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DEVELOPMENT AND TEST OF HIGH ENERGY SOLID PROPELLANTS (U)

REPORT NO. AFRPL-TR-66-11

28 JANUARY 1966

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

(Prepared under Contract No. AF 04(611)-10754 By

HERCULES POWDER COMPANY
INCORPORATED
BACCHUS WORKS ● MAGNA , UTAH

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FOREWORD

This is the second quarterly report under Contract AF 04(611)-10754. This report was prepared by the Propellant Development Group, Bacchus Works, Chemical Propulsion Division, Hercules Powder Company. The report was written by R. F. Keller, J. L. Judkins, and G. R. Gibson, and approved by J. W. Schowengerdt and Dr. R. L. Schaefer. (Unclassified)

Preparation of this report is authorized under Contract AF 04(611)-10754, in accordance with Exhibit B, paragraph 2.1. (Unclassified)

This report is classified Confidential because it contains theoretical and experimental data on beryllium and LMH-2 propellants. (CONFIDENTIAL)

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ABSTRACT

The objective of this contract is to conduct theoretical and experimental investigations resulting in the demonstration of beryllium hydride (LMH-2) solid propellants delivering in excess of 280 lbf-sec/lbm at standard conditions. The program consists of three tasks: Task I, Analysis and Data Correlation; Task II, Formulation and Ballistic Evaluation; and Task III, Advanced Concepts. (CONFIDENTIAL)

During the first quarter, data correlations were completed under Task I and LMH-2 formulations were designed which were predicted to meet the program objective. In addition, beryllium analog formulations of the proposed LMH-2 systems were also designed for formulation screening. (CONFIDENTIAL)

Under the Task II effort during this quarter, all the laboratory formulation work was completed on the beryllium analog formulations. The laboratory work included determination of processibility, stability, sensitivity, mechanical properties, strand burning rates, and combustion efficiency. Thirty-nine 15-lb castings and eleven 15-lb-charge firings of the beryllium propellants were made. Preliminary data from the beryllium firings indicate that impulse efficiency is still increasing with increased flame temperatures above 3900° K and that good impulse efficiency (~93 percent) can be achieved with 15.5 percent beryllium systems, which are equivalent to the metal level obtained with 19 percent LMH-2 loadings. (CONFIDENTIAL)

Evaluation continued to determine the effect that surface-treated LMH-2 has on propellant processibility. Lots of AP-treated LMH-2 were found to consistently decrease propellant mixing viscosities by a factor of three from that obtained with untreated LMH-2. Samples of wax treated LMH-2 also improved processing but to a lesser extent than AP treating. A serious gassing problem occurred with some "as-received" LMH-2 lots, resulting in porous grains. Heating the LMH-2 under vacuum and surface treating the LMH-2 with AP and wax eliminated the gassing for most lots. (CONFIDENTIAL)

In Task III, additional effort was spent on characterization of AP-treated LMH-2. Photomicroscopy and liquid separation techniques indicated that the material primarily was free LMH-2 and free AP; a portion of the material was 75- to 150-micron agglomerates with AP in the center and LMH-2 particles adhered to the surface of the AP crystal. Active hydrogen analysis showed no loss in purity for the treated LMH-2. The AP-treated LMH-2 showed no improvement in combustion efficiency over untreated LMH-2, based on combustion bomb tests at Allegany Ballistics Laboratory. (CONFIDENTIAL)

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LIST OF ABBREVIATIONS

Abbreviation	Meaning
2-NDPA	2-Nitrodiphenylamine
15PC	15-pound charge
AL	Aluminum
AN	Ammonium nitrate
AP	Ammonium perchlorate
Be	Beryllium
BeO	Beryllium oxide
CMDB	Composite modified double-base
DTA	Differential thermal analysis
ESD	Electrostatic discharge
FPC	40-pound charge
HMX	Cyclotetramethylene tetranitramine
K	Kelvin
LMH-2	Beryllium hydride
NFPA	1, 2-bis (Difluoramino) propyl acetate
NG	Nitroglycerin
PNC	Plastisol nitrocellulose
P-K-r	Pressure-K-burning rate
RES	Resorcinol
TA	Triacetin
TAGN	Triaminoguanidine nitrate
TDI	Toluene diisocyanate
TFLN	Teflon

LIST OF ABBREVIATIONS (Cont)

Abbreviation	Meaning
TVOPA	Hexakis (Difluoramino) vinoxy propane
VCP	Be/HMX propellant formulation

