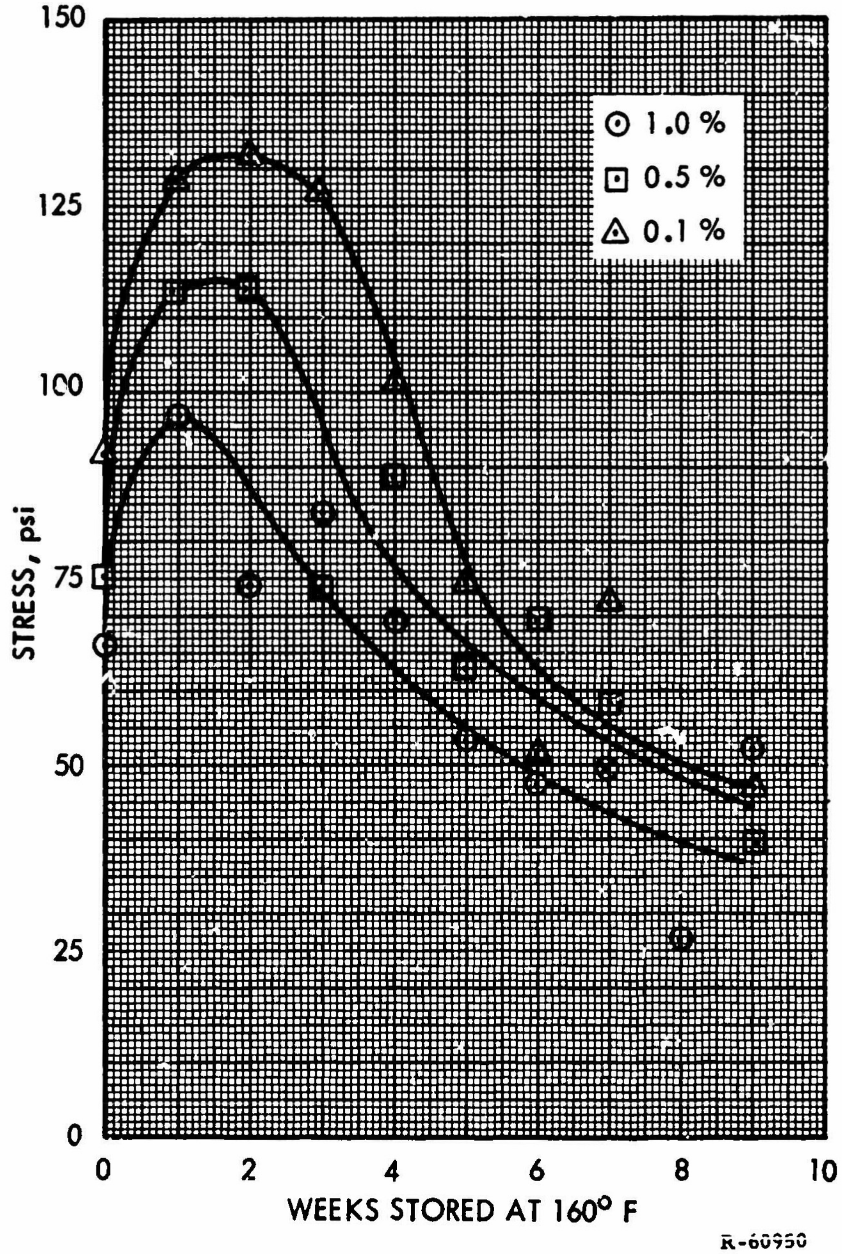

Figure 40. (U) Effect of High Temperature Storage on Stress Properties of Propellant as a Function of Ethyl Centralite Concentration

(U) From the stress values obtained there was no obvious correlation between long-term high-temperature stability and ethyl centralite concentration. However, the ethyl centralite did have a significant effect on the extent of postcure experienced during the first 3 weeks of storage. Considerably less postcure occurred at the higher ethyl centralite concentrations. This is consistent with subsequent observations in which the ethyl centralite was found to reduce the rate of propellant cure, apparently in direct relationship to the concentration of the stabilizer present.

(U) It was noted also that test samples from UTX 7708 and UTX 7709 showed evidence of crack formation and a decided lack of elastomeric qualities after 7 weeks of storage. It was concluded from these tests that 1.00 wt-% ethyl centralite was desirable for stability in a MAPO/Epon 812 curative system.

(U) After the selection of the polymer and curative system for the final propellant formulation (Uteflex/HX-874/Epon 812) it was noted that the processing characteristics of the propellant and the propellant cure rate were profoundly influenced by the stabilizer. Ethyl centralite increases the propellant pot life. Pot life had been a critical processing problem because of the extremely reactive nature of the azirdinyl curative. However, if excessive amounts of ethyl centralite are used, the required cure time is extended to an unacceptable length.

(U) A series of tests was conducted in an aluminized formulation to optimize the curative ratio at a constant 0.5 wt-% ethyl centralite. Table LVI contains the formulations and measured stress-strain properties after a 5-day cure at 120° F. The dogbone ends remaining from the tensile tests were stored at 160° F, and Shore A hardness measurements were made weekly for 11 weeks. These results are presented graphically in figure 41. Primarily as a result of these tests, the curative ratio for the final propellant formulation was selected. Formulation UTX 6565, had the following equivalent ratios and measured tensile properties:

Uteflex	1.0
HX-874	1.4
Epon 812	0.6

σ_m = 61.2 psi

ϵ_m = 28.5%

TABLE LVI

(U) EFFECT OF CURATIVE RATIO ON UTEFLEX/HX-874 PROPELLANT WITH 0.5% ETHYL CENTRALITE

UTX No.	6564-1	6565-1	6566-1	6567-1	6568-1
Uteflex 64S	(1.0) 7.11	(1.0) 7.00	(1.0) 6.89	(1.0) 7.02	(1.0) 6.58
HX-874	(1.3) 0.70	(1.4) 0.74	(1.5) 0.78	(1.5) 0.80	(1.4) 0.69
Epon 812	(0.5) 0.41	(0.6) 0.48	(0.7) 0.55	(0.5) 0.40	(0.6) 0.45
DOA	0.50	0.50	0.50	0.50	1.00
TMETN	8.68	8.68	8.68	8.68	8.68
E.C.	0.50	0.50	0.50	0.50	0.50
Al(H-322)	16.00	16.00	16.00	16.00	16.00
AP (coarse)	35.70	35.70	35.70	35.70	35.70
AP (fine)	30.40	30.40	30.40	30.40	30.40
	σ_m ϵ_m	σ_m ϵ_m	σ_m ϵ_m	σ_m ϵ_m	σ_m ϵ_m
Measured	Too Soft	61.2 28.5	87.1 20.7	57.2 22.4	43.5 40.

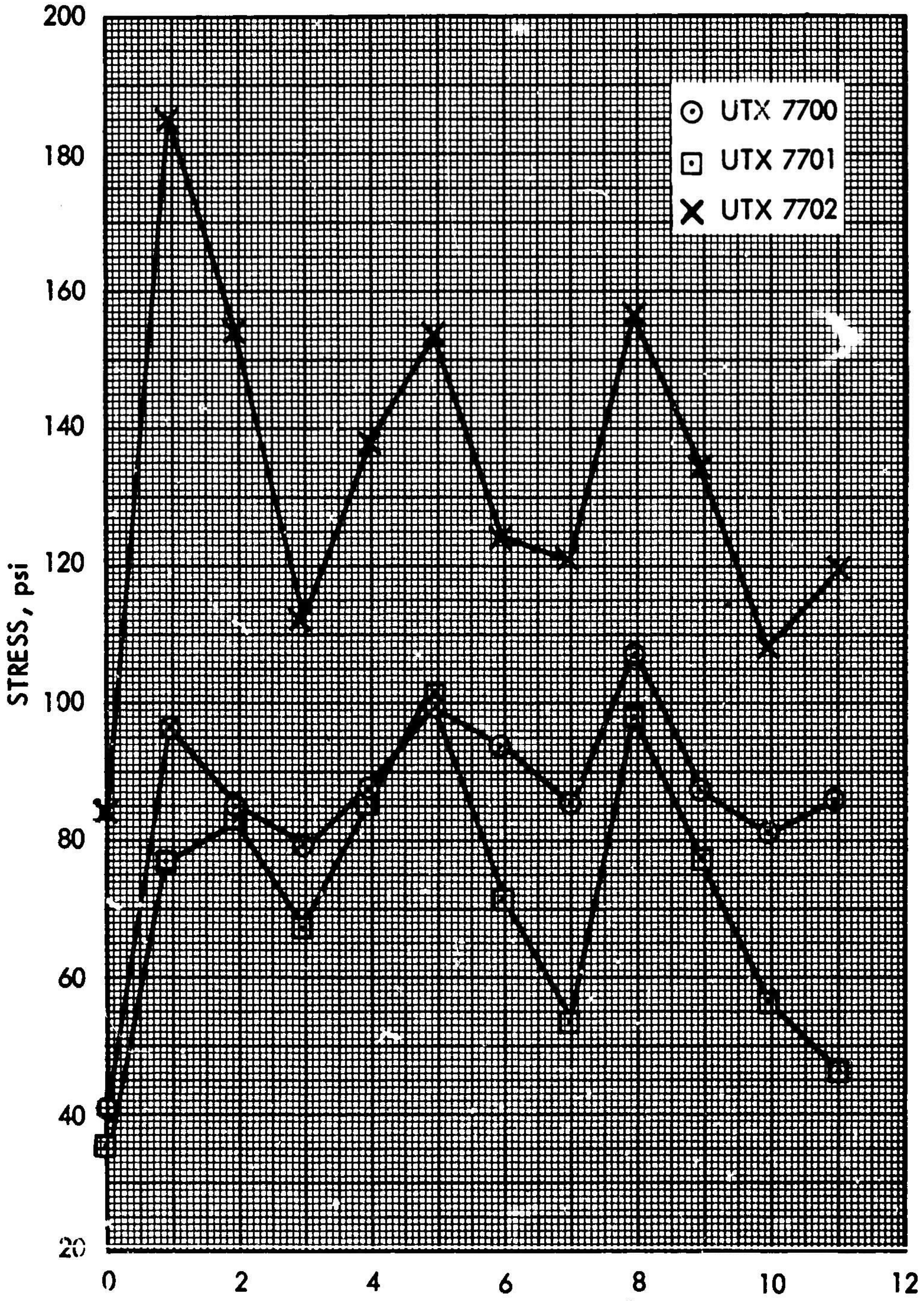

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Figure 39. (U) Effect of High Temperature Storage on Stress Properties of Propellant (HX-735 Polymer System)

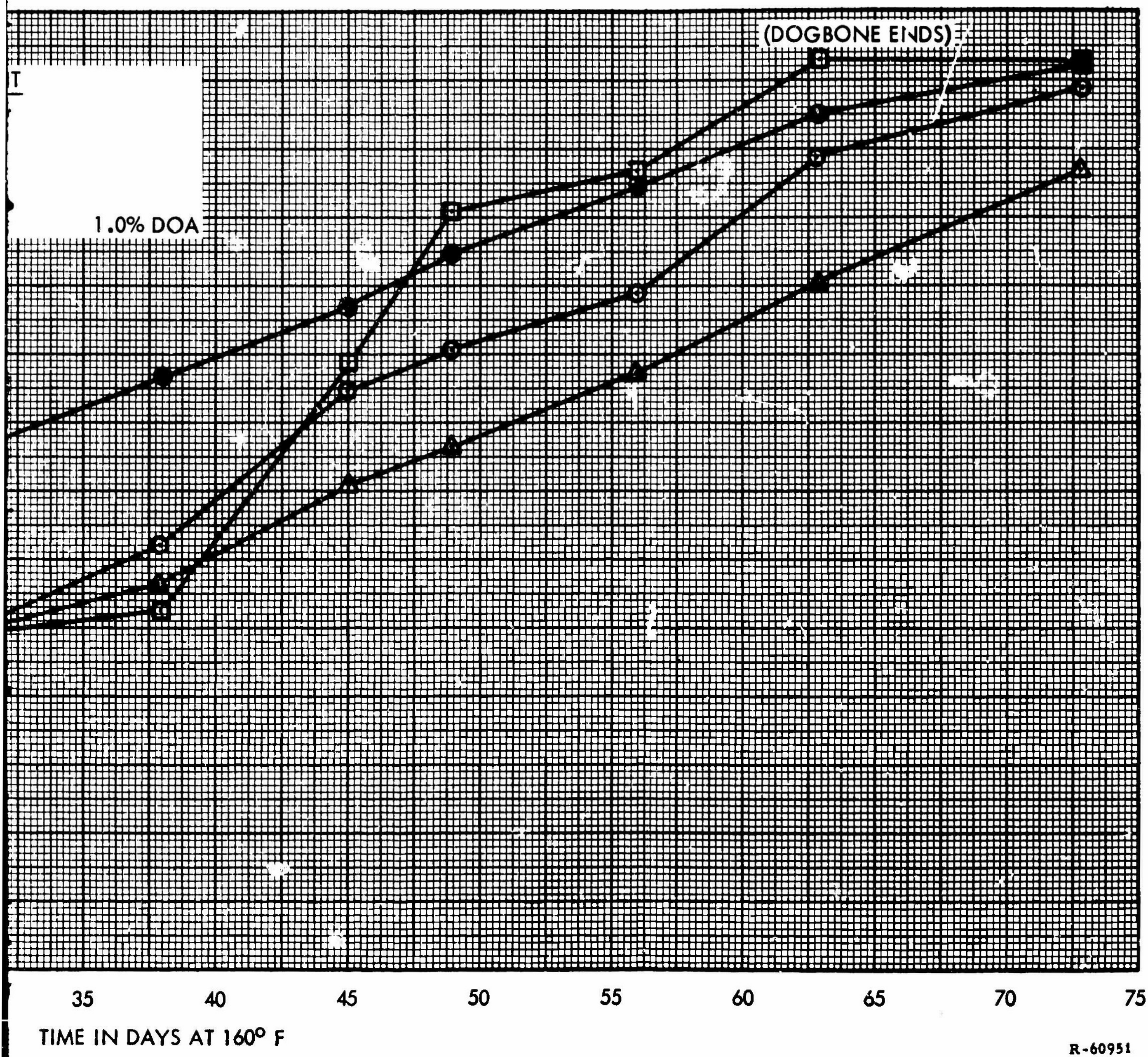

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Figure 41. (U) Effect of 160° F Storage on Uteflex Propellant as a Function of Curative Level

12

(U) According to the hardness data obtained on the end portions of the JANAF specimens which were subjected to 160° F storage, the 0.5% ethyl centralite concentration was adequate to prevent binder degradation over the 11-week storage period. However, because hardness appeared to be gradually increasing after 11 weeks, it is not clear if this is the result of continued crosslinking or the result of plasticizer loss.

(U) In order to gain additional perspective on the role of the stabilizers in effecting cure rate, mechanical properties, and stability, three of the most commonly used stabilizers — ethyl centralite, 2-nitrodiphenyl amine (2-NDPA), and resorcinol — were compared in an aluminized Uteflex formulation. Two curative levels were used — 1.0/1.5/0.5 and 1.0/1.3/0.5 equivalents of Uteflex/HX-874/Epon 812. The formulations and tensile properties, after a 5-day cure at 120° F, are presented in table LVII. Shore A hardness measurements as a function of storage time are shown in figures 42 and 43. The propellant specimens held in storage were 3-in. x 3-in. x 5-in. blocks of propellant so that any tendency to swell or crack in bulk could be more readily observed. It is obvious from both figures 42 and 43 that the propellant containing resorcinol attains essentially a complete cure in 5 days at 120° F, whereas formulations with the other stabilizers experience a degree of postcure at 160° F.

(U) Unfortunately, the effect of resorcinol is so pronounced that propellant pot life is seriously reduced, and a considerable increase in mix viscosity occurs during processing. According to the hardness data obtained, it appears that 0.5% resorcinol ethyl centralite or 2-NDPA is effective as a stabilizer for the conditions tested at an HX-874 curative level of 1.5 equivalents per equivalent of polymer. If the aziridine equivalent level is reduced to 1.3 then 2-NDPA is not effective at a concentration of 0.5%, although resorcinol is still quite effective. Ethyl centralite was tested at the reduced curative level at a concentration of 0.2% and was not effective at this concentration.

(U) On the basis of the data obtained it is reasonable to speculate that the measured tensile properties obtained by UTX 6573 ($\sigma_m = 80$, $\epsilon_m = 43$) at equivalent ratios of 1.0/1.3/0.5 using 0.5% resorcinol as a stabilizer might be

TABLE LVII

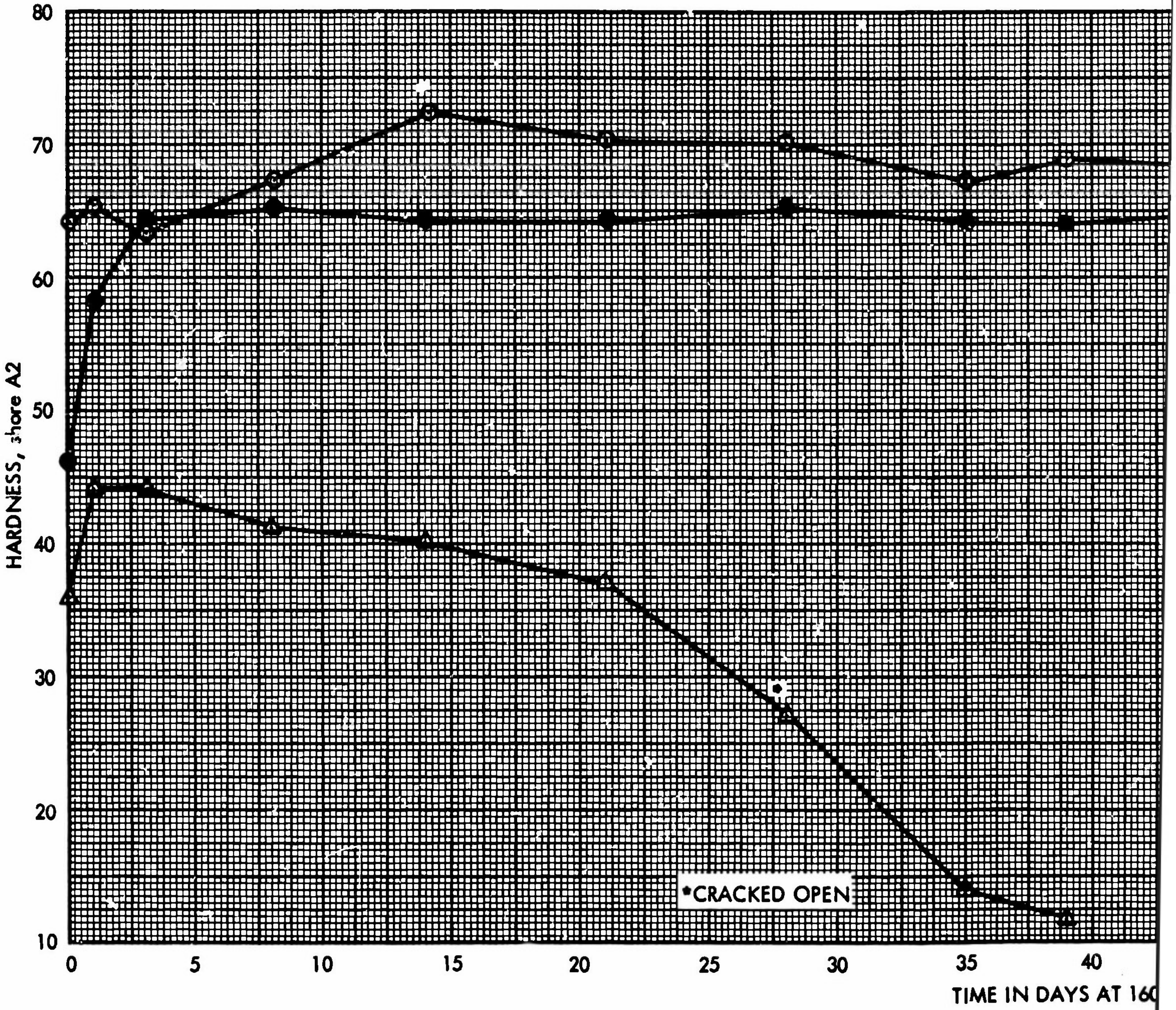
(U) EFFECT OF CURATIVE RATIO AND STABILIZER ON MECHANICAL PROPERTIES

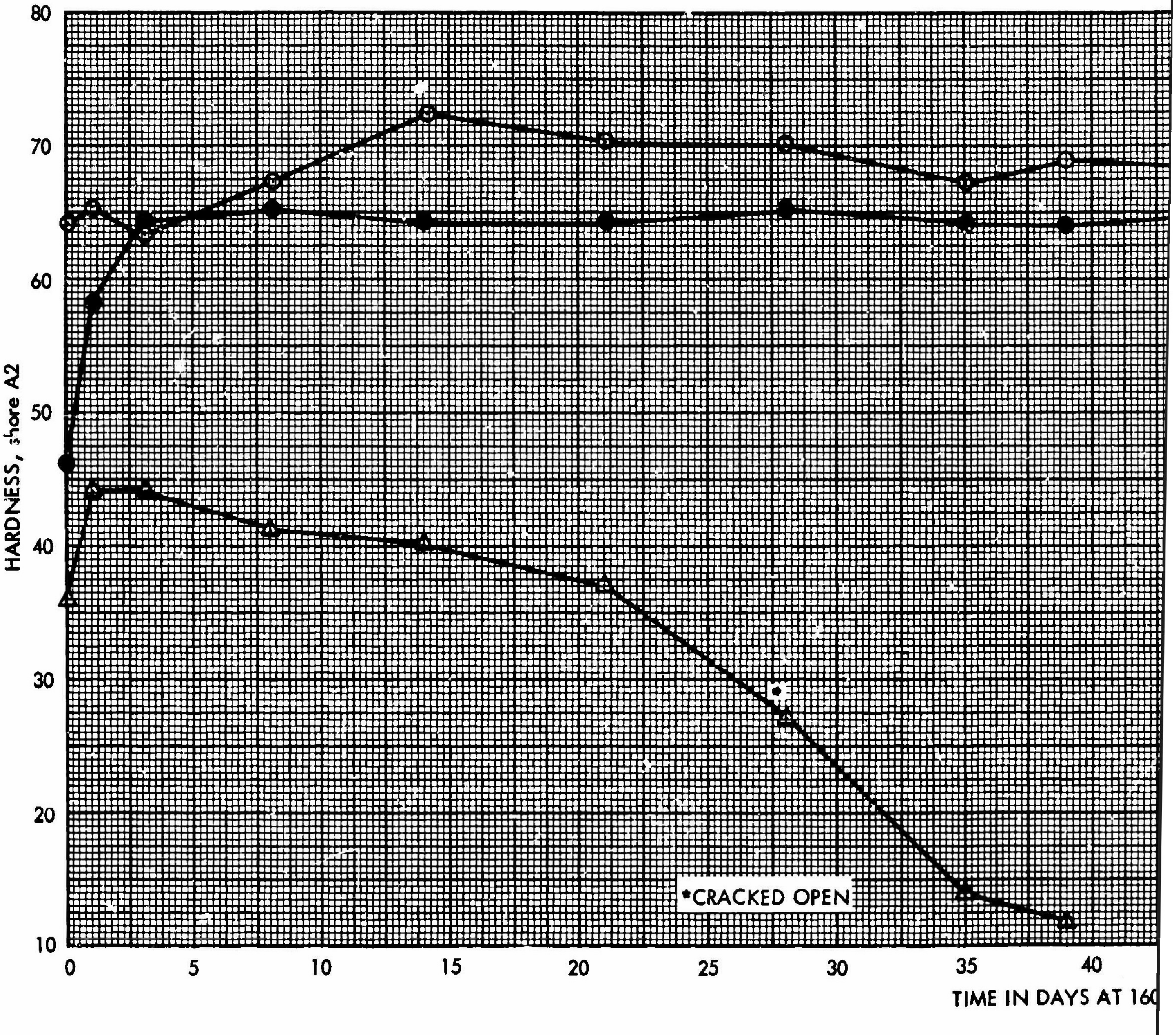
(Initial and Postcure Conditions)

	UTX-6567-2	UTX-6570-1	UTX-6571-1	UTX-6572-1	UTX-6573-1	UTX-6574-1	UTX-6575-1	UTX-6576-1
Curative ratios (equivalents)								
HX-874	1.5	1.5	1.5	1.5	1.3	1.3	1.3	1.3
Epon 812	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Stabilizer, wt-%								
Ethyl centralite	0.50	---	---	---	---	---	---	0.20
2-NDPA	---	---	0.50	---	---	0.50	---	---
Resorcinol	---	0.50	---	---	0.50	---	---	---
Measured								
σ_m	74.5	116	72.6	58	80	40	37	24
ϵ_m	25.9	22	25	27	43	50.4	54	55

Basic Formulations

Uteflex HX-874 Epon 812	8.22	TMETN	8.68
		AlH-322	16.00
		AP	66.10
DOA	0.50	Stabilizer	0.50


80
70
60
50
40
30
20
10
HARDNESS, shore A2
0
5
10
15
20
25
30
35
40
TIME IN DAYS AT 16
*CRACKED OPEN



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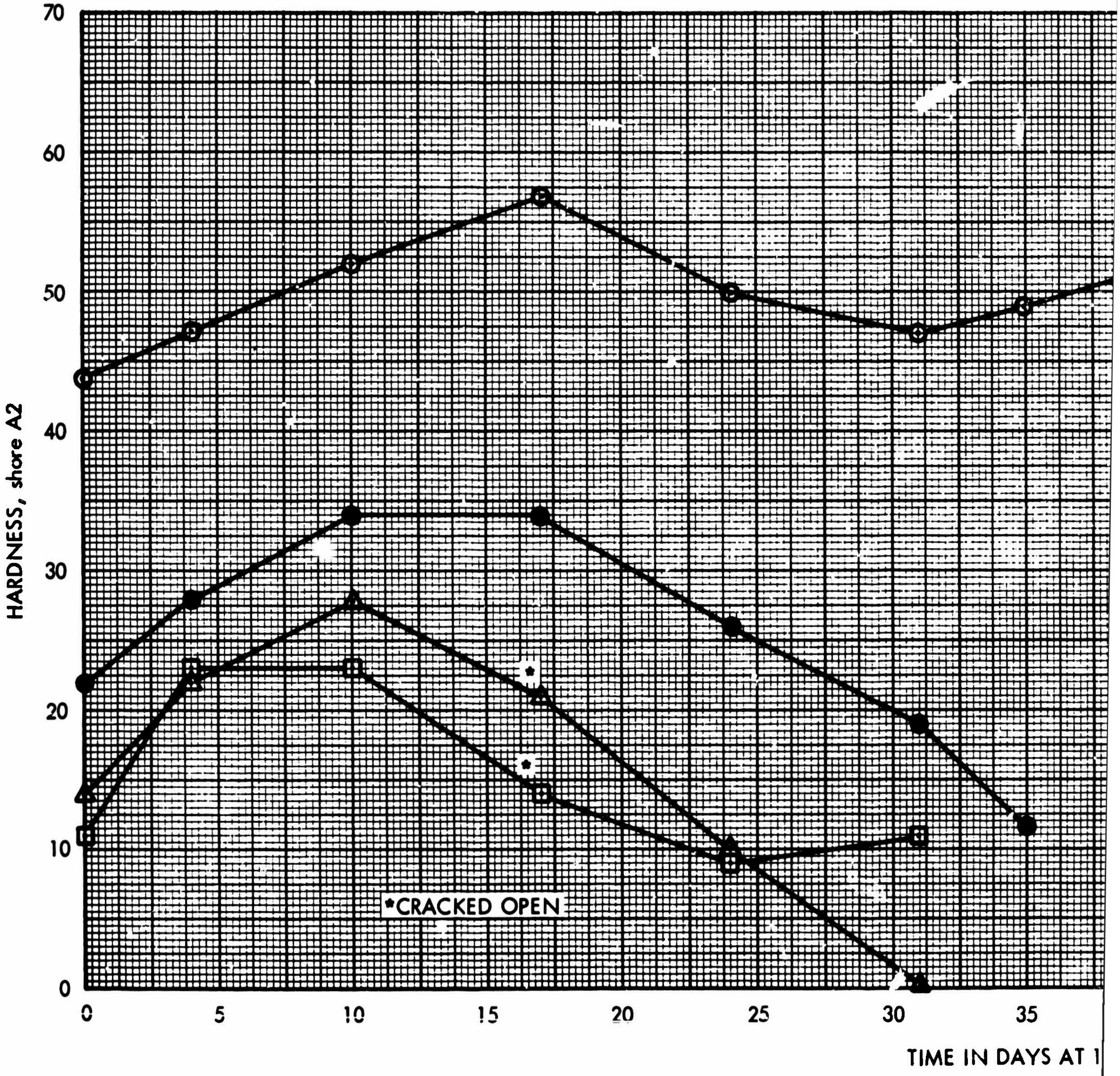
70
60
50
40
30
20
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HARDNESS, shore A2
0
5
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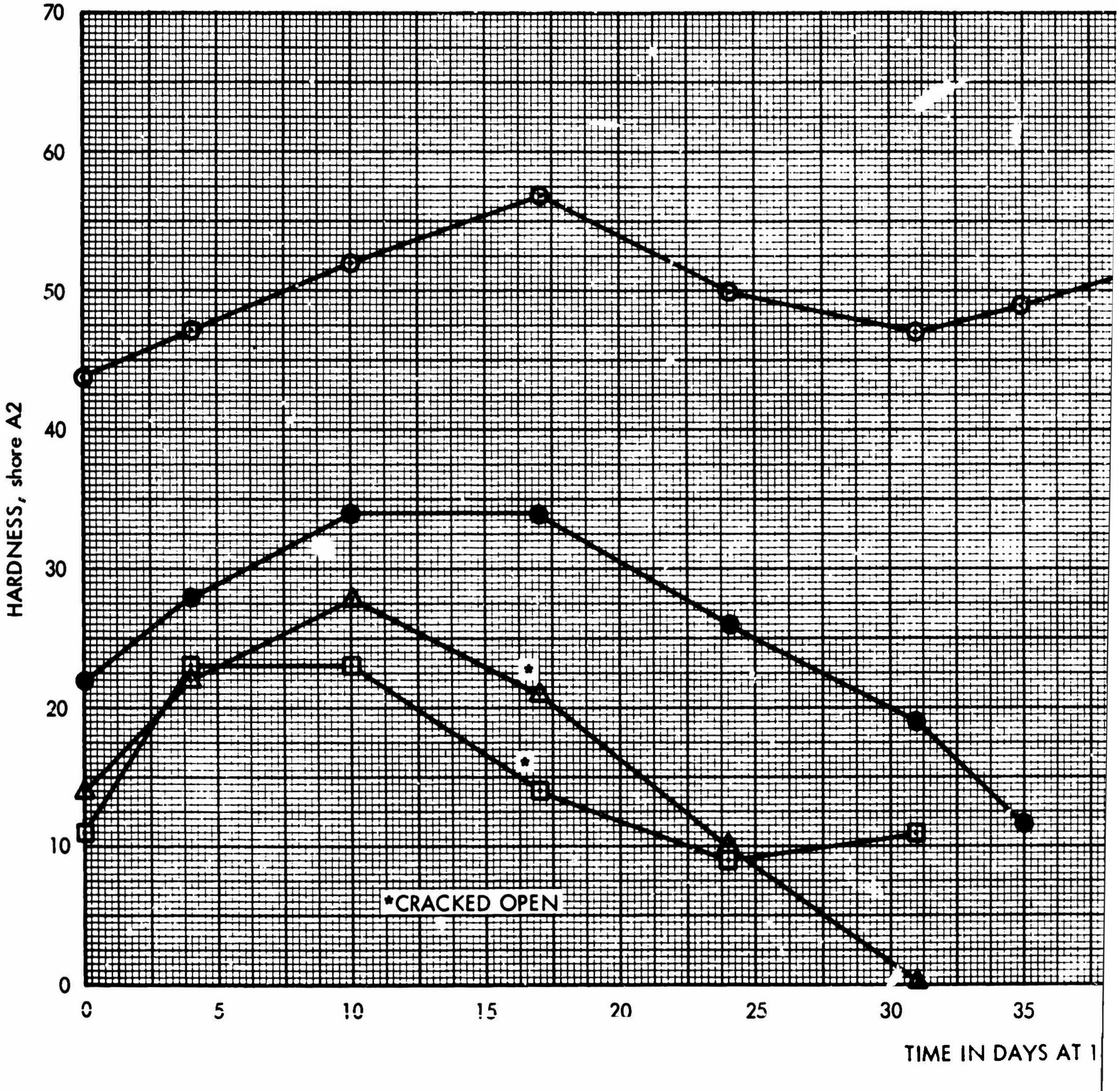
35
TIME IN DAYS AT 1
*CRACKED OPEN



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approximated in a processable LMH-1 formulation by a mixture of stabilizers that would cause the cure reaction to approach completion without eliminating the required pot life. However, the program was essentially completed before these tests were finished.

(5) Development of Improved Liner

(U) The liner development and improvement program was founded on the results of the binder tests used in the propellant development program. Ten basic binder formulations were chosen as prospective candidates. Each binder was mixed with Elftex-5, a carbon black, and Cab-O-Lite, a trade designation for wollastonite, both of which act as reinforcing fillers. Small cups were partially filled with the selected liner formulations and cured at 140° F for 16 hr. Following this cure, UTX 7702 propellant was cast over the liner and cured at 120° F for 168 hr. After the propellant cure, a qualitative evaluation was made of the propellant-to-liner bond. This examination shows that the best bonds were observed with L200-36-4, L200-38-10, and L-200-41-1 liners. (See table LVIII.)

(U) Quantitative evaluations were made with these three liners and a comparison was made with a control formulation (CTL-162-58) which had been used regularly in previous LMH-1 programs. The propellants selected for the quantitative tests were the same formulations that had been used in the accelerated aging study which are described in table LIX. A summary of the quantitative values at failure and points of failure values show only slight difference for the systems tested. However, the type of failure showed that the CTL-162-58 liner had better propellant-to-liner adhesion.

(U) One of the primary disadvantages of the CTL 162-58 liner was the presence of ground ammonium sulfate in the filler portion of the formulation. A formulation modification was made by substitution of wollastonite (Cab-O-Lite) for the ammonium sulfate. Preliminary qualitative tests showed good propellant-to-liner adhesion with this substitution. Shear test samples were prepared with the modified liner CTL 200-83) and UTX 7700 propellant. The liner was subjected to two cures. One was 16 hr at 160° F, and the

TABLE LVIII

(U) SUMMARY OF LINER FORMULATIONS

	CTL 200-83		CTL 200-36-4		CTL 200-38-10		CTL 200-41-1		CTL 162-58	
Material	Each	wt-%	Each	wt-%	Each	wt-%	Each	wt-%	Each	wt-%
HX-735	1.0	72.5	1.0	70.0	1.0	70.0	1.0	70.0	1.0	72.5
HX-874	---		---		1.5		1.0		---	
MAPO	2.0		---		---		---		2.0	
TBM	---		1.0		---		0.8		---	
NTPB	---		1.0		---		---		---	
Epon 812	1.4		---		0.2		---		1.4	
P-33 Carbon Black		1.3		---		---		---		1.3
Elftex-5		5.0		5.0		5.0		5.0		5.0
Cellulose Acetate		2.5		---		---		---		2.5
Cab-O-Lite		18.7		25.0		25.0		25.0		---
Ammonium Sulfate		---		---		---		---		18.7

TABLE LIX

(U) PROPELLANT-TO-LINER BOND SHEAR TESTS

Liner CTL	Propellant UTX	Stress at Failure psi	Type of Failure
200-36-4	7700	40	50% Propellant Cohesive Failure 50% Propellant Liner Adhesive Failure
200-36-4	7701	55	25% Propellant Cohesive Failure 75% Propellant Liner Adhesive Failure
200-36-4	7702	59	10% Propellant Cohesive Failure 90% Propellant Liner Adhesive Failure
200-38-10	7700	45	50% Propellant Cohesive Failure 50% Propellant Liner Adhesive Failure
200-38-10	7701	50	15% Propellant Cohesive Failure 85% Propellant Liner Adhesive Failure
200-38-10	7702	57	5% Propellant Cohesive Failure 95% Propellant Liner Adhesive Failure
200-41-1	7700	44	10% Propellant Cohesive Failure 90% Propellant Liner Adhesive Failure
200-41-1	7701	53	10% Propellant Cohesive Failure 90% Propellant Liner Adhesive Failure
200-41-1	7702	55	5% Propellant Cohesive Failure 95% Propellant Liner Adhesive Failure
162-58	7700	43	50% Liner Cohesive Failure 50% Propellant Liner Adhesive Failure
162-58	7701	46	40% Liner Cohesive Failure 60% Propellant Liner Adhesive Failure
162-58	7702	55	10% Liner Cohesive Failure 90% Propellant Liner Adhesive Failure

second was 8 hr at 160° F. After liner cure, the propellant was cast and cured 120 hr at 120° F. In all tests the failure occurred within the propellant. Table LX summarizes the data. From the data it appears that CTL 200-83 liner is an adequate, if not an improved, formulation for PEP propellants.

TABLE LX

(U) PROPELLANT-TO-LINER BOND SHEAR TESTS

Liner	CTL-200-83
Propellant	UTX-7700

Liner Cure hr	Stress at Failure psi	Type of Failure
8	43	100% Propellant Cohesive Failure
16	42	100% Propellant Cohesive Failure

3. PHASE II – PROPELLANT CHARACTERIZATION

a. Ballistic Characterization – Sea Level

(C) As a result of the development studies conducted on phase I of this program, and described in section 2. of this report, a propellant formulation was selected for intensive characterization of ballistic, mechanical, hazard and storage properties. The formulation, UTP 8812, is based [illegible] PEP [illegible]0 binder system containing Uteflex, HX-874, an[illegible] 812 to form the cured polymeric structure. TMETN is the nitrato-ester plasticizer. A nonenergetic plasticizer, dioctyl adipate, is used in small concentrations as a processing aid.

(U) The solids loading for this formulation (75 vol-% solids, 25 vol-% binder) represents a slight retreat from the 23 vol-% binder processed in phase I with the HX 735/MAPO binder system. The increase in binder level was made because of the greater reactivity of the Uteflex/HX 874 system which was initially more difficult to process because of pot-life limitations.

(U) The selection of the final formulation for detailed characterization studies was influenced by the unexpected effect of ethyl centralite on the propellant cure process. The effects of three candidate stabilizers, ethyl centralite, 2-NDPA, and resorcinol were compared for their effects on the cure process and on physical properties, as discussed in detail in section II, e., (4). It was apparent from these studies that ethyl centralite reduced the cure rate substantially, and that resorcinol increased the cure rate to the extent that the pot life of the propellant was almost eliminated. The curative level and stabilizer level selected, therefore, was a tradeoff between adequate physical properties after 3 days' cure at 120° F and minimum postcure.

(U) Studies concluded after all of the propellant had been processed for the characterization tests indicated that resorcinol was a superior stabilizer for this binder in an aluminized system in terms of high temperature storage. If methods could have been developed for its use, a substantial improvement in mechanical properties and in the stability of mechanical properties would have accrued to this propellant system.

(C) The selected propellant is the following:

UTP 8812

	Equivalents	wt-%
Uteflex	1.0	9.20
HX-874	1.4	
Epon 812	0.6	
DOA		0.60
TMETN		9.50
LMH-1		24.00
AP		56.50
Ethyl centralite		0.20
O/F ratio		1.4
Theoretical I_{sp} (sec)		280.9
Theoretical density (lb/in.^{3})		0.059

(C) A total of 14 10-lb motors were tested at UTC at nominal sea-level conditions. An average I_{sp} efficiency of 92.7% has been obtained. The test results are presented in table LXI. Specific impulse was corrected from measured P_c to 1,000 psia by two methods (1) the standard C_f correction using a γ of 1.18, and (2) by multiplying the theoretical I_{sp} at 1,000/14.7, optimum expansion, 0° exit angle by the I_{sp} efficiency, where I_{sp} efficiency = measured I_{sp}/theoretical I_{sp} at the measured chamber pressure and expansion ratio. Method (1) has been traditionally used in the industry for many years but is recognized as a tool of limited accuracy, particularly as γ is not well characterized for AlH_3 propellant systems. Method (2) is independent of γ but assumes constant efficiency at varying pressures. The increased use of computers has recently allowed this method to be more universally employed and is the preferred method of normalizing I_{sp} data.

(C) By using the C_f correction method, an average I_{sp} (1,000/14.7, 0°) of 258.3 sec was obtained in 10-lb motors. The use of the I_{sp} efficiency as a correction factor for these tests yields an average value of 260.4 sec. At pressures close to 1,000 psia methods (1) and (2) agree closely. At low pressures, method (2) gives significantly higher numbers.

(C) Tests of 12 control motors containing UTP 3001 propellant yielded an average I_{sp} efficiency of 93.2%. The corrected

TABLE LXI

(U) BALLISTIC CHARACTERIZATION

Test No.	Nominal P_c psia	K_{ni}	ϵ	r_b in./sec	P_{c_a} psia	P_{c_b} psia	c* ft/sec	c* Theoretical ft/sec	c* Efficiency %
									UTP 8812
288	1,000	256	8.693	0.387	880.7	916.0	5,319	5,536	96.1
289	450	←							Poor tr
291	250	74.6	2.929	0.283	187.5	194.9	5,3[illegible]	5,484	98.4
292	750	214.9	6.984	0.335	638.8	657.8	5,345	5,525	96.7
295	250	93.1	---	0.306	233.5	246.0	←		
297	750	264.4	---	0.349	780.7	820.2	←		
299	250	92.4	4.467	0.295	232.4	242.2	5,212	5,492	94.9
300	450	154.9	5.276	0.344	476.2	492.0	5,382	5,516	97.6
301	750	238.2	7.829	0.329	700.7	721.0	5,300	5,527	95.9
302	1,000	312.0	9.019	0.445	1,188.8	1,249.8	5,329	5,542	96.2
305	250	92.8	3.17	0.336	253.4	265.4	5,224	5,495	95.1
307	450	148.3	6.64	0.335	420.9	439.9	5,403	5,512	98.0
309	750	241.2	6.973	0.362	766.7	790.3	5,347	5,530	96.7
311	1,000	276.2	9.039	0.388	897.7	951.0	5,370	5,535	97.0
							5,330		96.6
									UTP 3001
287	1,000	265.0	8.703	0.414	984.1	1,028.9	5,070	5,167	98.1
290	1,000	262.8	8.791	0.401	940.3	987.4	5,090	5,164	98.6
293	1,000	262.1	8.771	0.428	974.0	1,028.6	5,026	5,165	97.3
294	1,000	265.6	8.723	0.445	979.1	1,020.0	5,058	5,165	97.9
296	1,000	265.6	---	0.480	945.2	1,029.8	5,050	←	
298	1,000	266.2*	7.636	0.429	952.2	1,034.2	5,089	5,165	98.5
303	1,000	267.3	8.784	0.413	946.4	1,003.9	4,964	5,163	96.1
304	1,000	265.0	8.68	0.480	952.0	1,040.6	4,984	5,165	96.5
306	1,000	265.0	8.71	0.394	945.3	975.9	5,061	5,165	98.0
308	1,000	265.6	8.725	0.386	970.0	990.1	5,113	5,167	99.0
310	1,000	263.3	8.651	0.432	912.9	999.2	5,061	5,163	98.0
312	1,000	263.3	8.650	0.390	932.9	964.2	5,110	5,164	99.0
							5,057		97.9

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* Long tailoff

TABLE LXI

ACTERIZATION, 10-lb MOTORS

al	c* Efficiency %	Delivered P_c sec	Corrected to 0° L at P_c	Corrected to 1,000/0° (A)	Corrected to 1,000/0° (B)	Theoretical at Test Conditions	I_{sp} Efficiency
UTP 8612							
	96.1	253.1	257.4	260.7	260.7	277.5	92.8
	Poor trace						
	98.4	205.2	208.7	253.2	258.7	226.6	92.1
	96.7	244.9	249.1	259.4	260.7	268.3	92.8
	Bent flexure – thrust measurements invalid						
	Bent flexure – thrust measurements invalid						
	94.9	213.4	217.0	258.5	262.1	232.5	93.3
	97.6	239.6	243.7	261.6	264.4	259.1	94.1
	95.9	246.9	251.1	259.4	260.2	271.1	92.6
	96.2	259.8	264.2	260.8	261.0	284.4	92.9
	95.1	213.4	217.0	252.0	256.5	237.8	91.3
	98.0	233.1	237.1	260.2	262.1	254.2	93.3
	96.7	248.1	252.3	258.5	259.6	273.0	92.4
	97.0	250.9	255.3	257.6	257.9	278.0	91.8
	96.6			258.3	260.4		92.7
UTP 3001							
	98.1	239.4	243.5	243.8	244.2	260.8	93.4
	98.6	237.7	241.7	243.0	243.2	259.9	93.0
	97.3	238.7	242.8	243.1	243.5	260.7	93.1
	97.9	239.1	243.2	244.2	244.0	260.8	93.3
	Bent flexure – thrust measurements invalid						
	98.5	239.9	244.0	244.9	245.8	259.5	94.0
	96.1	239.5	243.6	244.6	245.0	260.0	93.7
	96.5	237.2	241.2	242.2	242.4	260.1	92.7
	98.0	238.4	242.5	243.5	244.0	260.0	93.3
	99.0	237.9	241.9	242.3	242.7	260.6	92.8
	98.0	235.3	239.3	241.2	241.4	259.2	92.3
	99.0	237.8	241.8	243.3	243.5	259.7	93.1
	97.9			243.3	243.6		93.2

average I_{sp} value for the control motors is 243.3 sec using method (1) or 243.6 sec using method (2). The range of values are within the range of a previous series of calibration tests for the test stand used, and the data obtained on the 10-lb tests of UTP 8812 are considered to be accurate.

(C) The burning rate-versus-pressure and K_n-versus-pressure curves for UTP 8812 generated from these tests are shown in figure 44. The r_b-versus-P_c curve has two inflection points, one at 490 and one at 720 psia. The burning rate values from these tests are:

r_b = 0.395 in./sec at 1,000 psia
r_b = 0.34 in./sec at 750 psia
r_b = 0.34 in./sec at 500 psia

n = 0.2 below 490 psia
n = 0.1 between 490 and 720 psia
n = 0.54 above 720 psia.

b. Ballistic Characterization — Simulated Altitude

(U) A total of 12 10-lb motors containing UTX 8812 and 14 10-lb motors containing UTP 3001, a standard aluminized formulation, were shipped to Arnold Engineering Development Center (AEDC) for testing at simulated high altitude. The motors were tested at a nominal altitude of 125,000 ft in Propulsion Engine Test Cell T-3 of the Rocket Test Facility at AEDC.

(U) Nozzles having area ratios of 20, 40, 60, and 80:1 were used for these tests. All motors were reported to have ignited and burned normally. The ignition altitude ranged between 116,000 and 131,000 ft, and the average altitude during motor operation ranged between 116,000 and 129,000 ft. Because of the high altitude maintained during motor operation, the maximum correction to the measured total impulse required to obtain vacuum total impulse data was 0.28%.