SECTION 1

INTRODUCTION

A. OBJECTIVE

The objective of this program is to conduct theoretical and experimental investigations resulting in the demonstration of beryllium hydride (LMH-2) solid propellants delivering a specific impulse in excess of 280 lb-sec/lb at standard conditions. (CONFIDENTIAL)

Solid propellants containing LMH-2 have a theoretical specific impulse up to 30 sec greater than that of beryllium-containing propellants. Thus far the firings of test motors containing LMH-2 propellants have not yielded the expected gain in delivered specific impulse. Motor and propellant parameters of chamber temperature, chamber pressure, mass flow rate, expansion ratio, oxidation ratio, oxidizer particle size, hydride content, metal/hydride ratio, and total metal content have been investigated under various government contracts over the past 2 years. Although much has been learned about LMH-2 propellants during this period, the high specific impulse promised by the hydride has not been realized. More research is required on these propellants before they can be efficiently utilized in large motors. (CONFIDENTIAL)

B. SCOPE

The program is a three-task effort. In task I, the results of previous beryllium and LMH-2 firings will be correlated to define the parameters important for higher delivered impulses. The results of this effort will be applied in task II to formulate and test candidate high-performance propellant systems. The objective of the task III effort will be to use advanced formulation or motor techniques to study and/or improve LMH-2 combustion. (CONFIDENTIAL)

SECTION 2

TASK I, ANALYSIS AND DATA CORRELATION

A. SCOPE

The objective of this task is to evaluate and correlate available beryllium and LMH-2 firings. Particular emphasis will be placed on the correlation of motor efficiency with motor and formulation variables. Theoretical specific impulse calculations will be conducted on a wide variety of formulations to establish the relationship between propellant parameters and theoretical performance of beryllium and LMH-2 propellants. Based on the motor efficiency correlations and theoretical performance calculations, formulations will be designed which are predicted to deliver specific impulse values capable of meeting the program objectives. In addition to the advanced formulation effort, propellants will be investigated to supplement or verify reported data on current beryllium and LMH-2 formulations. The performance of beryllium systems containing high metal levels equivalent to those in high energy LMH-2 systems will also be investigated. (CONFIDENTIAL)

B. DATA CORRELATIONS

As reported in the first quarterly report,[1] a literature survey was conducted and available data on the impulse efficiency of beryllium and LMH-2 motor firings were correlated on the basis of motor and propellant parameters. (CONFIDENTIAL)

In summary, the following were found to be important factors influencing the motor efficiency of beryllium and LMH-2 propellants:

(1) High flame temperature and high oxidation ratio propellants show no loss in efficiency with decreasing pressure down to 500 psia.

(2) Low flame temperature or low oxidation ratio propellants show loss in efficiency with decreasing pressure. This effect could not be completely separated from mass flow or residence time effects on efficiency.

(3) Both beryllium and LMH-2 propellants show a strong dependence on high flame temperatures for good efficiency.

(4) Oxidation ratio has a strong effect on the efficiency of low temperature beryllium propellants. There is an indication that oxidation ratio is important for LMH-2 efficiency, but only a general trend could be obtained.

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[1] Development and Test of High Energy Solid Propellants, Report No. HPC-230-12-5-1, dated 28 October 1965

(5) Correlations based on total LMH-2 content show a loss in efficiency at high hydride loadings (greater than 15 percent).

(6) More intimate contact of oxidizer and fuel in LMH-2 systems should improve efficiency based on efficiency effects of AP particle size, AP/LMH-2 ratios, and ground LMH-2.

(CONFIDENTIAL)

The importance of flame temperature on obtaining good efficiencies with beryllium and LMH-2 propellants is illustrated in Figures 1 and 2, respectively. (CONFIDENTIAL)

C. FORMULATION AND TEST DESIGN

The correlations show several areas in which further investigation is needed before reliable predictions can be made for the performance of both beryllium and LMH-2 propellants. In general, the beryllium correlations are significantly better than for LMH-2 systems. Additional beryllium testing is still needed in the following areas for further clarification of propellant parameters on impulse efficiency:

(1) High metal levels (> 15.5 percent) at high flame temperatures and oxidation ratios

(2) High metal levels and high flame temperatures at low oxidation ratios

(3) Evaluation of oxidizers different from AP and HMX, such as AN and $TAGNO_3$

(4) Additional testing at high oxidation ratios and high flame temperatures for determining the effect of AP particle size

(CONFIDENTIAL)