(U) A summary of the individual motor tests is presented in table LXII. These results are considered by ARO, Inc. to be preliminary, and a final report will be issued by them. Theoretical I_{sp} calculations for each test, and I_{sp} efficiencies are included. The I_{sp} data are based on propellant weight data

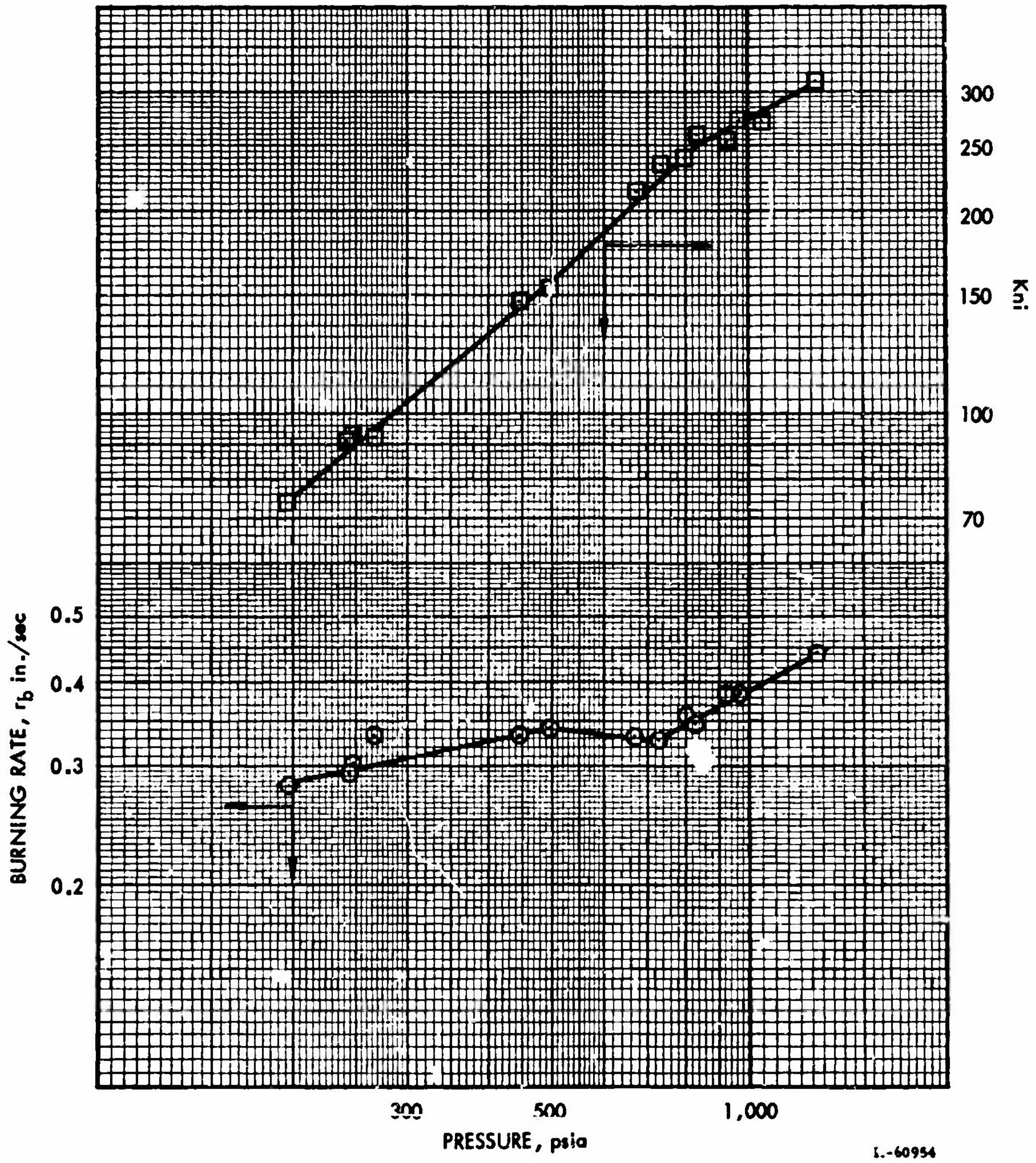

Figure 44. (U) Burning Rate and K_n vs Pressure for UTP 8812

TABLE LXII

(U) VACUUM I_{sp} TESTS CONDUCTED AT AEDC IN 10-lb MOTORS

UTP 8812

						Specific Impulse			
Test No.	Nominal Area Ratio	Average Altitude 1,000 ft	P_{ca} psia	$P_{ambient}$ psia	Measured Area Ratio	Measured seconds	Corrected to 0° seconds	Theoretical at Test Conditions	I_{sp} Efficiency
01	80	125	950.3	0.054	81.51	309.2	314.5	345.7	91.0
02	80	125	1042.5	0.053	79.95	309.9	315.2	345.5	91.2
03	80	126	910.3	0.051	80.07	307.8	313.0	345.4	90.6
04	60	127	904.8	0.048	59.62	305.4	310.6	341.3	91.0
06	60	125	902.5	0.052	59.83	304.7	309.9	341.3	90.8
09	60	128	858.7	0.049	60.02	304.6	309.8	341.4	90.7
10	40	127	850.4	0.050	41.21	300.3	305.4	335.5	91.0
16	40	122	874.5	0.061	40.89	300.9	306.0	335.3	91.3
22	40	116	975.9	0.074	41.38	302.9	308.0	335.5	91.8
23	20	124	984.6	0.052	20.04	291.9	296.9	322.3	92.1
24	20	124	971.7	0.054	19.98	291.3	296.3	322.2	92.0
25	20	123	1006.4	0.056	19.81	291.5	296.5	322.0	92.1
					UTP 3001				
05	80	127	1043.7	0.049	81.26	291.0	295.9	320.7	92.3
06	80	125	1064.4	0.054	81.53	291.1	296.0	320.7	92.3
07	80	128	1063.1	0.048	80.52	292.0	297.0	320.7	92.6
11	60	127	1037.8	0.049	60.30	288.1	293.0	317.0	92.4
12	60	129	1013.3	0.045	59.46	287.5	29[illegible]	316.8	92.3
13	60	128	1024.20	0.047	60.47	287.6	292.5	31[illegible]	92.3
14	60	126	1027.3	0.050	59.55	287.5	292.4	316.8	92.3
15	60	126	1010.2	0.050	60.33	287.4	292.3	317.0	92.2
17	40	123	997.2	0.058	41.18	283.7	288.5	311.6	92.6
18	40	124	999.7	0.051	40.41	283.8	288.6	311.3	92.7
19	40	126	1005.8	0.052	41.49	283.8	288.6	311.7	92.6
20	20	125	1012.0	0.052	19.88	274.7	279.4	299.4	93.3
21	20	123	983.1	0.054	20.49	274.4	279.1	300.0	93.0
26	20	125	1017.9	0.052	20.02	275.3	280.8	299.6	93.5

supplied by UTC rather than expended propellant weight determined by weight of the motor before and after testing.

(C) The average performance data as a function of area ratio are summarized in table LXIII. The average I_{sp} efficiency for each formulation decreases slightly as expected at the higher expansion ratios. For example, the I_{sp} efficiency of UTP 3001 dropped from 93.2% to 92.4% as the expansion ratio increased from 20:1 to 80:1, a decrease of about 0.8%. The AlH_3 propellant yielded an average I_{sp} efficiency of 92.1% at an expansion ratio of 20:1 which was reduced to 90.9% at the 80:1 expansion ratio, a decrease of 1.14%. Because the AlH_3 propellant contains 21.6% aluminum metal compared to 16% metal in UTP 3001, it is not surprising that it would incur a slightly higher penalty because of a two-phase flow loss at the higher expansion ratio.

TABLE LXIII

(U) SUMMARY OF VACUUM TESTS IN 10-lb MOTORS

UTX 8812

Nominal Area Ratio	No. of Tests	Vacuum Corrected, I_{sp} 15° Exit Angle sec	I_{sp} Efficiency %
80	3	309.0	90.9
60	3	304.9	90.8
40	3	300.9	91.4
20	3	291.5	92.1
Sea-level tests at UTC			92.7

UTP 3001

Nominal Area Ratio	No. of Tests	Vacuum Corrected, I_{sp} 15° Exit Angle sec	I_{sp} Efficiency %
80	3	291.3	92.4
60	5	287.6	92.3
40	3	283.8	92.6
20	3	274.8	93.3
Sea-level tests at UTC			93.2

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c. BATES Motors

(U) A total of six BATES motors, each containing a nominal 60-lb of UTP 8812 propellant, were processed and shipped to the Air Force for testing. Based on propellant grain weights and measurements, all propellant grains had densities greater than 99% of the theoretical value.

d. Mechanical and Physical Properties

(U) During the course of the program the complete uniaxial tensile, viscoelastic and failure properties of the AlH_3 propellant, UTP 8812, were characterized. In addition, all the major physical properties of this system were evaluated.

(1) Viscoelastic Characterization

(U) Viscoelastic properties of UTP 8812 propellant were measured by both constant strain-rate and stress-relaxation tests with bonded-end tensile specimens. The constant strain-rate tests were conducted at four temperatures, and data from these tests were shifted to obtain a master stress-strain curve and time-temperature shift factor. Differentiation of the master stress-strain data yielded a master modulus curve shown in figure 45. A positive modulus slope for some individual constant strain-rate tests indicate gross strain nonlinearity. Data scatter also was excessive because porosity varied from one sample to another. Because of the excessive data scatter and nonlinear stress-strain behavior, stress-relaxation tests were conducted at ten temperatures. These relaxation modulus curves were shifted using experimental time-temperature shift factors which are comparable to the WLF* values used for the constant rate data. These data are shown on the master modulus curve (figure 46) and the time-temperature shift factor curve (figure 47).

(U) The overall master modulus curve shows relaxation behavior over a reduced time range from 10^{-13} to 10^{-4} min with a glassy modulus of 100,000 psi and an equilibrium modulus in the 84 to 160 psi range.

(2) Failure Characterization

(a) Uniaxial Tension

(U) Tensile failure properties of UTP 8812 were obtained by testing milled JANAF specimens with a

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* Williams, M. L., R. F. Landell, and J. P. Ferry, J. Am. Chem. Soc., 77: 374, 1955.

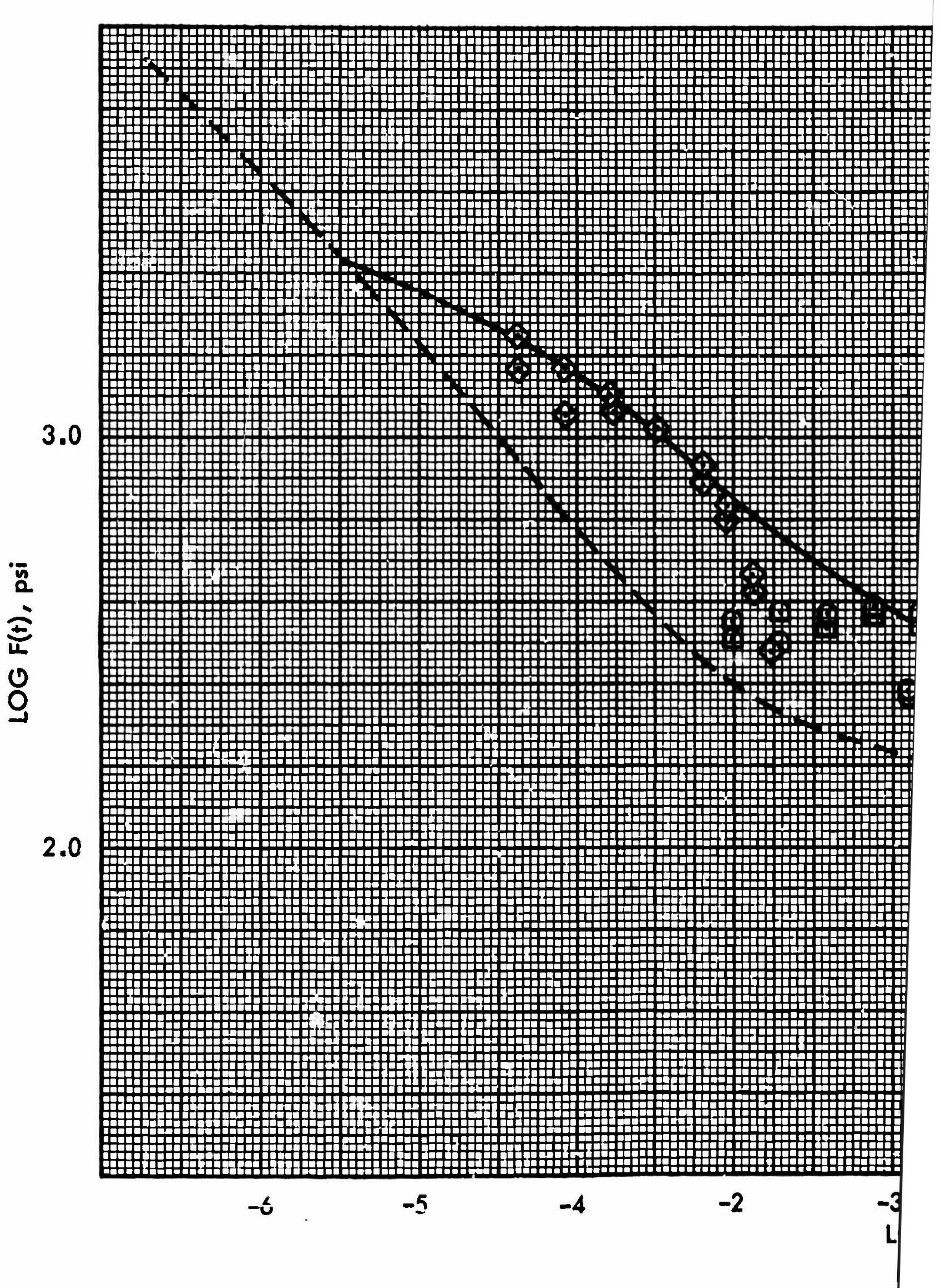
plastic gauge, as described in the Interagency Chemical Rocket Propulsion Group (ICRPG) test manual. Multiple samples were tested at eight temperatures from +128° to -70° F at 2.0 in./min crosshead speed (table LXIV). The measured stress and strain data as a function of test temperature are shown in figures 48 and 49. Measured strain values are approximately 35% at 76° F. Below -10° F the measured strain capability decreases rapidly reaching 5% at -30° F. Measured stress values are approximately 28 psi at 76° F and reach a peak of 750 psi at -30° F.

(b) Uniaxial Shear

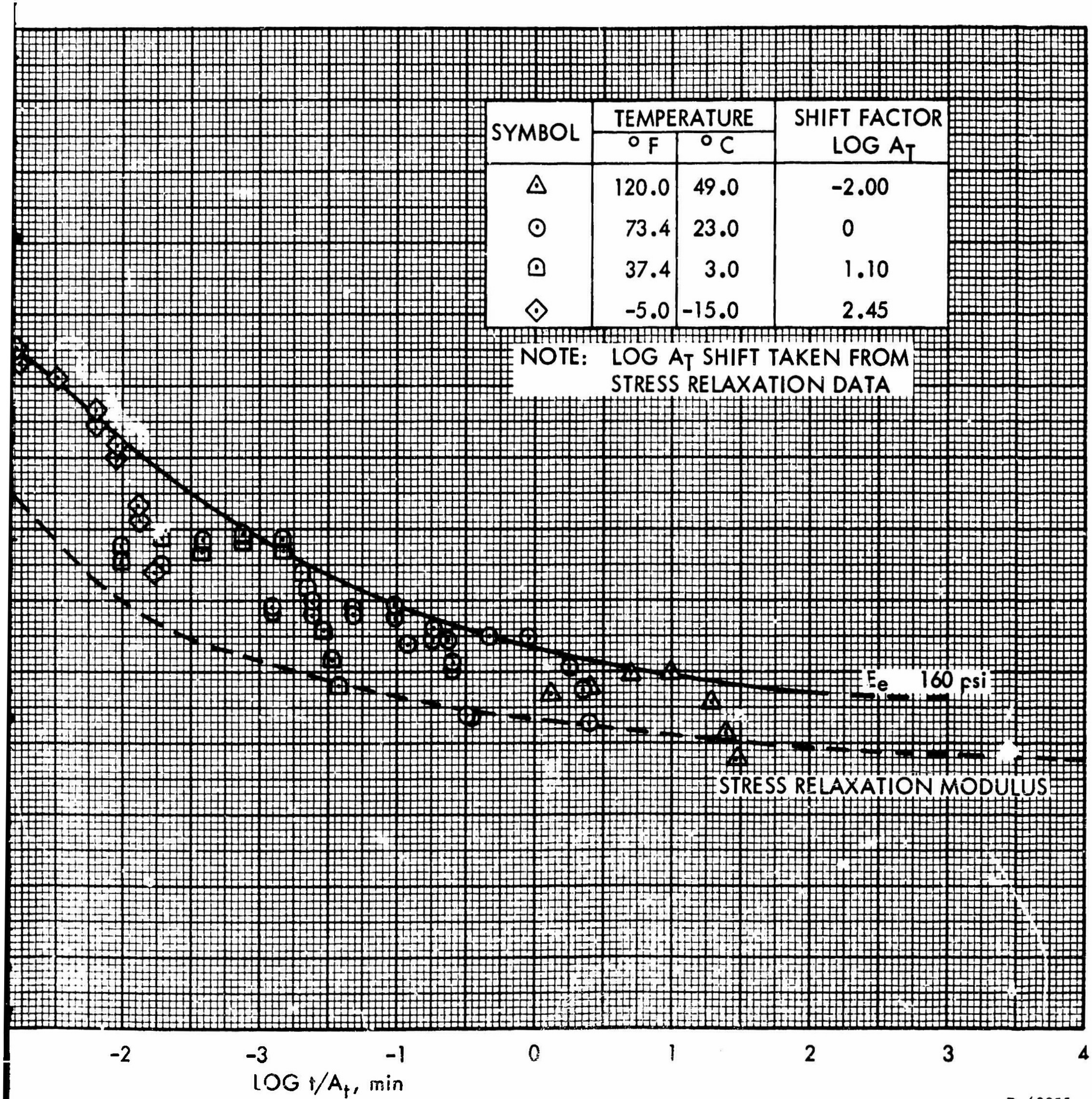
(U) Uniaxial shear-failure properties of UTP 8812 were measured by testing double-shear specimens at a crosshead speed of 2.0 and 0.2 in./min on an Instron Tester. Samples were prepared by casting the propellant into prelined metal fixtures that are used as part of the test assembly. Failure of the shear sample occurs by cracking in a direction parallel to the shearing direction in the weakest region of the liner propellant bond area. A total of 7 shear samples were tested at 3 temperatures (figure 50). All samples failed in the propellant. The average shear-failure stress was about equal to the tensile-failure stress at 76° F. The shear-failure strain at 76° F was 45%.

(c) Biaxial Tension

(U) Biaxial tension failure properties of UTP 8812 were measured by testing the bonded-end filleted-sheet specimens shown in figure 51 at a crosshead speed of 2.0 in./min. Samples were prepared by casting the propellant into prelined wooden boxes which were sawed into slabs for milling. Failure of the biaxial strip samples occurred in the central uniform region of the propellant. A total of 9 samples were tested at three temperatures. The data presented in figure 52 show that the biaxial strain values are approximately one-half of the tensile data at 76° F or above. The biaxial strain values at 10° F are approximately equal to measured JANAF tensile data.



LOG F(t), psi
3.0
2.0
-6
-5
-4
-2
-3
L



SYMBOL	TEMPERATURE °F	TEMPERATURE °C	SHIFT FACTOR LOG A_T
△	120.0	49.0	-2.00
⊙	73.4	23.0	0
⌂	37.4	3.0	1.10
◇	-5.0	-15.0	2.45

NOTE: LOG A_T SHIFT TAKEN FROM STRESS RELAXATION DATA

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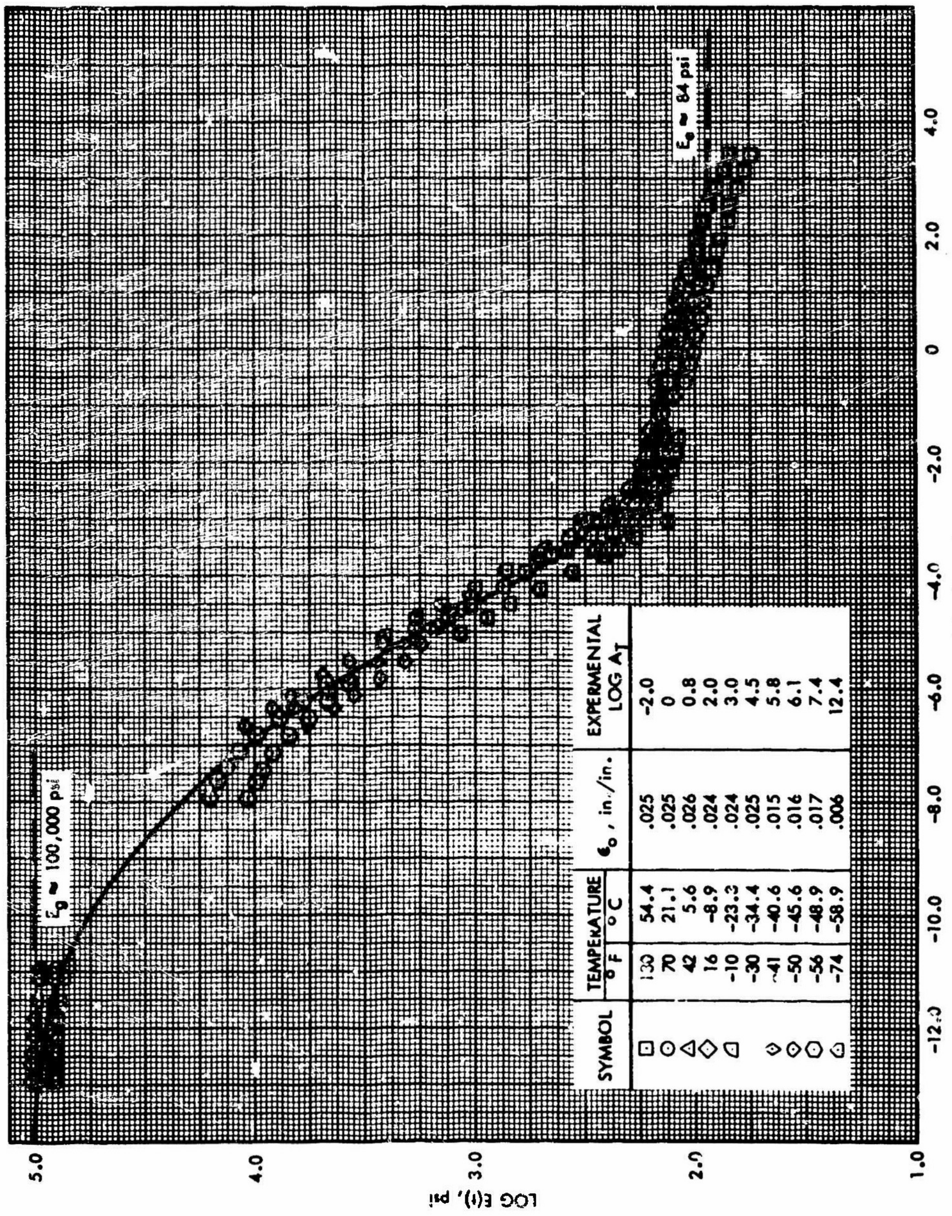
Figure 45. (U) Constant Strain Rate Tensile Modulus of UTP 8812

12



SYMBOL	TEMPERATURE °F	°C	ϵ_o, in./in.	EXPERMENTAL LOG A_T
⊡	130	54.4	.025	-2.0
⊙	70	21.1	.025	0
△	42	5.6	.026	0.8
◇	16	-8.9	.024	2.0
◁	-10	-23.3	.024	3.0
◇	-30	-34.4	.025	4.5
⊙	-41	-40.6	.015	5.8
⬡	-50	-45.6	.016	6.1
⌂	-56	-48.9	.017	7.4
	-74	-58.9	.006	12.4

Figure 46. (U) Stress Relaxation Tensile Modulus of UTP 8812

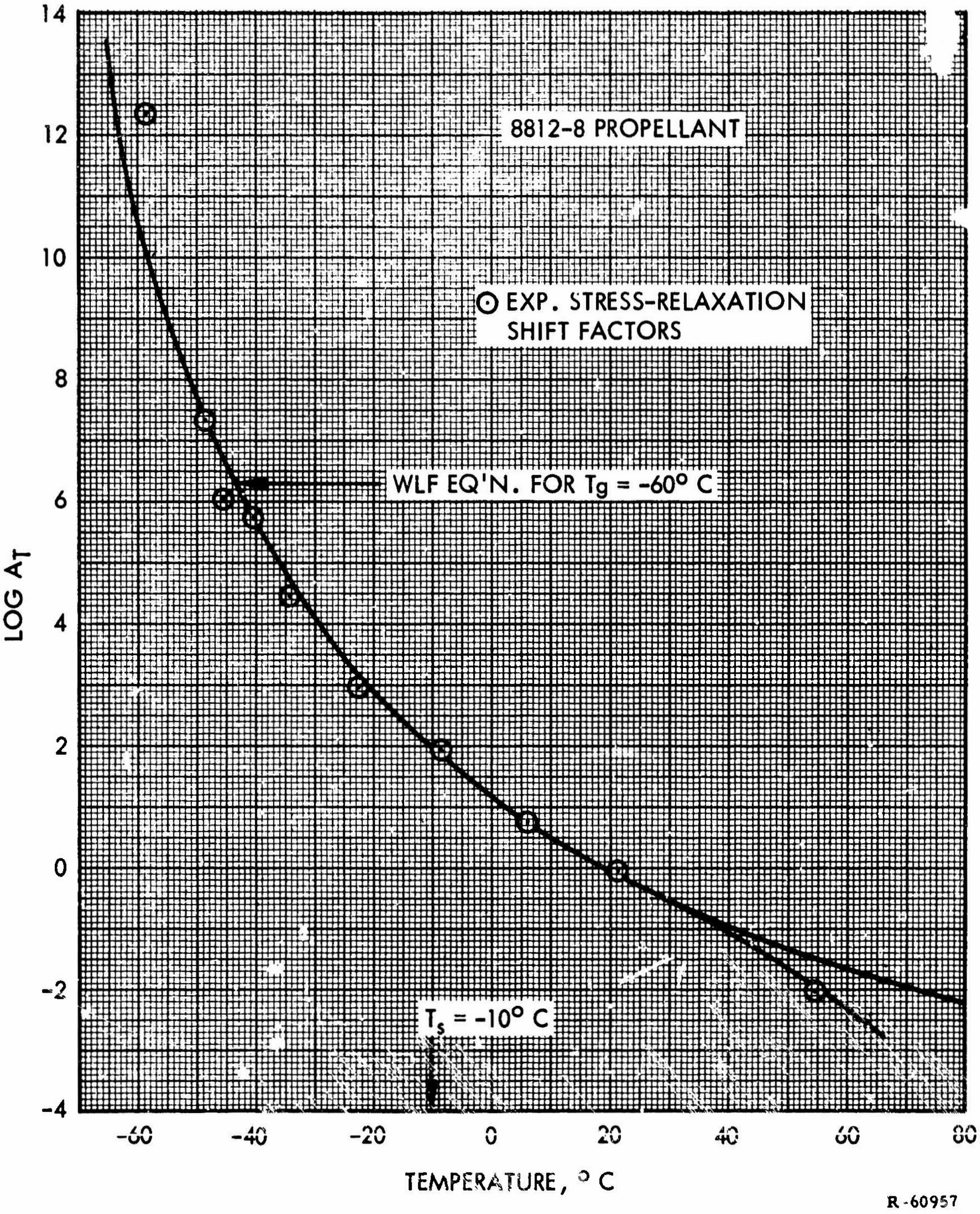


Figure 47. (U) Time Temperature Shift Factors for UTP 8812

TABLE LXIV

(U) TENSILE FAILURE PROPERTIES OF UTP 8812 PROPELLANT

Temperature °F	Crosshead Speed in./min	Crosshead Data Maximum Load σ_m psi	Crosshead Data Maximum Load ϵ_m %	Measured Data Maximum Load σ_m psi	Measured Data Maximum Load ϵ_m %
+128	2.0	17.8	40.8	23.4	31.2
		16.6	34.5	21.6	30.0
		18.1	40.0	23.7	31.1
+76	2.0	24.0	40.4	31.7	31.9
		22.4	39.0	28.4	26.9
		19.1	44.2	25.9	35.5
		23.7	37.5	30.6	29.2
		15.2	48.5	20.2	33.1
		23.9	43.5	31.1	30.0
		24.0	36.9	30.6	27.7
	0.2	12.1	40.5	15.7	30.0
		16.9	32.8	22.0	30.0
		18.5	40.3	24.5	32.5
-70	2.0	386	.9	---	---
		227	1.0	---	---
		238	2.2	---	---
		244	.8	---	---
		271	1.0	---	---
-50	2.0	608	1.1	---	---
		382	2.2	---	---
		458	4.8	---	---
	0.2	1224	2.8	---	---
		1189	2.7	---	---
		1099	2.5	---	---
+40	2.0	29.6	45.8	37.9	28.2
		36.6	43.4	49.0	33.8
-30	2.0	713	8.2	747	4.7
		687	10.0	421	5
		654	11.4	---	---
+10	2.0	72.7	39.0	92.9	27.8
		58.1	59.8	81.3	40.0
		80.4	40.0	104	28.8
-10	2.0	188	20.2	216	15.0
		178	22.0	196	10.0
		168	24.3	196	16.4
	0.2	83.9	38.8	106	26.9
		86.3	36.1	105	21.1
		90.6	36.0	113	25.0

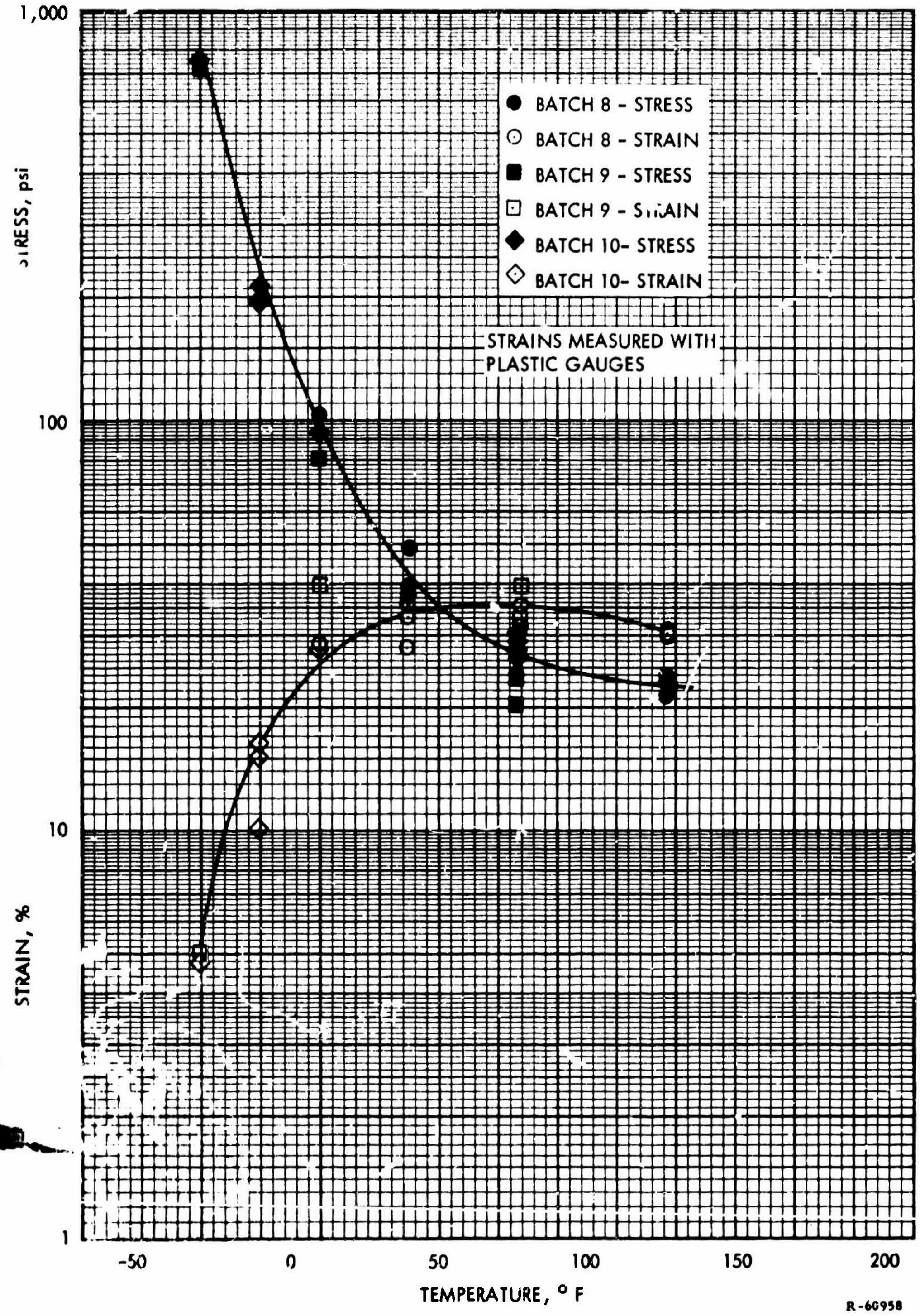


Figure 48. (U) Failure Properties of UTP 8812 at 2 in. /min Crosshead Rate

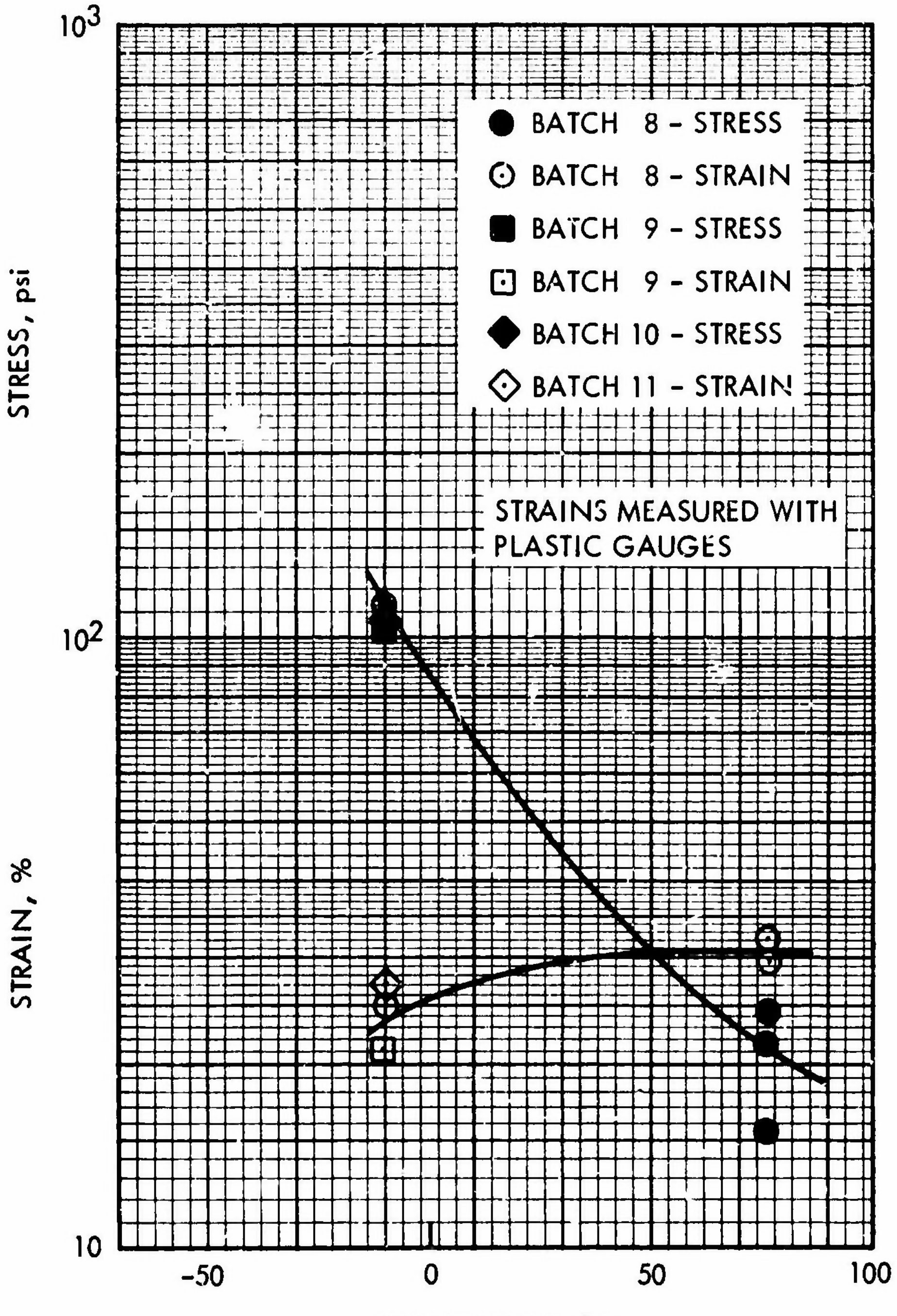


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Figure 49. (U) Failure Properties of UTP 8812 at 0. 2 in. /min Crosshead Rate

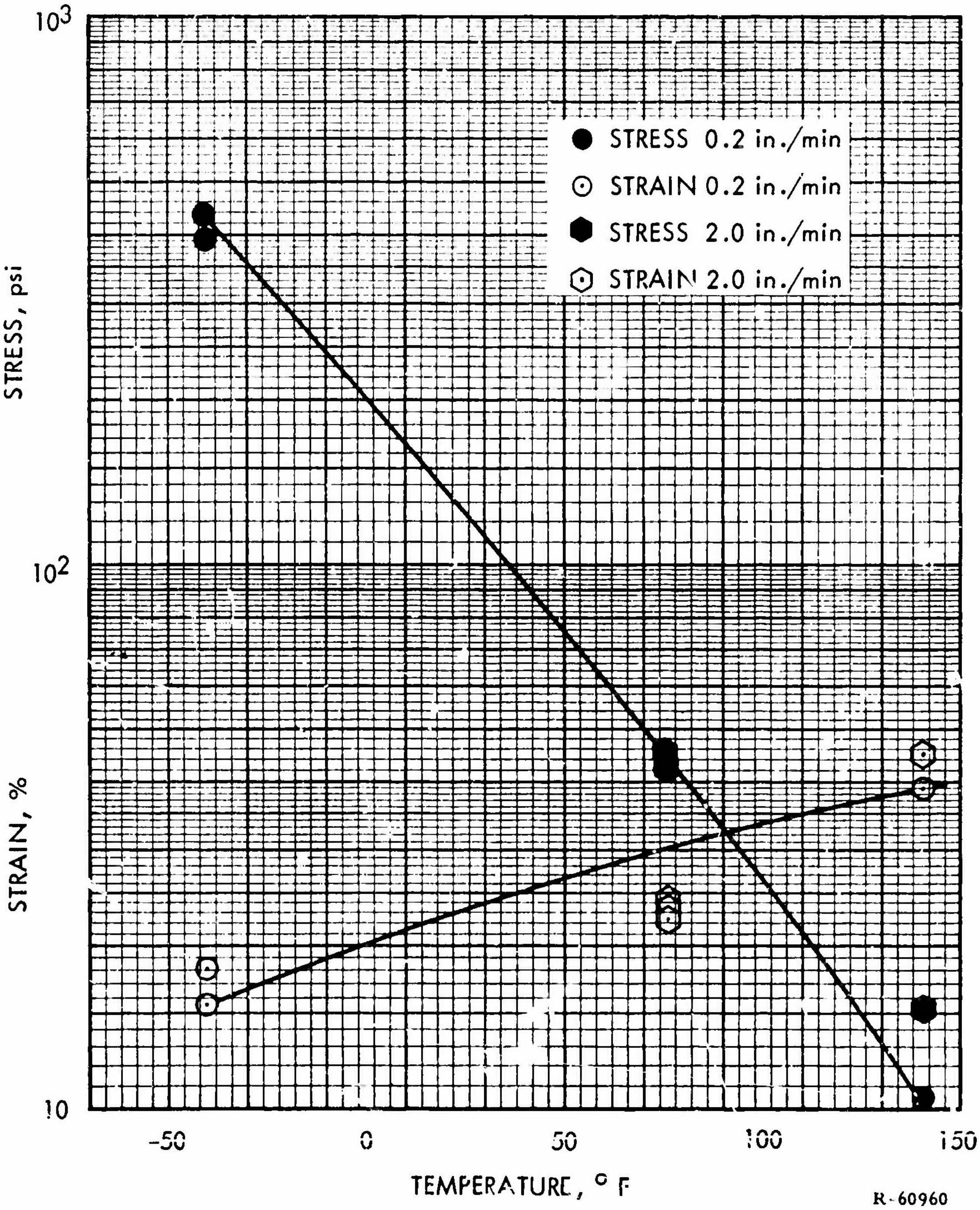

Figure 50. (U) Shear Failure Properties of UTP 8812-10

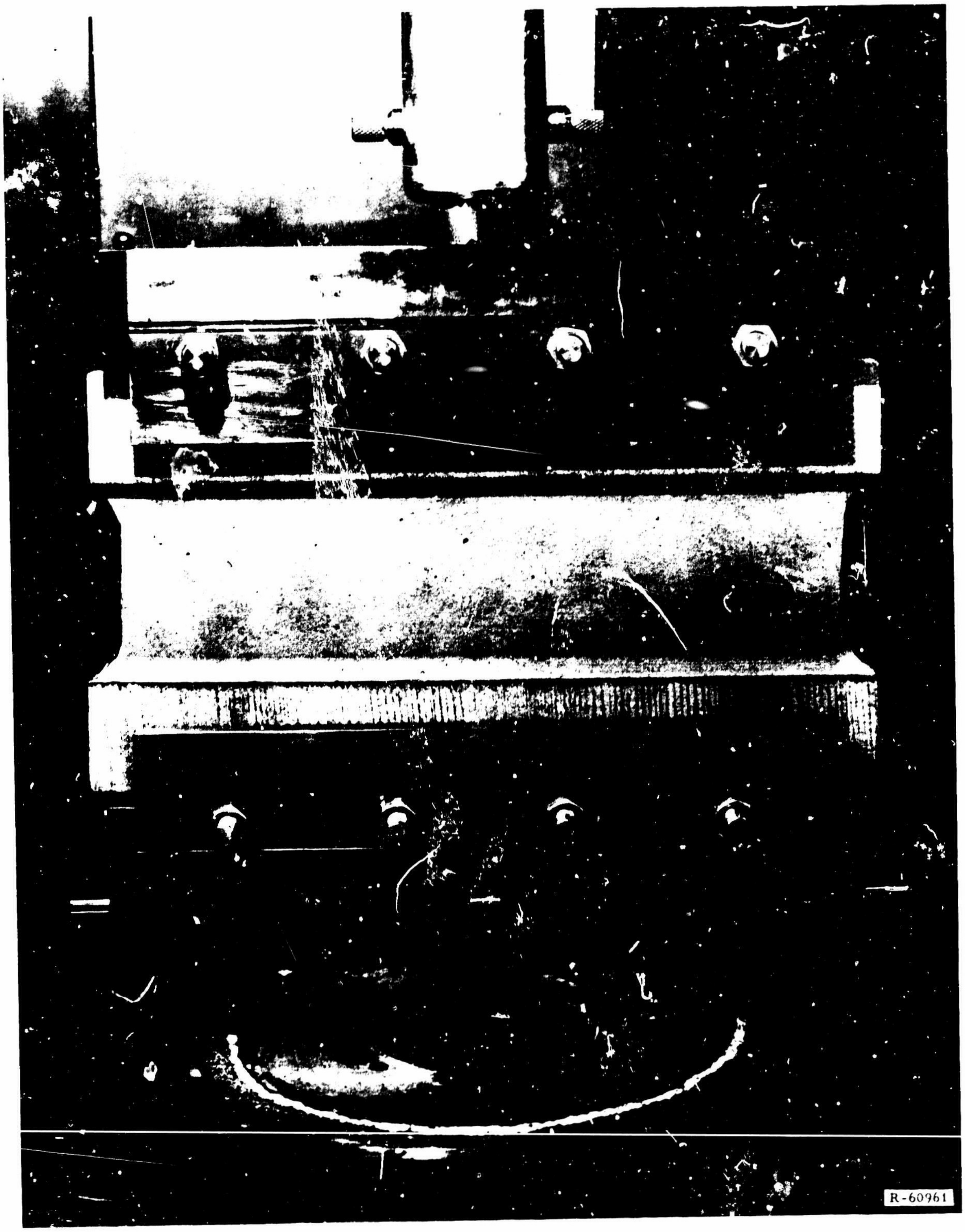

Figure 51. (U) Biaxial Tension Sample

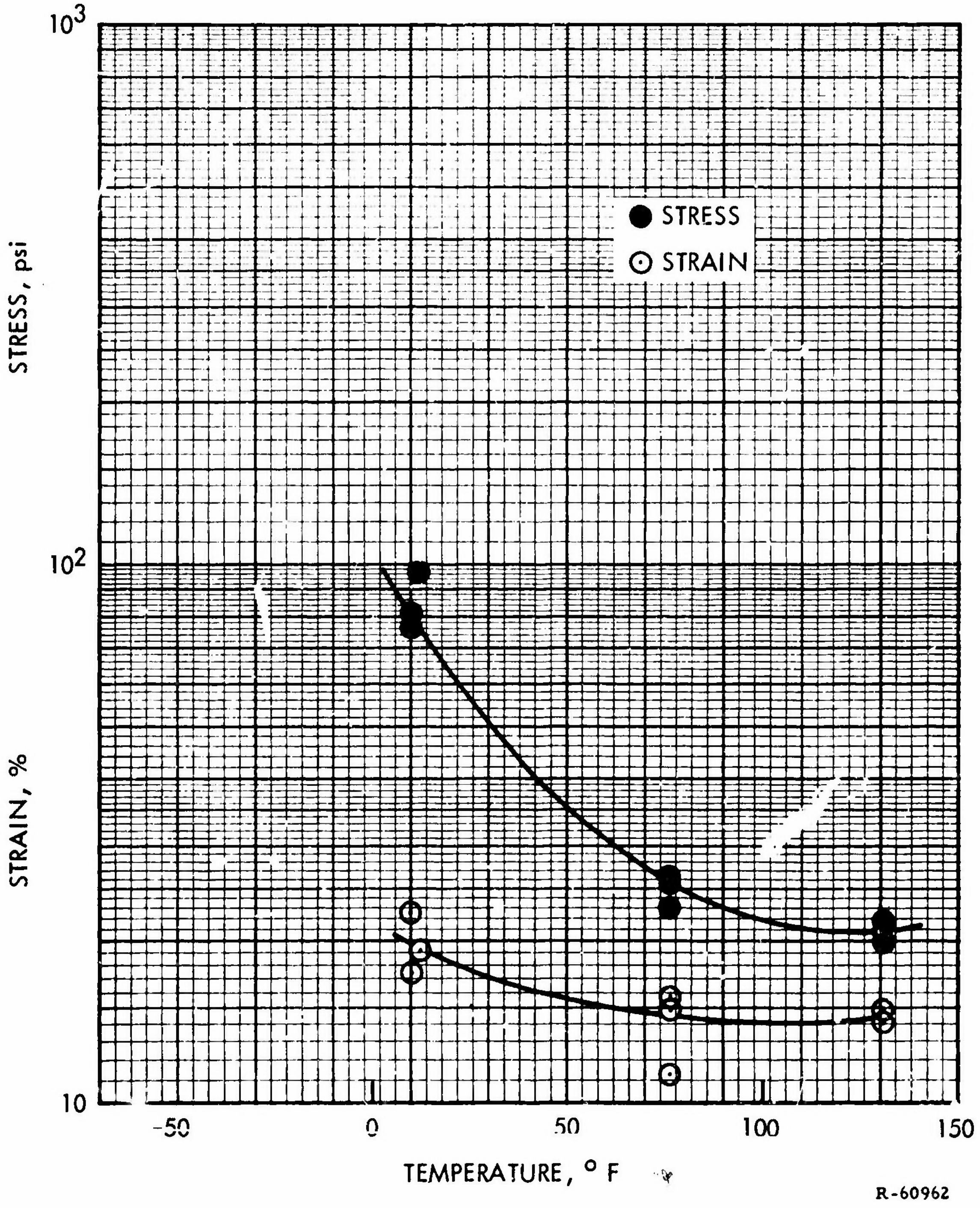

Figure 52. (U) Biaxial Tension Failure Properties of UTP 8812-9 at 2 in. /min Crosshead Rate

(d) Biaxial Compression

(U) Biaxial compression failure properties of UTP 8812 were measured by testing 3.3-in.-diameter circular disks of propellant at a crosshead speed of 0.2 in./min on an Instron Tester. Samples were prepared by casting propellant into circular cartons which were sawed into 1-in.-thick slabs. Failure of the biaxial compression samples occurred by shear cracks along 45° lines from the vertical compression axis. The failure strain values (figure 53) were less than the JANAF tensile data at 76° F. At 10° F the biaxial compression strain values were approximately equal to measured JANAF tensile values.

(e) Dilatational Behavior

(U) Tensile dilatational tests were performed with UTX-8812 propellant in the gas dilatometer shown in figure 54. These tests were made to evaluate Poisson's ratio of the propellant by measuring volume change of the propellant as a function of strain and temperature. A total of five tests were conducted at four temperatures. Volume change measurements for each test are presented in figure 55. The volume change increased slightly with decreasing temperature. Bulk modulus values were determined from the volume change measurements. Poisson's ratio was calculated from these bulk modulus values and the corresponding tensile moduli measured during the constant strain rate tests (figure 56). Poisson's ratio decreased rapidly between 5% and 25% strain and then leveled off prior to rupture of the propellant samples.

(f) Constant Load Properties

(U) Constant load failure properties of UTP 8812 were measured by loading ICRPG samples (class B) on the Instron Tester to a constant load value and maintaining this load with the Instron cam-microswitch system until rupture occurs. These tests were conducted at 72° F, and the strain was measured using plastic extensometers. Data presented in figure 57 show an approximate stress rupture asymptote of 13 psi and a strain asymptote of 20%. Batch 20 propellant used for these tests contained Mg-doped LMH-1 material.

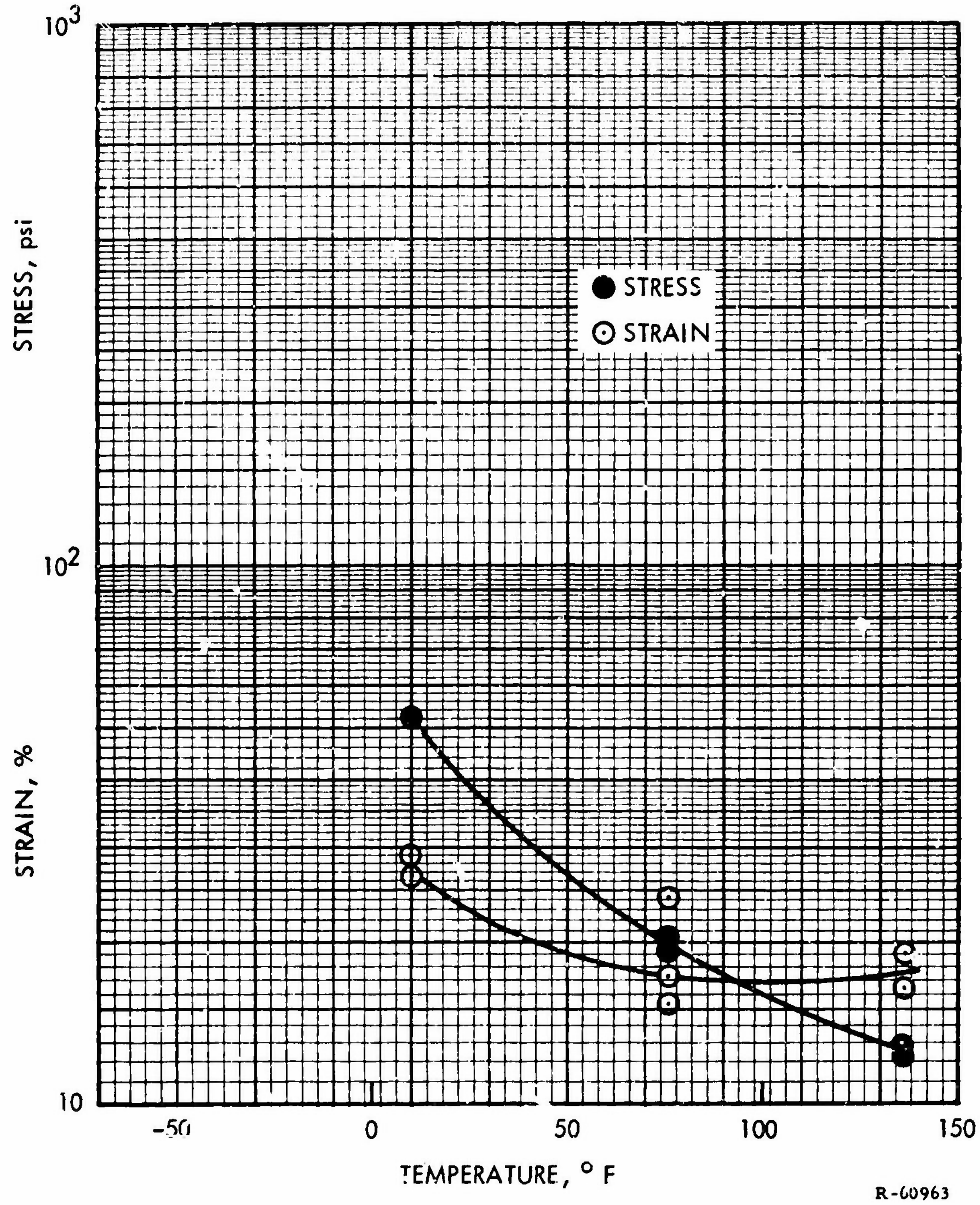

Figure 53. (U) Biaxial Compression Failure Properties of UTP 8812 at 0. 2 in. /min Crosshead Rate

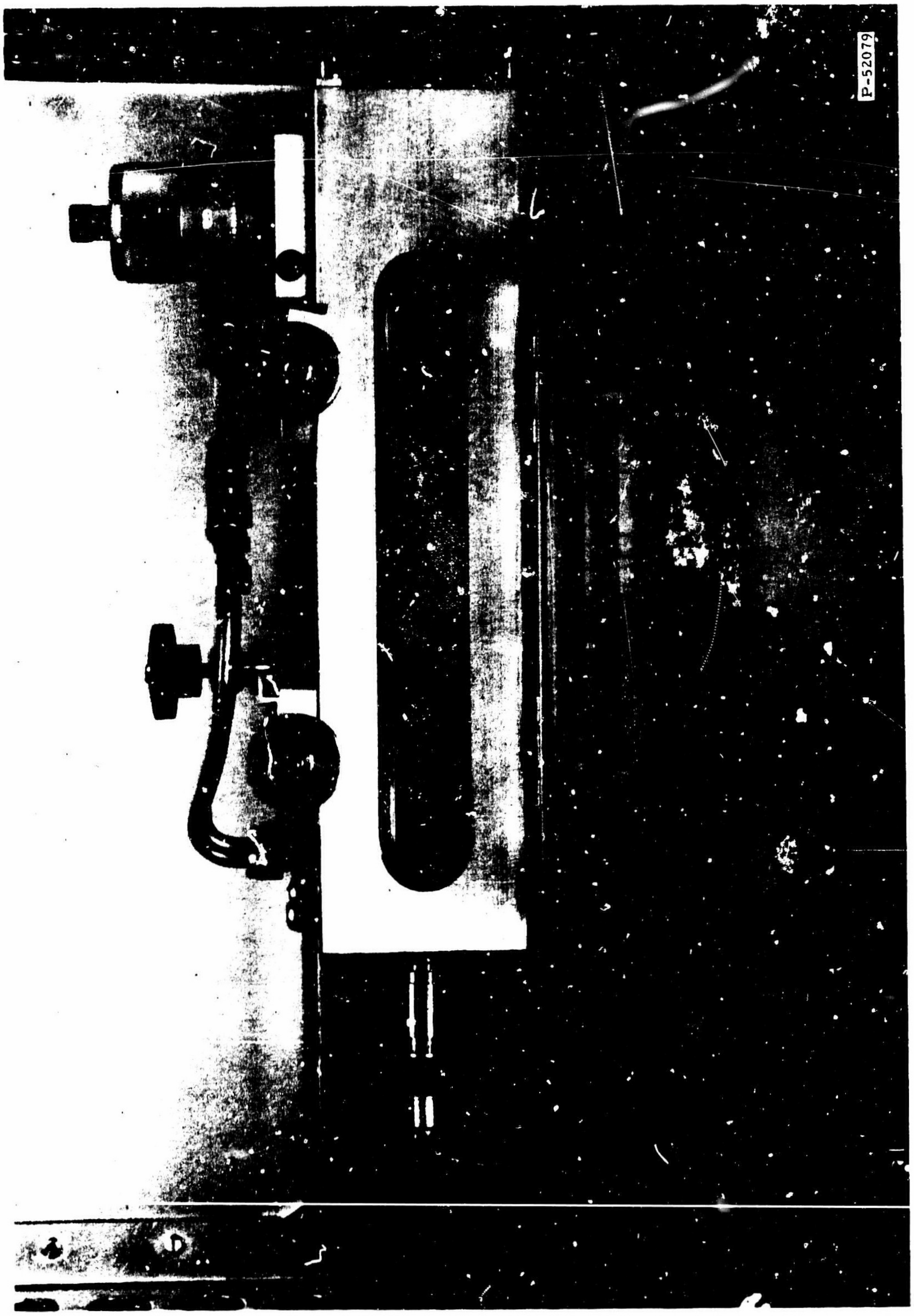

Figure 54. (U) UTC Gas Dilatometer

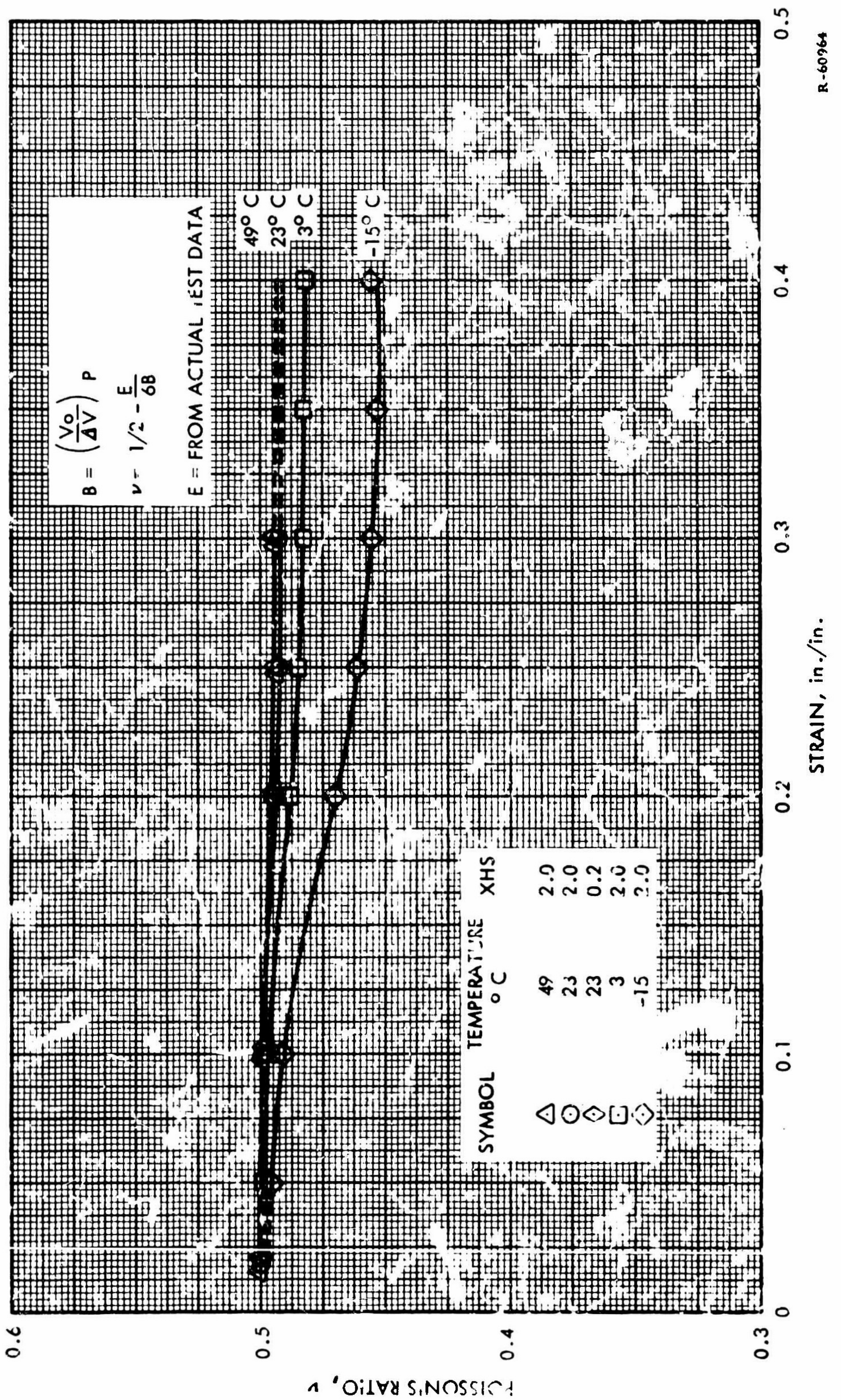

Figure 55. (U) Poisson's Ratio vs Strain for UTP 8812-8 Propellant

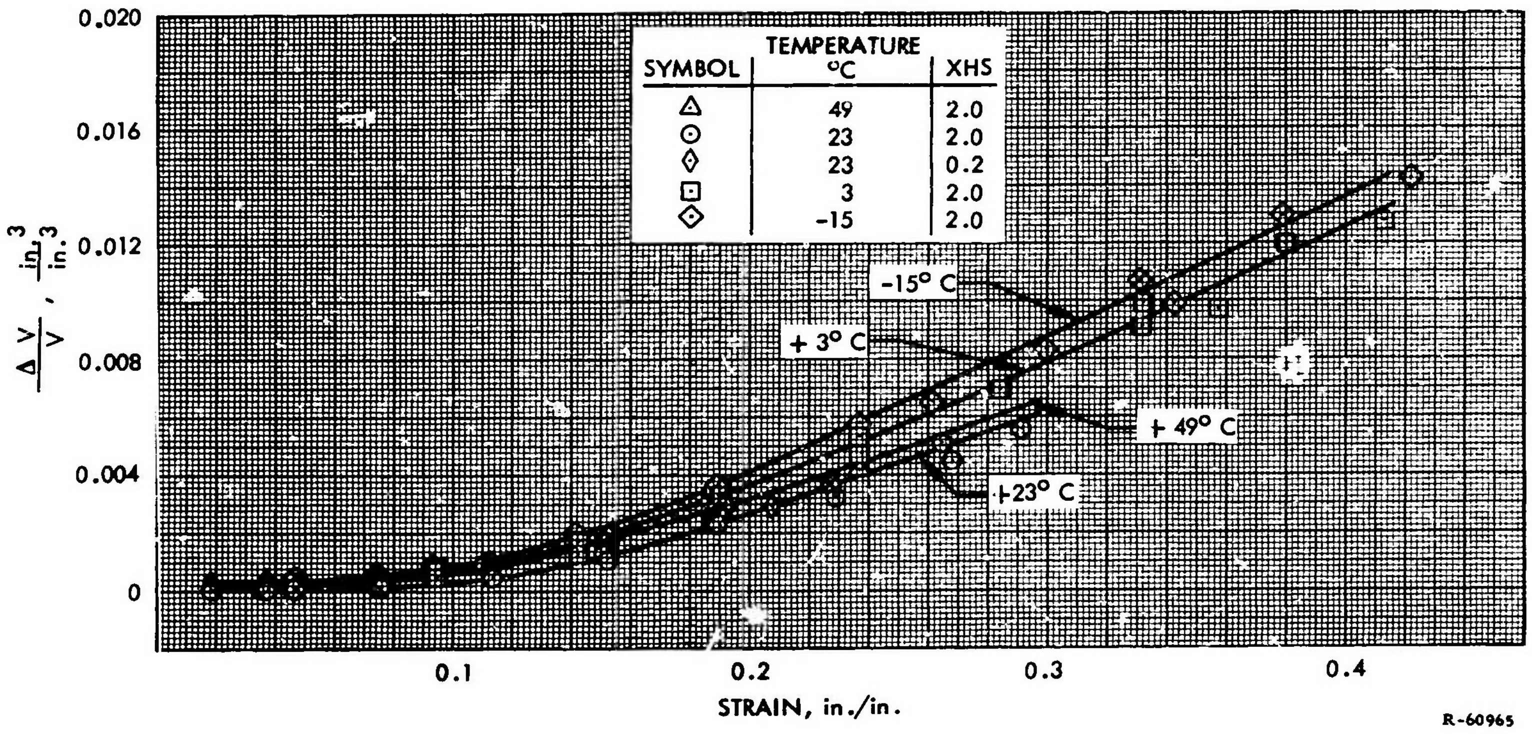

Figure 56. (U) Volume Change Measurement vs Strain for UTP 8812-8 Propellant

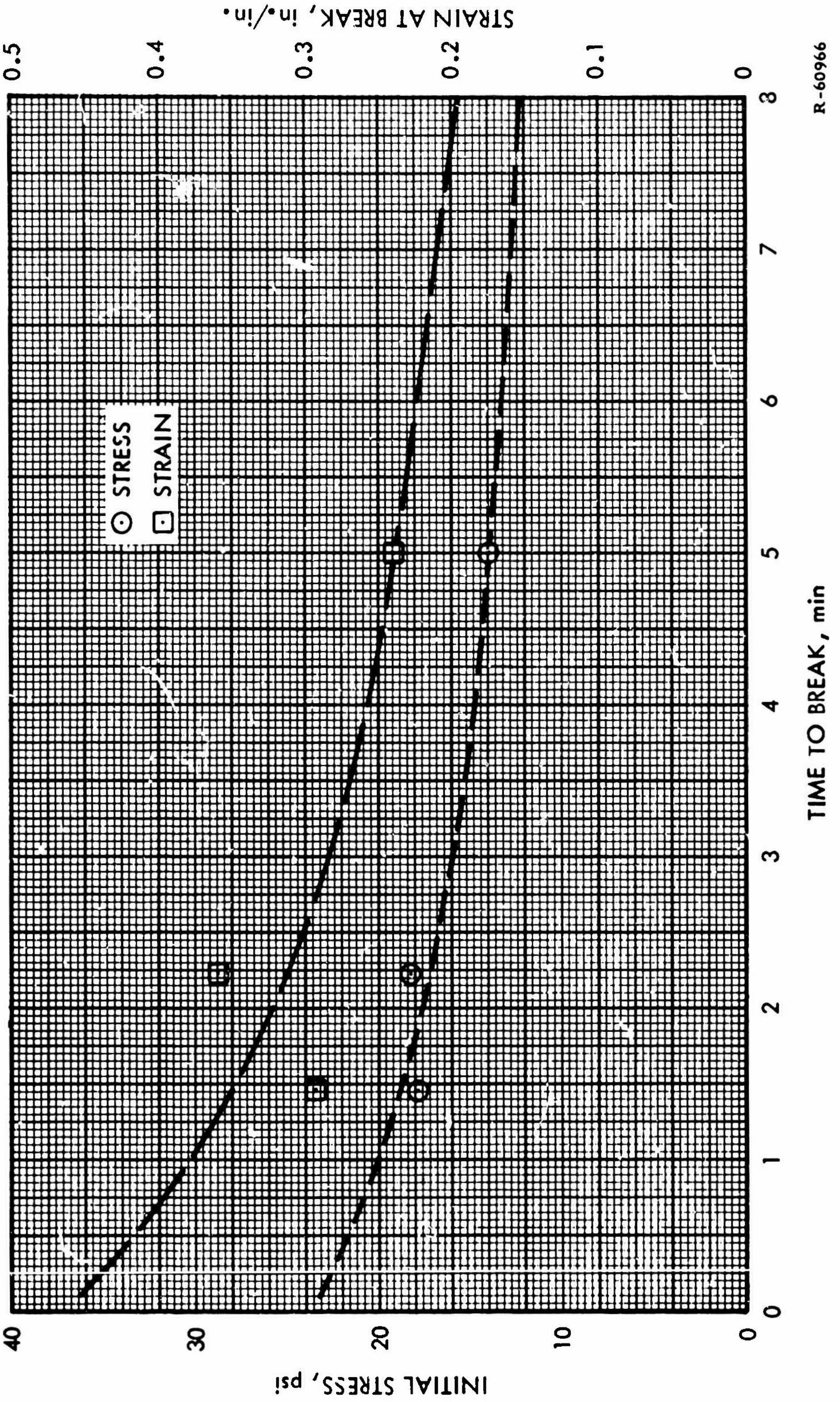

Figure 57. (U) Constant Load Properties of UTP 8812-20

(g) Steel Case Analogue Motor Tests

(U) Propellant thermal endurance limits were investigated with steel case analogue motors. Both UTP 8812 and a reference batch of UTP 5143 propellant were tested in 3-in.-diameter by 8.2 in. long tubes. Circular port grains of each propellant type were subjected to a constant cooling rate of approximately 20° F/day. The reference data for four different circular port geometries and UTP 5143 propellant (CT-3 type) is presented in figure 58. Bore strain measurements taken with the modified Mueller gage shown in figure 59 are displayed for different test temperatures. Two motors of each grain geometry were selected to evaluate strain versus temperature behavior, but the test freezer malfunctioned at -50° F causing the motors to drop below -100° F and therefore crack. Borescope examination of these motors showed a brittle-type failure with numerous small cracks throughout each grain. None of these motors had cracks at -60° F as the JANAF strain limits would indicate.

(U) Six LMH-1 analogue motors were tested with the referenced UTP 5143 tests, but the UTP 8812 propellant swelled prior to testing causing a decrease in port diameter. Four motors without Mg-doped LMH-1 swelled excessively making it impossible to take bore measurements. The two Mg-doped LMH-1 motors looked better, but swelling had reduced the internal strain well below that expected. Data for these tests are presented in figure 60. These grains were not cracked at -60° F before the freezer malfunctioned, but they exhibited brittle fracture when inspected at -100° F. The analogue motor tests with the LMH-1 propellant show that swollen grains reduce grain strain and therefore extend low temperature thermal failure limits of a grain design. However, the swelling behavior makes grain strain predictions impossible.

(h) Fiberglass Analogue Motor Evaluation

(U) Fifteen fiberglass analogue motor tests were conducted with UTP 8812 and UTP 5143 propellants. The fiberglass analogue motor case used for propellant evaluation is shown in figure 61. The case is a 54°

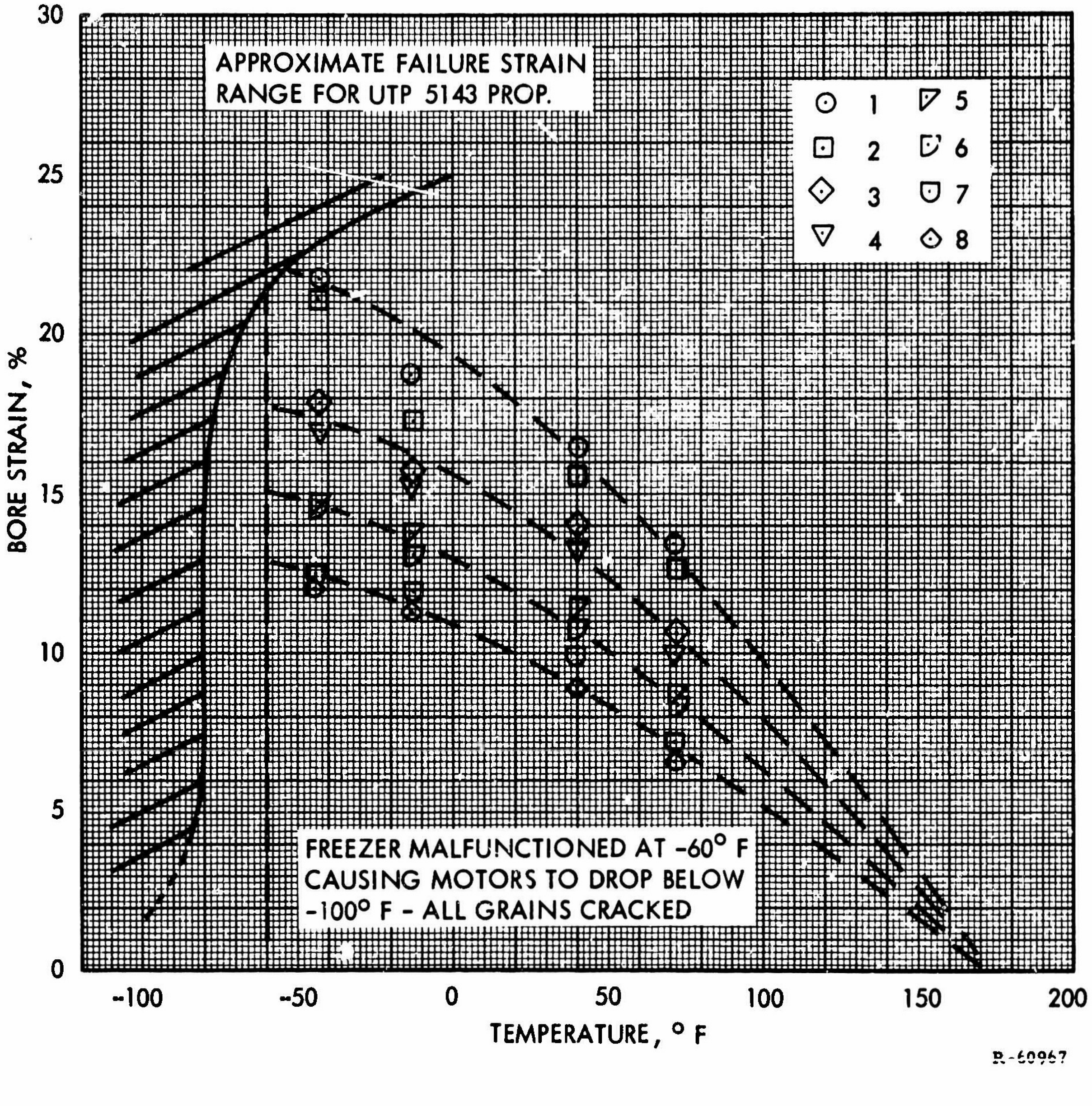

Figure 58. (U) Analogue Motor Data for UTP 5143 Propellant

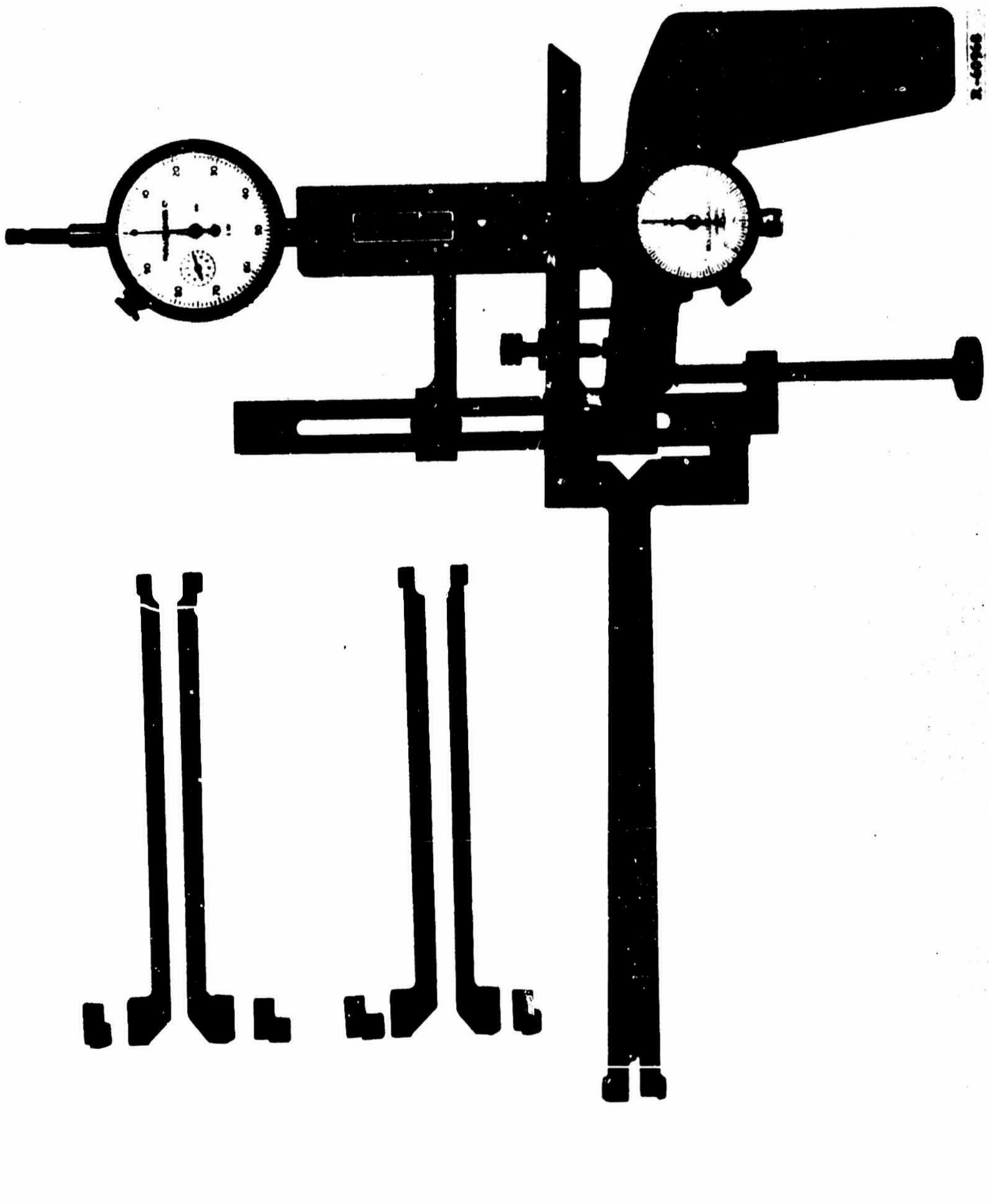

Figure 59. (U) Modified Mueller Gage

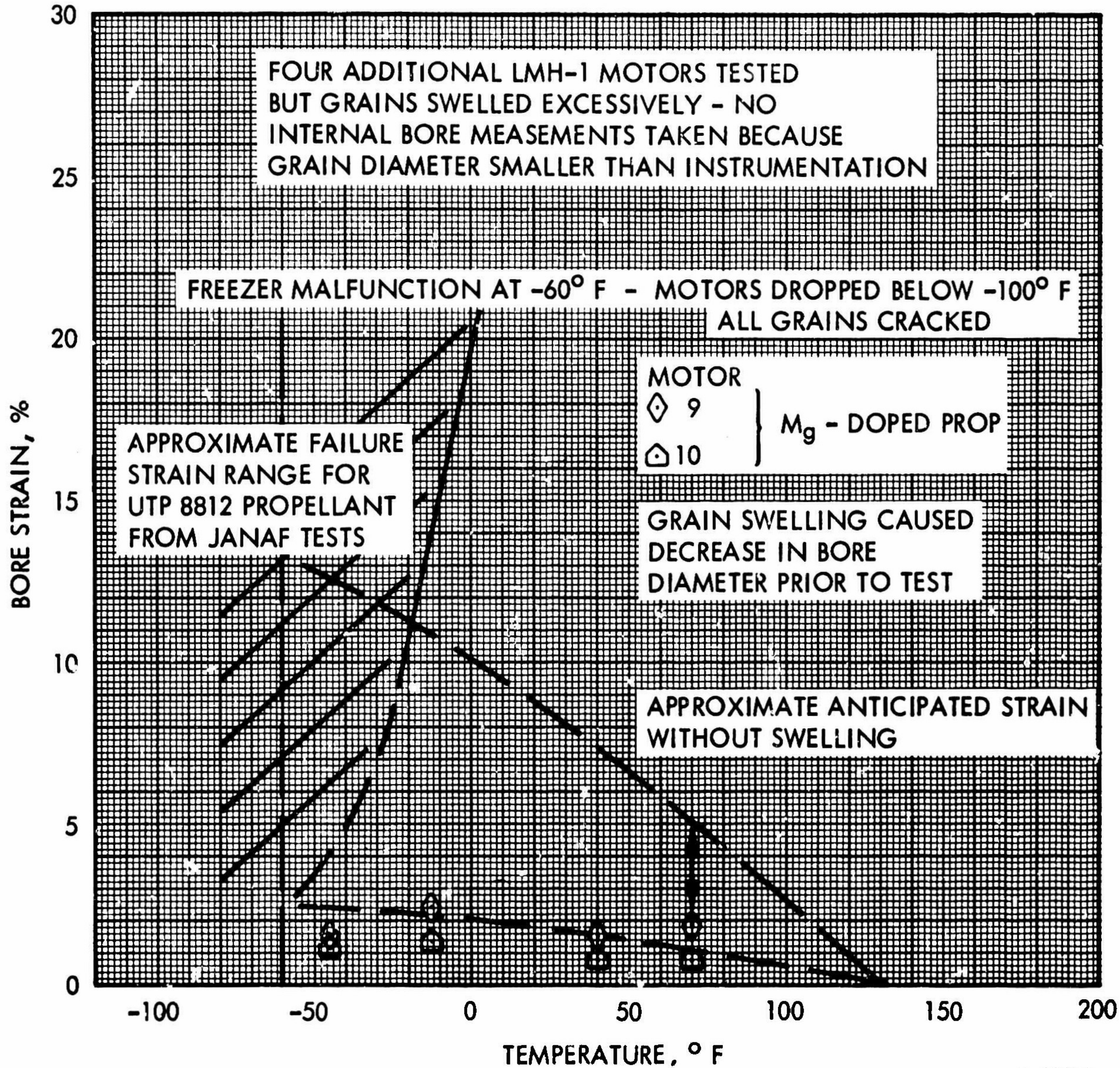

Figure 60. (U) Analogue Motor Data for UTP 8812 Propellant

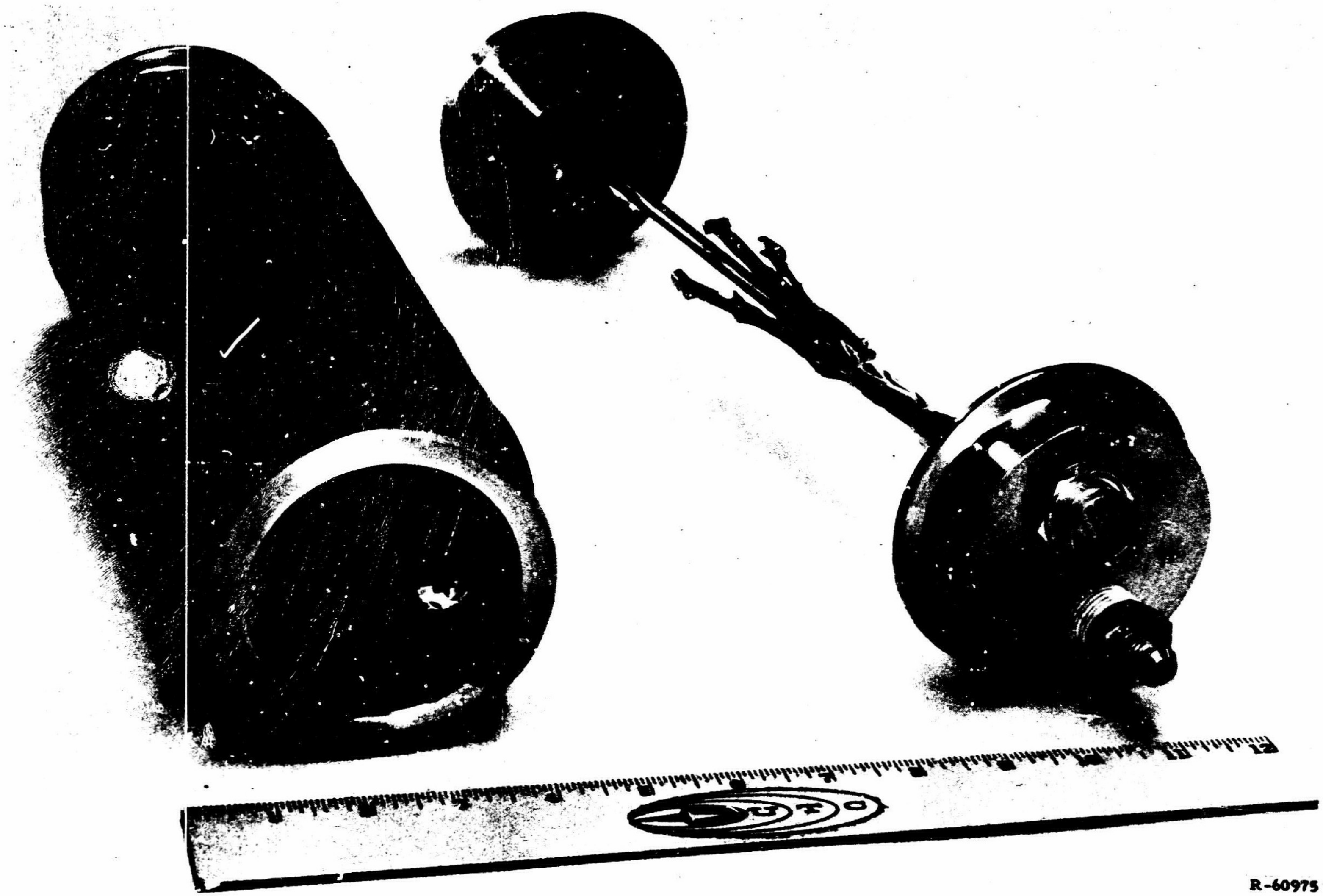

Figure 61. (U) Fiberglass Motor and End Closures with Internal Bore Gage

helically wound fiberglass container with a case thickness of 0.030 in. and a flange-flange dimension of 13.5 in. Fiberglass analogue motors were cast with circular port grains of two different web thicknesses.

(U) The hydraulic system used to rapidly pressurize the fiberglass analogue motors is shown in figure 62. A diagram of the entire flow system is shown in figure 63. Pressure is obtained with a 5,000 psi hydraulic pump which loads a 2-1/2 gal. accumulator tank at or above the desired motor pressure. When the high-speed solenoid valve opens, oil flows into the motor at high rates through a 3/4-in. discharge line. The desired motor test pressure is set on a nitrogen-filled surge tank located behind a one-way check valve. When the motor pressure exceeds the surge tank pressure, the check valve opens and additional oil flows into the surge tank instead of the fiberglass motor. The maximum flow rate of the 3/4-in. -line system is 60 gal./min which will allow pressurization of the fiberglass motors in approximately 50 msec using a special 3/4-in. -orifice high-response solenoid valve. The motor is attached to the hydraulic system with a non-leak quick disconnect which is located inside a one-in. -thick plexiglass conditioning box. Tests can be conducted at any desired temperature using a portable temperature control source.

(U) The propellant grain dimensions were measured prior to pressure testing using the modified Mueller gage shown in figure 59. This instrument has a direct dial gage readout with 0.001-in. accuracy. The major difficulty with propellant measurements is that contacts may sink into the soft material. This is overcome by using large contact surfaces which are matched to or slightly less than the propellant port diameter. The modified Mueller gage has removable contact points which have been specifically designed to eliminate propellant indentation. Contacts are 1/4-in. long and different contact radii are available for each specific grain dimension.

(U) Failure strains under high rate pressurization conditions are measured using strain gage cantilevered beams (figure 64). Four beams with both compression

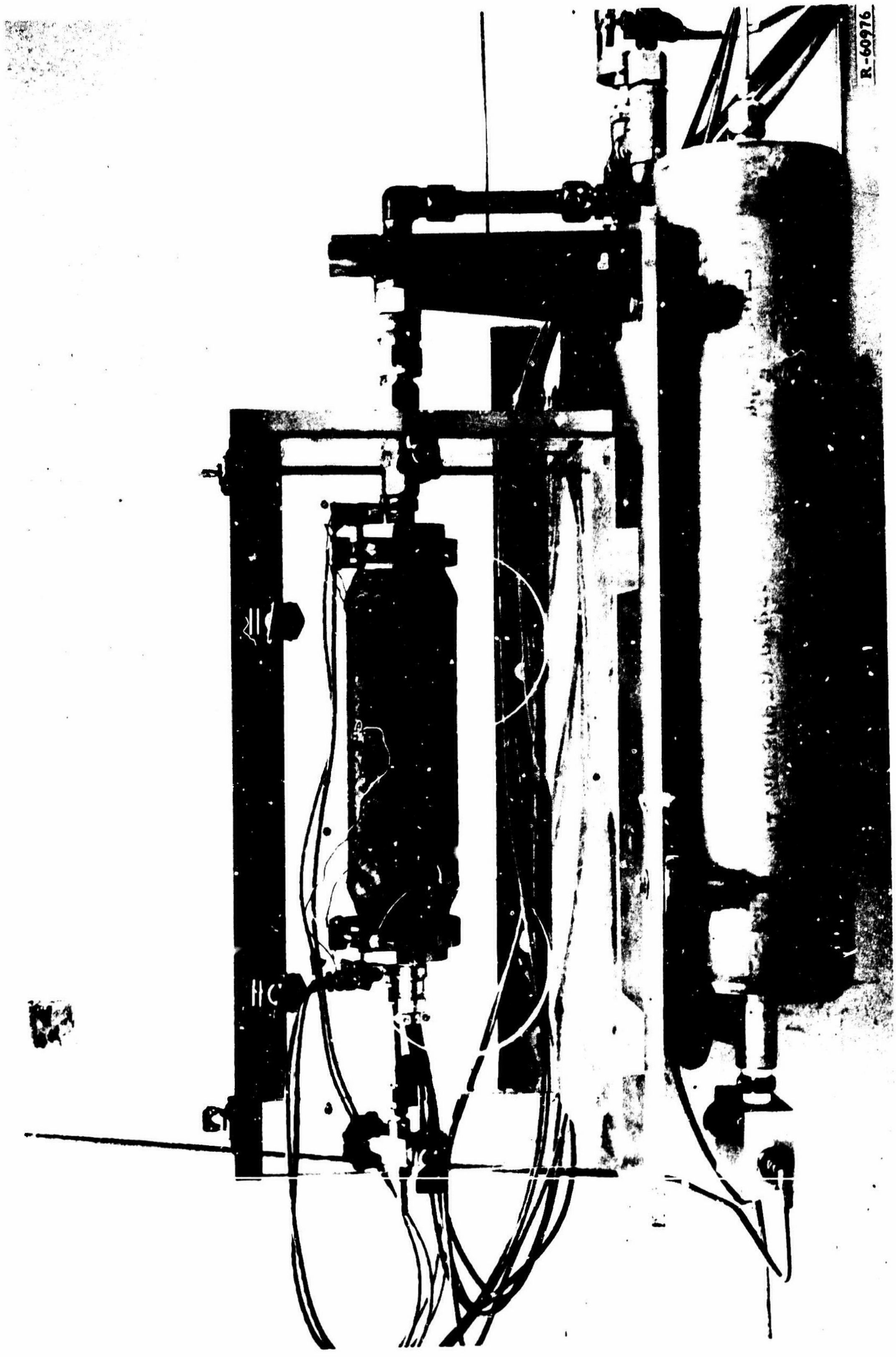

Figure 62. (U) Hydraulic Pressurization Equipment

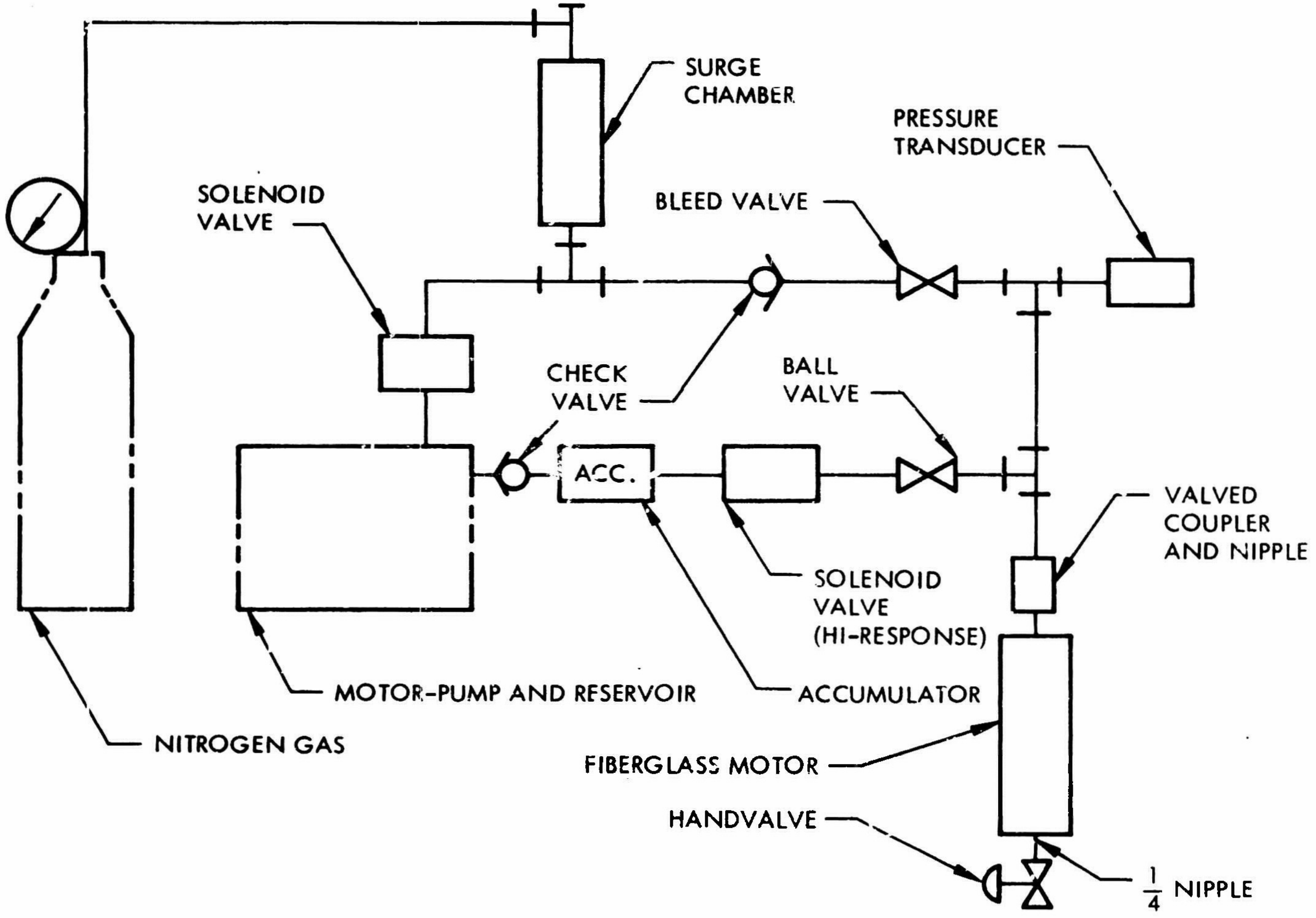

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Figure 63. (U) Diagram of Hydraulic Pressurization System

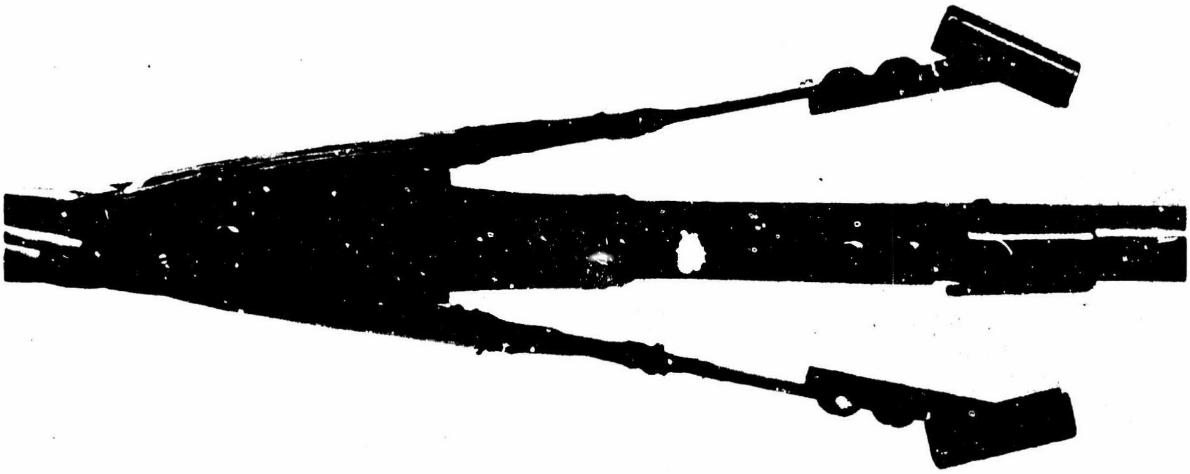

Figure 64. (U) Internal Bore Gages

and tension strain gages are mounted on a rigid rod which extends through the entire length of the motor. Contact points are removable and pivoted on the end of each cantilevered beam. Cantilevered beam thickness, width, length, initial deformation, and contact geometry were selected to maintain a contact stress of 1 to 2 psi. This is sufficient to maintain surface contact during motor pressurization and yet small enough to minimize grain compression or indentation.

(U) The test procedure was:

1. Install beam assembly at center of grain

2. Load motor with hydraulic oil by gravity flow until air is entirely replaced with inert fluid

3. Temperature condition motor for 1-1/2 hr

4. Load accumulator tank with oil to approximate pressure of 100 to 400 psi above the desired pressure

5. Set desired test pressure on surge tank

6. Test-trip high-speed solenoid on and off quickly (test time approximately 0.25 sec)

7. After test is completed, open discharge solenoid valve and let oil return to the pump sump

8. After motor pressure has returned to zero, inspect grain surface ruptures.

(U) Six fiberglass analogue motors were tested with a CT-3 propellant at 150° F, 76° F, and 0° F with propellant grains rupturing at each test temperature. Two motors, each of different web thickness, were tested at 150° F, 76° F, and 0° F. Table LXV lists each port radius, test temperature, ramp time, maximum pressure, approximate propellant hoop strain, and final grain condition. Large grain ruptures on test No. 2 and No. 4 were detectable by abnormal cantilevered beam data. The smaller grain ruptures on test No. 6 (3 — 1 in. cracks) were not detectable on the oscillograph data but were apparent on visual examination of the grain.

(U) The maximum propellant hoop strains at each temperature together with the approximate laboratory propellant failure strains are presented in figure 65. This figure shows that the three motors which ruptured all cracked in a strain range anticipated from laboratory tensile data, and the three grains which did not crack were in the safe strain range.

(C) Nine fiberglass analogue motors were tested with UTP 8812 propellant at 120° F, 76° F, and +4° F without grain cracking. Three motors were tested at each test temperature. Table LXVI lists each grain geometry and measured test parameters. The motors used for these tests did not contain Mg-doped LMH-1 material and experienced various degrees of swelling. The bore strain measurements are extremely high for motors No. 7 and No. 11 because strain is calculated using the measured swollen bore diameter. The 9 LMH-1 propellant analogue motor test geometries and measured strains indicate that this test method is extremely sensitive to propellant volume change.

TABLE LXV

(U) ANALOGUE MOTOR TEST DATA FOR UTP 5143 PROPELLANT

Test	Case Temperature ° F	Port Radius in.	Case Radius in.	Ramp Time sec	Peak Pressure %	Case Strain %	Propellant Strain %	Grain Condition
1	76	0.524	2.00	0.070	750	---	10.9 to 12.3	Good
2	76	0.473	2.00	0.088	810	0.91	22.8 to 27.0	4 in. long crack
3	150	0.526	2.00	0.070	865	---	12.8 to 16.4	Good
4	150	0.466	2.00	0.085	760	---	22.3 to 29.0	3 in. long crack
5	0	0.527	2.00	0.050	845	0.35 to 0.48	7.3 to 7.7	Good
6	0	0.471	2.00	0.050	895	0.42 to 0.44	14.7 to 18.1	3 to 1 in. long cracks

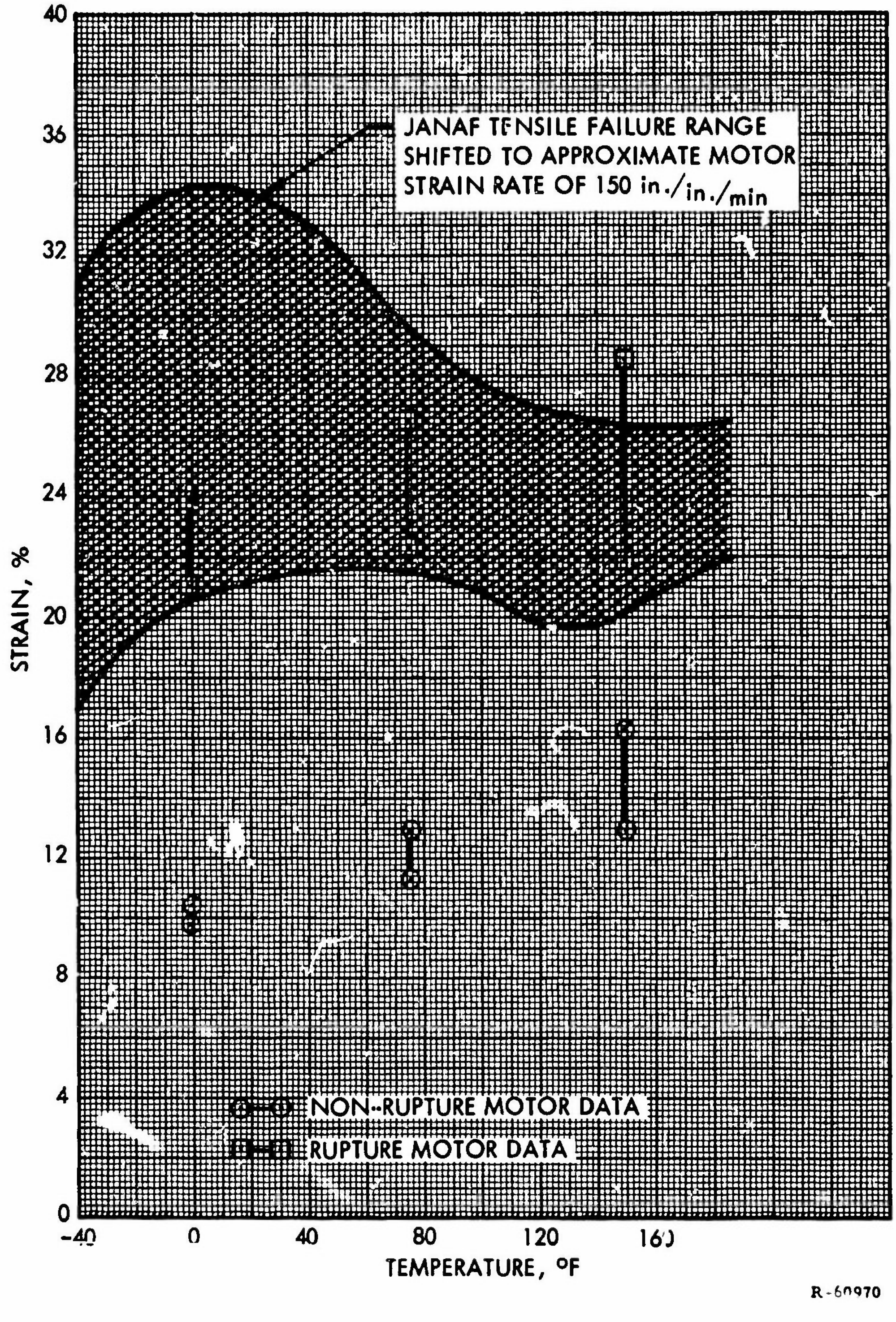

Figure 65. (U) Fiberglass Motor Hoop Strains and Laboratory Tensile Failure Limits for UTP 5143 Propellant

TABLE LXVI

(U) LMH-1 FIBERGLASS MOTOR DATA*

Test	Temperature °F	Port Diameter inches	Ramp Time sec	Peak Pressure psi	Case Strain %	Propellant Strain† %	Grain Condition After Test
7	76	0.629 center 0.963 outer edge	0.103	610	0.678-0.776	66.3-87.7	good
8	76	1.118 center	0.092	690	0.860	18.3-30.4	good
9	76	0.954 outside edge inside very swollen	0.198	692	0.655-.830	Too swollen for measurements	good
10	120	1.103 center 1.202 outside edge	0.103	725	---	24.3-25.6	good
11	120	0.672 center 0.871 outside edge	0.091	710	0.423-0.758	57.2-75.7	good
12	120	0.540 center 0.912 outside edge	0.098	586	0.492	Too swollen for measurements	good
13	+4	swollen - no measurements	0.050	855	.212	Too swollen for measurements	good
14	+4	swollen - no measurements	0.112	860	---	Too swollen for measurements	good
15	+4	swollen - no measurements	0.088	836	---	Too swollen for measurements	good

* All fiberglass analogue motors cast with non-Mg-doped LMH-1 material (C)

† Hoop strain calculated using measured bore diameter.
Actual strain value would be muchless than shown for swollen grains.

(i) Physical Properties

Density

(U) Laboratory density measurements for UTP 8812 propellant are presented in table LXVII. The laboratory density values are generally lower than bulk values because the small laboratory samples are able to absorb sufficient amounts of the reference material Kel-F into the surface pores and effectively reduce the density.

TABLE LXVII

(U) DENSITY VALUES FOR UTP 8812 PROPELLANT

Batch	Density g/cc
8	1.511, 1.495, 1.504
10	1.499, 1.499

(j) Hazard Characterization

(C) Laboratory hazard tests have been conducted to evaluate the potential hazards inherent in the handling, processing, and testing of AlH_3-containing propellants in the laboratory and pilot plant. In addition, an explosive hazard classification of UTP 8812, the propellant chosen for scaleup, has been conducted in accordance with the "Explosive Hazard Classification Procedure," Technical Order 11A-1-47, phase I, dated 31 July 1962.

(C) The laboratory hazard classification tests include those measuring sensitivity to impact, friction, spark, and the autoignition temperature. The results of these tests show the AlH_3 propellant to be more sensitive to impact and friction than other composite propellants.

1. Laboratory Hazard Classification

a. Impact Sensitivity

(U) Impact sensitivity of UTC propellants is determined with an Olin Mathieson impact tester with variable weight and variable drop height. The apparatus has a striker diameter of 0.8 cm, a maximum drop height of 50 cm, and a variable weight of 1 to 6 kg (a 2-kg weight is standard). A Bruceton-type procedure is used to establish a 50% and 0% initiation drop height for the weight.

(U) The impact sensitivity value determined for cured UTP 8812 propellant is 6.0 kg/cm.

b. Spark Sensitivity

(U) Propellant spark sensitivity is determined by discharging a spark from a needle point through the sample to a metal sample support. Electrostatic discharge energies of 0.001 to 10 joules with corresponding voltages from 3,000 to 7,000 are applied to samples of 0.025-in. thickness and 0.175-in. diameter. The spark sensitivity value for UTP 8812 propellant is above 10 joules.

c. Friction Sensitivity

(U) Friction sensitivity is measured using an Esso (screw) friction tester. The Esso screw friction tester was designed to determine the relative hazards of handling high-sensitivity materials which cannot be evaluated on pendulum friction testers. Samples are placed between the two plattens on the test apparatus, figure 66, and the top screw is slowly lowered until the rotating torque exceeds 60 ft/lb. The severity of the test is modified by adding diamond or grit glass. Three categories of friction sensitivity are considered:

Category	No. Grit	Glass 5.5 Moh Hardware	Diamond 10 Moh Hardware	Remarks
I	0[*]	0	0	Manual handling OK
II	0	+[†]	+	Remote handling advised
III	+	+	+	High sensitivity to friction

[*] 0 — no ignition
[†] + — ignition

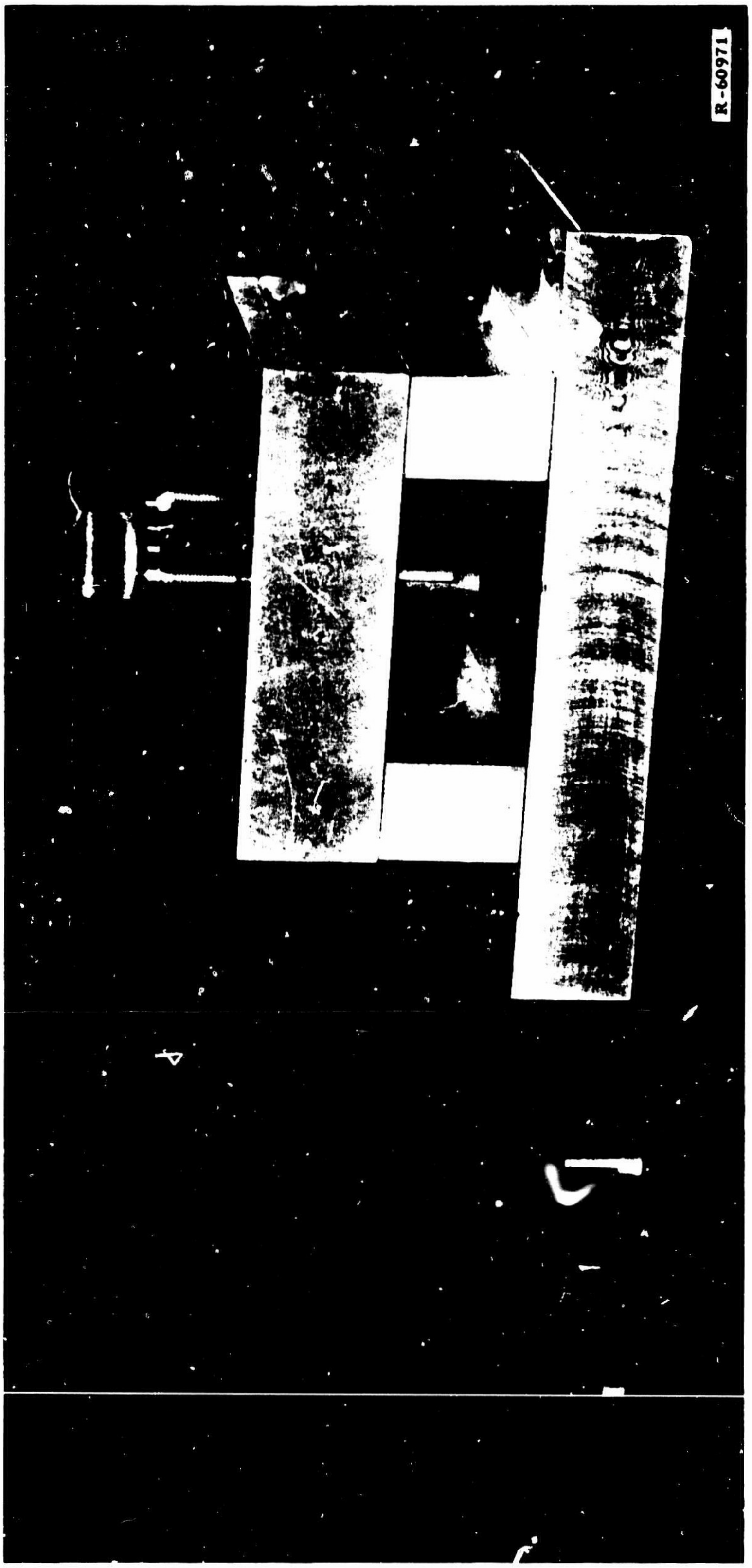

Figure 66. (U) Esso Friction Tester

Cured UTP-8812 propellant ignited with bare platens and is therefore extremely friction sensitive.

d. Autoignition

(U) Autoignition temperatures are determined in a Wood's metal bath. Samples of 0.175-in. diameter and 0.05-in. thickness are dropped into a stainless-steel tube inserted in the Wood's metal bath, and time required for ignition is recorded. Four temperatures (starting at 500° F) with five tests at each temperature are performed to establish an approximate time-temperature ignition boundary. A 10-sec ignition temperature of 670° F and a 30-sec ignition temperature of 558° F were determined for UTP 8812 propellant. These autoignition temperatures for LMH-1 propellant show it to be similar to other composite propellants. This test is specifically limited to short-time, high-temperature exposure conditions as opposed to storage stability.

2. Phase I — Explosive Hazard Classification Tests

(U) The phase I explosive hazards tests conducted in accordance with TO 11A-1-47 showed UTP 8812 propellant to be similar to other composite propellants. These tests expose the propellant to heat, flame, and mild shock.

a. Detonation

(U) Detonation tests were performed with UTP 8812 propellant by placing 2-in. propellant cubes in contact with No. 8 blasting caps. The cubes were fragmented, but ignition did not occur.

b. Ignition and Unconfined Burning

(U) Ignition and unconfined burning tests with UTP 8812 propellant were normal for composite propellant. The 1-in cube burned 8 sec, and the 2-in. cubes burned an average of 14 sec.

c. Differential Thermal Analysis

(U) A DTA was run on UTP 8812, and the thermogram is presented in figure 67.

d. Thermal Stability Tests

(U) Thermal stability tests were run with 1-in. and 2-in. cubes of UTP 8812. The propellant did not ignite and appeared to be essentially unaffected by the test.

e. Impact Tests

(U) Impact tests were conducted on UTP 8812 propellant with the Bureau of Explosives impact tester according to the procedures in TO 11A-1-47. Ten ignitions were recorded at both the 3-3/4-in. and 10-in. drop height.

3. Card-Gap Tests

(U) Card gap tests were conducted on the UTP 8812 propellant to evaluate sensitivity in a confined condition. Data presented in table LXVIII shows a card-gap value of approximately 85 cards.

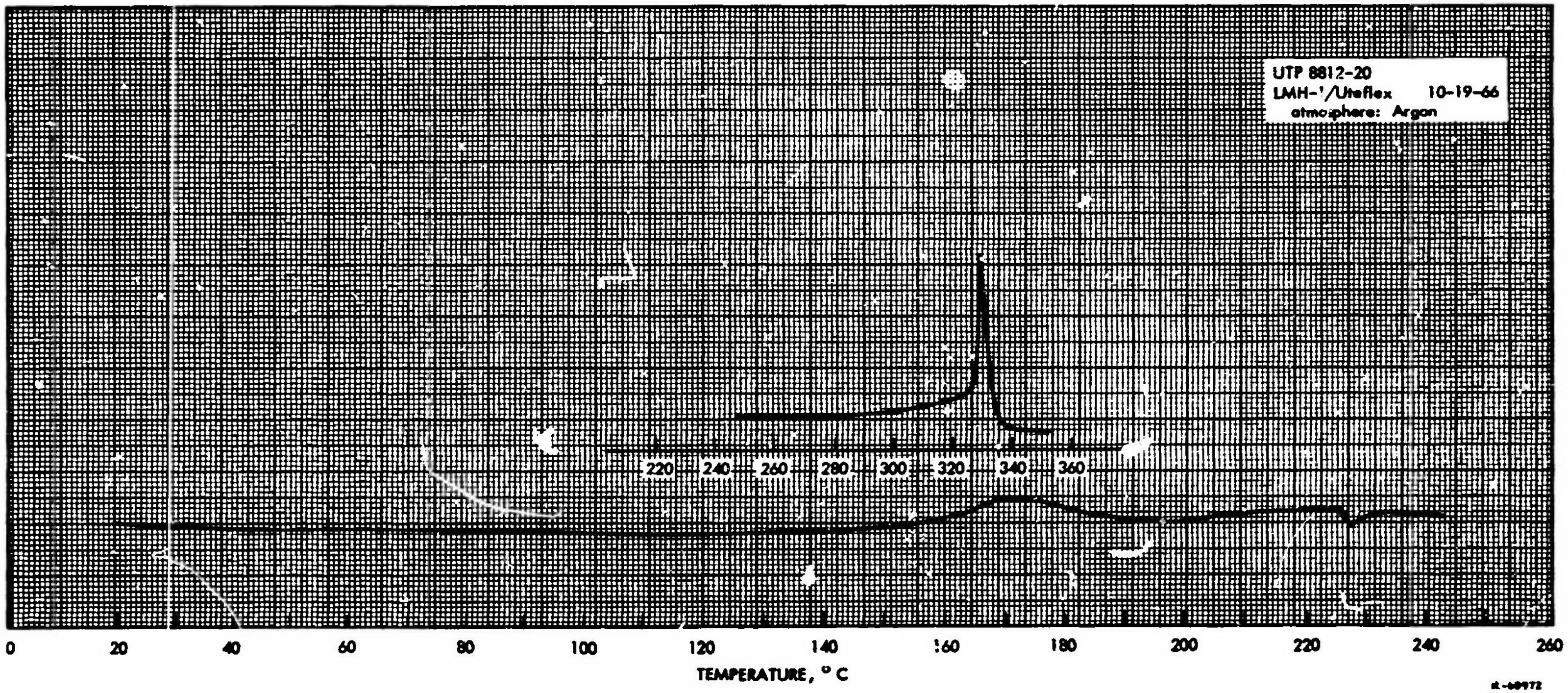

Figure 67. (U) Differential Thermal Analysis for UTP 8812

TABLE LXVIII

(U) CARD-GAP DATA

Cards	Sample No.											
	1	2	3	4	5	6	7	8	9	10	11	12
142	0											
100			0*			0						
95												
90					X†							
85												
80								X		0		
75				X			X				X	
48		X										

---

* 0 — no detonation

† X — detonation

SECTION III

CONCLUSIONS

(C) The primary objectives of the present program were the development of a spectrum of burning rates in AlH_3 propellants, the improvement of theoretical I_{sp} and density over that of propellants from Contract AF 04(611)-9570, the improvement of processing techniques to yield consistently higher density propellant, the improvement of physical properties, and the development of a body of propellant surveillance data.

(C) The large-scale processing techniques used in the present program have proved capable of delivering high-density propellant. The availability of higher stability AlH_3 from Dow Chemical Company has resulted in dramatic improvements in propellant stability. These improvements have not been evaluated fully because of the late availability of high-stability DPA-treated hydride, but routine processing of high-stability propellant may be reasonably predicted.

(C) Although elongation and tensile properties of the new Uteflex polymer system with AlH_3 have improved sufficiently to allow application to high-strain motors, a shortening of the cure cycle and the lowering of the cure temperature is still desirable. The improvement of this system is continuing under UTC in-house programs, and more favorable cure kinetics may be reasonably expected.

(C) The wide range of burning rates now available in AlH_3 -PEP propellants is remarkable in that no other propellant system offers such a spectrum. The influence of burning rate on I_{sp} efficiency in motors of large residence time has not been evaluated fully, but the influence appears to be a kinetic one which disappears in larger motors.

(C) Generally, sea-level and vacuum performance data have shown AlH_3 propellant to be competitive with beryllium propellants. The hazard data on AlH_3-PEP propellants indicate that they may be handled with no more danger than many operational propellants. Because complete surveillance data are not available yet, the shelf life of current aluminum propellants exposed to temperatures in the range normal to present high-energy systems should be competitive. The present propellant systems thus offer very high performance with unusual internal ballistic versatility and physical properties applicable to high-strain motors such that they may be logically considered for advanced operational application.

APPENDIX A

FORMULATION INDEX
ALUMINIZED PROPELLANTS

Aluminized Propellants

	7700	7701	7702	7708	7709	8258	8259	8260	8261	3262	8263	8264
HX-735	7.32	7.30	6.98	7.30	7.30	7.31	7.20	7.10	7.23	7.37	7.53	7.68
HX-874	---	---	---	---	---	0.75	0.88	0.9[illegible]	0.95	0.90	0.84	0.79
MAPO	0.26	0.77	0.74	0.77	0.77	---	---	---	---	---	---	---
TBM	0.48	---	---	---	---	---	---	---	---	---	---	---
Epon 812	0.74	0.73	0.74	0.73	0.73	0.74	0.72	0.71	0.62	0.53	0.43	0.33
NTPB	---	---	0.34	---	---	---	---	---	---	---	---	---
TMETN	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80
Al H-322	16.00	16.00	16.00	16.50	16.90	16.00	16.00	16.00	16.00	16.00	16.00	16.00
AP (190μ)	45.78	45.78	45.78	45.78	45.78	46.41	46.41	46.41	46.41	46.41	46.41	46.41
AP (10μ)	19.62	19.62	19.62	19.62	19.62	19.89	19.89	19.89	19.89	19.89	19.89	19.89
Ethyl centralite	1.00	1.00	1.00	0.50	0.10	---	---	---	---	---	---	---
Lecithin	---	---	---	---	---	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Uteflo A-2	---	---	---	---	---	0.05	0.05	0.05	0.05	0.05	0.05	0.05

	8289	8292	8294	8295	8297	8298	8608	8609	8610	8611
Uteflex 43S	---	6.92	---	---	---	---	---	---	---	---
Uteflex 44S	7.01	---	6.94	7.27	---	---	---	---	---	---
Uteflex 64S	---	---	---	---	7.51	7.56	---	---	---	---
L 1806	---	---	---	---	---	---	7.13	---	7.41	---
L 1807	---	---	---	---	---	---	---	7.24	---	7.51
HX-874	1.04	1.10	1.08	0.89	0.75	0.72	0.97	0.91	0.87	0.81
Epon 812	0.75	0.78	0.78	0.64	0.54	0.52	0.70	0.65	0.52	0.48
TMETN	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80
Al H-322	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00
AP (190μ)	46.48	46.48	46.13	46.13	46.48	46.48	46.48	46.48	46.48	46.48
AP (10μ)	19.92	19.92	19.77	19.77	19.92	19.92	19.92	19.92	19.92	19.92
Carbon black	---	---	0.50	0.50	---	---	---	---	---	---

	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 (190μ)	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50
AP (10μ)	20.40	20.40	20.40	20.40	20.40	20.35	20.30	20.40	20.10
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
Calcium hydroxide	---	---	---	---	---	0.05	0.10	---	---

	7945	7577	7578	7579	7580	7581	7584	7585	7586
HX 735	5.99	11.15	10.29	10.68	10.56	10.91	10.15	10.56	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

CONFIDENTIAL

	8289	8292	8294	8295	8297	8298	8608	8609	8610	8611
Uteflex 43S	---	6.92	---	---	---	---	---	---	---	---
Uteflex 44S	7.01	---	6.94	7.27	---	---	---	---	---	---
Uteflex 64S	---	---	---	---	7.51	7.56	---	---	---	---
L 1806	---	---	---	---	---	---	7.13	---	7.41	---
L 1807	---	---	---	---	---	---	---	7.24	---	7.51
HX-874	1.04	1.10	1.08	0.89	0.75	0.72	0.97	0.91	0.87	0.81
Epon 812	0.75	0.78	0.78	0.64	0.54	0.52	0.70	0.65	0.52	0.48
TMETN	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80
Al H-322	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00
AP (190μ)	46.48	46.48	46.13	46.13	46.48	46.48	46.48	46.48	46.48	46.48
AP (10μ)	19.92	19.92	19.77	19.77	19.92	19.92	19.92	19.92	19.92	19.92
Carbon black	---	---	0.50	0.50	---	---	---	---	---	---

	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 (190μ)	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50
AP (10μ)	20.40	20.40	20.40	20.40	20.40	20.35	20.30	20.40	20.40
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
Calcium hydroxide	---	---	---	---	---	0.05	0.10	---	---

	7945	7577	7578	7579	7580	7581	7584	7585	7586
HX 735	5.99	11.15	10.29	10.68	10.56	10.91	10.15	10.56	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 (190μ)	47.50	32.10	32.10	32.10	32.10	32.10	32.10	32.10	32.40
AP (10μ)	20.325	21.40	21.40	21.40	21.40	21.40	21.40	21.40	21.60
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	7595	7596	7597	7598	7599
HX-735	10.65	10.48	10.46	10.50	10.52	10.44	10.46	10.42	10.40	10.31	10.34	10.43	10.24
MAPO	---	0.51	0.44	0.59	0.66	0.37	0.44	0.29	0.22	0.72	---	---	---
Epon 812	---	1.02	1.01	1.02	1.02	1.01	1.01	1.01	1.00	0.99	0.71	0.35	1.06
TBM	1.00	0.49	0.59	0.39	0.30	0.68	0.59	0.78	0.88	0.48	1.45	1.71	1.20
TMETN	12.50	12.50	12.50	12.50	12.50	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	21.00	21.00	21.00	21.00	21.00
AP (190μ)	32.40	32.40	32.40	32.40	32.40	32.40	32.40	32.40	32.40	32.40	32.40	32.40	32.40
AP (10μ)	21.60	21.60	21.60	21.60	21.60	21.60	21.60	21.60	21.60	21.60	21.60	21.60	21.60
UNOX 201	0.85	---	---	---	---	---	---	---	---	---	---	---	---

	7703	7704	7705	7706	7707	8200	8201	8226	8227	6641
HX-735	14.59	13.13	11.68	10.22	8.75	9.78	10.05	---	---	7.71
MAPO	1.53	1.38	1.22	1.07	0.92	---	---	---	---	---
Epon 812	1.48	1.33	1.18	1.03	0.84	1.35	1.04	0.76	0.51	0.87
TBM	---	---	---	---	---	1.37	1.41	1.10	0.75	1.27
TMETN	---	1.76	3.52	5.28	7.04	12.50	12.50	8.80	8.80	11.93
Aluminum	16.00	16.00	16.00	16.00	16.00	21.00	21.00	16.00	16.00	22.50
AP (190µ)	45.78	45.78	45.78	45.78	45.78	32.40	32.40	45.78	45.78	---
AP (10µ)	19.62	19.62	19.62	19.62	19.62	21.60	21.60	20.62	20.62	19.68
AP (300µ)	---	---	---	---	---	---	---	---	---	35.92
Ethyl centralite	1.00	1.00	1.00	1.00	1.00	---	---	---	---	0.12
Emery 1025-9R	---	---	---	---	---	---	---	6.94	---	---
Emery 1025-94R	---	---	---	---	---	---	---	---	7.54	---

	7321	7322	7323	7324	7325	7326	7327	7328	7331	7332	7333
HX-735	8.24	8.18	8.12	8.07	8.02	8.06	8.00	7.93	7.62	7.74	7.51
MAPO	0.95	1.01	1.07	1.13	1.18	---	---	---	---	---	---
Epon 812	0.66	0.66	0.66	0.65	0.65	0.66	0.65	0.64	0.62	0.62	0.61
TMETN	12.05	12.05	12.05	12.05	12.05	12.05	12.05	12.05	12.05	12.05	12.05
Aluminum H-322	22.50	22.50	22.50	22.50	22.50	22.50	22.50	22.50	22.50	22.50	22.50
AP (190µ)	35.92	35.92	35.92	35.92	35.92	35.92	35.92	35.92	35.92	35.92	35.92
AP (10µ)	19.68	19.68	19.68	19.68	19.68	19.68	19.68	19.68	19.68	19.68	19.68
DGAP	---	---	---	---	---	1.13	1.20	1.28	---	---	---
NTPB	---	---	---	---	---	---	---	---	1.61	1.49	1.73

	6564	6565	6566	6567	6568	6570	6571
Uteflex 64S	7.11	7.00	6.89	7.02	6.58	7.01	6.92
HX-874	0.70	0.74	0.78	0.80	0.69	0.80	0.86
Epon 812	0.41	0.48	0.55	0.40	0.45	0.41	0.44
DOA	0.50	0.50	0.50	0.50	1.00	0.50	0.50
TMETN	8.68	8.68	8.68	8.68	8.68	8.68	8.68
Ethyl centralite	0.50	0.50	0.30	0.50	0.50	---	---
Al H-322	16.00	16.00	16.00	16.00	16.00	16.00	16.00
AP (300µ)	35.70	35.70	35.70	35.70	35.70	35.70	35.70
AP (10µ)	30.40	30.40	30.40	30.40	30.40	30.40	30.40
Resorcinol	---	---	---	---	---	0.50	---
2-NDPA	---	---	---	---	---	---	0.50

HX-874	0.70	0.74	0.78	0.80	0.69	0.80	[illegible]
Epon 812	0.41	0.48	0.55	0.40	0.45	0.41	0.44
DOA	0.50	0.50	0.50	0.50	1.00	0.50	0.50
TMETN	8.68	8.68	8.68	8.68	8.68	8.68	8.68
Ethyl centralite	0.50	0.50	0.50	0.50	0.50	---	---
Al H-322	16.00	16.00	16.00	16.00	16.00	16.00	16.00
AP (300μ)	35.70	35.70	35.70	35.70	35.70	35.70	35.70
AP (10μ)	30.40	30.40	30.40	30.40	30.40	30.40	30.40
Resorcinol	---	---	---	---	---	0.50	---
2-NDPA	---	---	---	---	---	---	0.50

	6572	6573	6574	6575	6576	8640	8641
Uteflex 64S	7.42	7.09	7.09	7.53	7.09	7.44	7.46
HX-874	0.86	0.71	0.71	0.75	0.71	0.88	---
Epon 812	0.44	0.42	0.42	0.44	0.42	0.48	0.49
DOA	0.50	0.50	0.50	0.50	0.50	---	---
TMETN	8.68	8.68	8.68	8.68	8.68	8.80	8.80
Ethyl centralite	---	---	---	---	0.30	0.30	0.30
Al H-322	16.00	16.00	16.00	16.00	16.00	16.00	16.00
AP (300μ)	35.70	35.70	35.70	35.70	35.85	---	---
AP (10μ)	30.40	30.40	30.40	30.40	30.55	19.83	19.83
Resorcinol	---	0.50	---	---	---	---	---
2-NDPA	---	---	0.50	---	---	---	---
AP (190μ)	---	---	---	---	---	46.27	46.27
TBM	---	---	---	---	---	---	0.85

	7900	7901	7907	7908	7909	7910	7911	7912	7913	7914
HX-735	6.02	6.04	5.96	5.88	5.80	5.94	5.86	6.12	6.10	5.78
TMETN	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80
Al	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00
AP (190μ)	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50
AP (10μ)	20.40	20.40	20.40	20.40	20.40	20.40	20.40	20.40	20.40	20.40
Ethyl centralite	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
NTPB	1.18	0.89	0.95	1.01	1.07	1.26	1.34	0.83	1.10	1.42
NTEB	---	0.27	0.29	0.31	0.33	---	---	0.25	---	---

	7915	7916	7917	7918	7919	7920	7921	7922	7923	7924
HX-735	6.18	6.10	6.02	5.95	5.87	6.14	6.06	5.98	5.90	5.83
TMETN	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80	8.80
Epon 812	---	---	---	---	---	0.23	0.25	0.27	0.29	0.30
Al	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00
AP (190μ)	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50	47.50
AP (10μ)	20.40	20.40	20.40	20.40	20.40	20.40	20.40	20.40	20.40	20.40
Ethyl centralite	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
NTPB	---	---	---	---	---	0.83	0.89	0.95	1.01	1.07
NTEB	1.02	1.10	1.18	1.25	1.33	---	---	---	---	---

APPENDIX B

FORMULATION INDEX DOWANE 1451 PROPELLANTS

CONFIDENTIAL

Dowane 1451 Propellants

	6822	6825	6826	6827	6829	6834	6842	6835	6844	6848	6849
HX-735	7.81	6.9[illegible]	6.64	9.26	8.86	7.32	9.74	7.20	9.53	7.96	10.70
MAPO	0.96	0.85	0.82	---	---	0.79	1.21	0.77	1.19	0.99	1.33
Epon 812	0.63	0.56	0.54	1.04	1.00	0.54	0.80	0.53	0.78	0.65	0.87
TBM	---	---	---	1.02	1.46	---	---	---	---	---	---
TMETN	9.40	10.20	9.80	14.01	14.01	8.75	11.75(1)	8.6(1)	11.50(1)	9.60(1)	12.90(1)
Dowane 1451	23.00	21.00	14.25	25.00	25.00	26.00	28.00	24.80	22.0	26.70	23.20
Aluminum	---	3.65	9.75	---	---	---	---	---	---	---	---
Ethyl centralite	0.10	0.10	0.10	0.15	0.15	---	---	---	---	---	---
Atlas G-2684	0.10	0.10	0.10	---	---	0.10	---	0.10	---	---	---
AP (300μ)	26.20	15.43	22.87	7.84	7.84	26.20	24.25	30.20	27.50	27.05	25.50
AP (10μ)	30.30	41.20	35.13	41.69	41.69	30.30	24.25	27.80	27.50	27.05	25.50

	6814	6840	6841	6843	6845	6846	6847	6850
HX-735	9.40	7.63	7.83	9.74	12.88	11.29	8.16	9.74
TBM	---	0.76	0.61	---	---	---	---	---
MAPO	1.16	0.38	0.33	1.21	1.61	1.41	0.88	1.21
Epon 812	0.76	0.63	0.63	0.80	1.05	0.92	0.60	0.80
TMETN(1)	14.15	9.50	9.50	11.75	6.66	9.08	14.46	11.75
Dowane 1451	25.00	23.00	23.00	24.50	22.60	23.30	26.50	24.50
AP (300μ)	7.84	29.00	29.00	26.00	27.60	27.00	24.70	31.20
AP (10μ)	41.69	29.00	29.00	26.00	27.60	27.00	24.70	20.80
Atlas 2684	---	0.10	0.10	---	---	---	---	---

	6851	6852	6858	6859	6860	6861	6862	6863	6864
Uteflex 35-S	7.85	7.74	6.23	9.46	9.32	9.55	9.40	9.72	9.61
TBM	1.02	1.14	0.63	1.22	1.37	0.79	0.95	---	---
MAPO	---	---	0.22	---	---	0.33	0.33	0.94	1.06
Epon 812	0.53	0.52	0.42	0.64	0.63	0.65	0.64	0.66	0.65
TMETN(1)	9.50	9.50	11.40	14.15	14.15	14.15	14.15	14.15	14.15
Dowane 1451	23.00	23.00	23.00	25.00	25.00	25.00	25.00	25.00	25.00
AP (300μ)	29.00	29.00	29.00	7.84	7.84	7.84	7.84	7.84	7.84
AP (10μ)	29.00	29.00	29.00	41.69	41.69	41.69	41.69	41.69	41.69
Atlas 2684	0.10	0.10	0.10	---	---	---	---	---	---

	6867	6868	6870	6871	6872	6873	6874	6875
HX-735	---	---	9.47	9.41	9.36	8.23	8.19	8.23
Uteflex 35-S	9.26	9.13	---	---	---	---	---	---
TBM	1.11	1.26	0.75	0.74	0.74	---	---	---
MAPO	0.32	0.31	0.41	0.48	0.54	0.89	0.88	0.89
Epon 812	0.63	0.62	0.69	0.69	0.68	0.59	0.59	0.59
TMETN(1)	14.15	14.15	14.15	14.15	14.15	14.57	14.49	14.57
Dowane 1451	25.00	25.00	25.00	25.00	25.00	23.48	23.37	23.48
AP (300μ)	7.84	7.84	7.84	7.84	7.84	10.35	10.30	10.35
AP (10μ)	41.69	41.69	41.69	41.69	41.69	41.39	41.18	41.39
Fe_2O_3	---	---	---	---	---	0.50	1.00	---
Ferrocene	---	---	---	---	---	---	---	0.50

	6876	6877	6878	6879	6880	6881	6882	6885	6886
HX-735	8.19	8.27	8.27	11.69	11.69	11.69	11.69	8.27	11.69
	0.88	0.89	0.89	1.26	1.26	1.26	1.26	0.89	1.26
						0.85	0.85	0.60	0.85

Uteflex 35-S	9.26	9.13	---	---	---	---	---	---
TBM	1.11	1.26	0.75	0.74	0.74	---	---	---
MAPO	0.32	0.31	0.41	0.48	0.54	0.89	0.88	0.89
Epon 812	0.63	0.62	0.69	0.69	0.68	0.59	0.59	0.59
TMETN(1)	14.15	14.15	14.15	14.15	14.15	14.57	14.49	14.57
Dowane 1451	25.00	25.00	25.00	25.00	25.00	23.48	23.37	23.48
AP (300μ)	7.84	7.84	7.84	7.84	7.84	10.35	10.30	10.35
AP (10μ)	41.69	41.69	41.69	41.69	41.69	41.39	41.18	41.39
Fe_2O_3	---	---	---	---	---	0.50	1.00	---
Ferrocene	---	---	---	---	---	---	---	0.50

	6876	6877	6878	6879	6880	6881	6882	6885	6886
HX-735	8.19	8.27	8.27	11.69	11.69	11.69	11.69	8.27	11.69
MAPO	0.88	0.89	0.89	1.26	1.26	1.26	1.26	0.89	1.26
Epon 812	0.59	0.60	0.60	0.85	0.85	0.85	0.85	0.60	0.85
TMETN(1)	14.49	14.64	14.64	9.20	9.20	9.20	9.20	14.64	9.20
Dowane 1451	23.37	23.60	23.60	25.00	25.00	25.00	25.00	23.60	25.00
AP (300μ)	10.30	10.40	10.40	41.60(4)	41.60(6)	44.60(5)	41.18	10.40	41.60
AP (10μ)	41.18	41.60(2)	41.60(3)	10.40(4)	10.40(6)	10.40(5)	10.30	41.60	10.40
Ferrocene	1.00	---	---	---	---	---	---	---	---
Sodium barbiturate	---	---	---	---	---	---	0.52	---	---

	8801	8802	8809	8812	8814	8815	8819	8820
HX-735	6.88	8.40	8.66	---	10.29	---	---	---
Uteflex 35-S	---	---	---	7.70	---	7.71	8.64	8.17
TBM	---	---	---	0.95	---	0.94	0.99	0.94
MAPO	0.71	0.87	0.96	---	1.05	---	---	---
Epon 812	0.51	0.63	0.63	0.55	0.76	0.55	0.62	0.59
TMETN	12.30(1)	10.00(1)	10.75	9.50	8.40	10.00	10.75	10.40
Dowane 1451	25.20	23.50	25.00	24.00	25.00	23.50	25.00	21.00
AP (300μ)	10.66	11.10	42.80(6)	30.50	43.20(6)	11.10	42.64	43.70
AP (10μ)	42.64	44.40	10.70(6)	26.00	10.80(6)	44.40	10.66	10.90
DOA	---	---	0.50	0.60	0.50	0.60	0.50	0.50
Ethyl centralite	---	---	---	0.20	---	0.20	0.20	0.20
Atlas 2684	0.10	0.10	---	---	---	---	---	---
Fe_2O_3	1.00	1.00	---	---	---	1.00	---	---
Aluminum	---	---	---	---	---	---	---	3.60

(1) Contains 1% ethyl centralite

(2) Contains 0.5% $FeNH_4SO_4$

(3) Contains 1% $FeNH_4SO_4$

(4) 0.5% coating of sodium barbiturate

(5) Contains 1% ammonium oxalate

(6) 1% coating of sodium barbiturate

APPENDIX C

COMPUTER PRINTOUT OF UTP 8812

```
UTC0805                                                              UTC080501-
                                                                        PAGE  1

INGREDIENTS                           WT.PCT.       ELEMENTS    GM ATOMS
0106  LMH-1                            24.00           AL       8.00266756D-01
0017  AMMONIUM PERCHLORATE             56.50           H        5.56075402D+00
0447  UTEFLEX                           7.68           CL       4.80851064D-01
0104  TMETN                             9.50           N        6.09401659D-01
0405  EPON 812                          0.51           O        2.44832167D+00
0045  DIOCTYL ADIPATE                   1.00           C        6.70519823D-01
0457  HX874                             0.81

                                               THROAT            EXHAUST[ 1]

AREA RATIO                                     1.00000000D+00    1.02158691D+01

OPTIMUM ISP, SEC                               1.13072255D+02    2.80942237D+02
VACUUM ISP, SEC                                2.12402238D+02    3.06786828D+02

C*, FT/SEC                                     5.53860346D+03

VELOCITY, FT/SEC                               3.63798674D+03    9.03903552D+03
DENSITY, GM/CC                                 3.68309355D-03    1.45103020D-04

                            CHAMBER            THROAT            EXHAUST[ 1]
PRESSURE, PSIA              1.00000000D+03     5.77012397D+02    1.46960000D+01
PRESSURE, ATM               6.80457267D+01     3.92632279D+01    1.00000000D+00
TEMPERATURE, DEG K          3.38905116D+03     3.18385262D+03    2.09800952D+03
HEAT CAP., CAL/DEG K/G      5.28204377D-01     5.25545776D-01    4.85358578D-01
ENTHALPY, KCAL/G           -5.19222122D-01    -6.66087989D-01   -1.42587873D+00
ENTROPY, CAL/DEG K/G        2.52229955D+00     2.52229968D+00    2.52229874D+00
MOLS OF GAS / 100 G         4.10626142D+00     4.07833252D+00    4.00128725D+00

COMBUSTION PRODUCTS

                            CHAMBER            THROAT            EXHAUST[ 1]
                             MOLS/100 G         MOLS/100 G        MOLS/100 G
AL            G             7.40893132D-04     2.85457466D-04    1.16482225D-08
ALCL          G             1.83521355D-02     1.09256529D-02    2.08968196D-05
ALCL2         G             2.50415687D-02     1.81418741D-02    2.15150369D-04
ALCL3         G             3.57501907D-04     2.93634173D-04    1.27444921D-05
ALH           G             1.80213041D-04     6.26425271D-05    1.67618254D-09
ALN           G             5.01713775D-07     1.14954878D-07    1.00000000D-10
ALO           G             2.70289886D-04     9.80918440D-05    2.90324545D-09
AL2O          G             1.42599363D-04     4.10404293D-05    1.00000000D-10
C             G             1.42391837D-08     3.86806842D-09    1.00000000D-10
CH            G             1.96285823D-08     5.50561228D-09    1.00000000D-10
CH2           G             1.10175481D-06     4.85296574D-07    9.26740635D-10
CH3           G             1.01454985D-06     4.78006863D-07    2.66500357D-09
CH4           G             4.05337260D-07     2.37480709D-07    1.42715087D-08
CO            G             6.44827079D-01     6.44725068D-01    6.35108226D-01
CO2           G             2.56901890D-02     2.57935458D-02    3.54115817D-02
CL            G             2.14305448D-02     1.77742512D-02    1.58216969D-03
```

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UTC0805                                                              UTC080501-
                                                                        PAGE  2

                          CHAMBER              THROAT               EXHAUST[ 1]
                        MOLS/100 G           MOLS/100 G            MOLS/100 G
CL2        G          2.51192121E-05       1.81084707E-05        5.53105615E-07
H          G          1.53644232E-01       1.19715285E-01        8.65367203E-03
HCL        G          3.89861902E-01       4.14950292E-01        4.78778357E-01
H2         G          1.90242790E+00       1.92502627E+00        1.99443910E+00
H2O        G          5.95007772E-01       5.80690611E-01        5.42068750E-01
N          G          1.72149459E-05       7.48035673E-06        3.78381727E-09
NH         G          6.61745147E-05       3.11797209E-05        4.66010869E-08
NH2        G          3.77484807E-05       2.02232089E-05        1.47401063E-07
NH3        G          9.95578395E-05       6.68125864E-05        5.59296627E-06
NO         G          1.12727125E-03       6.50494548E-04        4.43933891E-06
N2         G          3.04026595E-01       3.04312677E-01        3.04695714E-01
O          G          1.00897725E-03       5.20932148E-04        8.63631266E-07
OH         G          2.17490922E-02       1.43166071E-02        2.89095561E-04
O2         G          1.25485793E-04       6.29598441E-05        1.00663024E-07
AL2O3      L          3.77519076E-01       3.85188603E-01        0.00000000E+00
AL2O3      S          0.00000000E+00       0.00000000E+00        4.00008975E-01
C          S          0.00000000E+00       0.00000000E+00        0.00000000E+00
```

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13. ABSTRACT (Unclassified)

The progress made in developing and characterizing an advanced LMH-1 solid propellant system is described. The objectives of this program were the development of a spectrum of burning rates in LMH-1 propellants, the improvement of theoretical specific impulse and density over that of propellants characterized under Contract AF 04(611)-9570, the improvement of processing techniques to yield consistently higher density propellant, the improvement of physical properties, and the development of a body of propellant surveillance data. (U)

An analysis of I_{sp} efficiencies measured in 10-lb motors at sea-level and altitude conditions (ϵ up to 80:1) was made. (U)

A wide range of burning rates has been demonstrated in 10-lb motors with negligible losses in performance efficiency. The factors affecting burning rates have been evaluated. Stability of the propellant under long-term storage was investigated by means of static and dynamic DTA, Taliani gas evolution measurements, and swelling measurements on 2-in. cubes. A section of a large motor (6-in. web) containing this material is in storage. (U)

A complete hazard and viscoelastic characterization was conducted on this system, and BATES motors were shipped to the Air Force for altitude testing. (U)

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
LMH-1 SOLID PROPELLANT						
MAXIMUM I_{sp} AND SOLIDS LOADING						
BURNING RATE STUDIES						
DENSITY-I_{sp} TRADEOFF STUDIES						
AGING AND TEMPERATURE LIMITS						
IMPROVEMENT OF MECHANICAL PROPERTIES						
BALLISTIC CHARACTERIZATION (SEA LEVEL AND ALTITUDE)						
PHYSICAL AND HAZARD CHARACTERIZATION						

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