Further testing is also needed in the following areas for clarification of motor parameters:

(1) Optimization of nozzle geometry at high metal levels

(2) Increased L* studies for both efficient and inefficient beryllium propellants and LMH-2 propellants

(CONFIDENTIAL)

For LMH-2 systems, the following testing is needed to clarify propellant parameters:

(1) High LMH-2 loadings at flame temperatures in excess of 3600° K and at high oxidation ratios

(2) Comparative evaluation of AP, AN, and HMX oxidizers

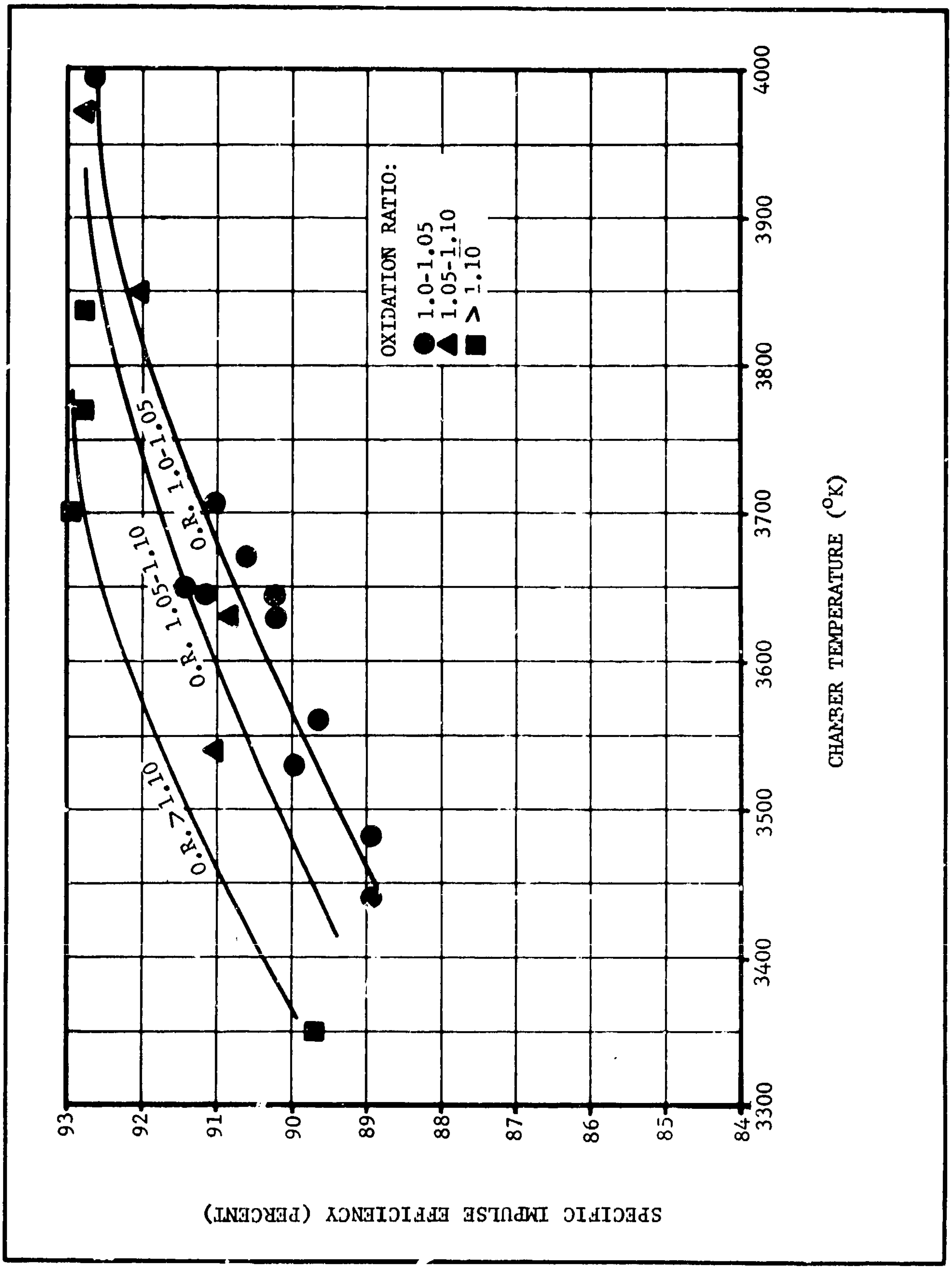
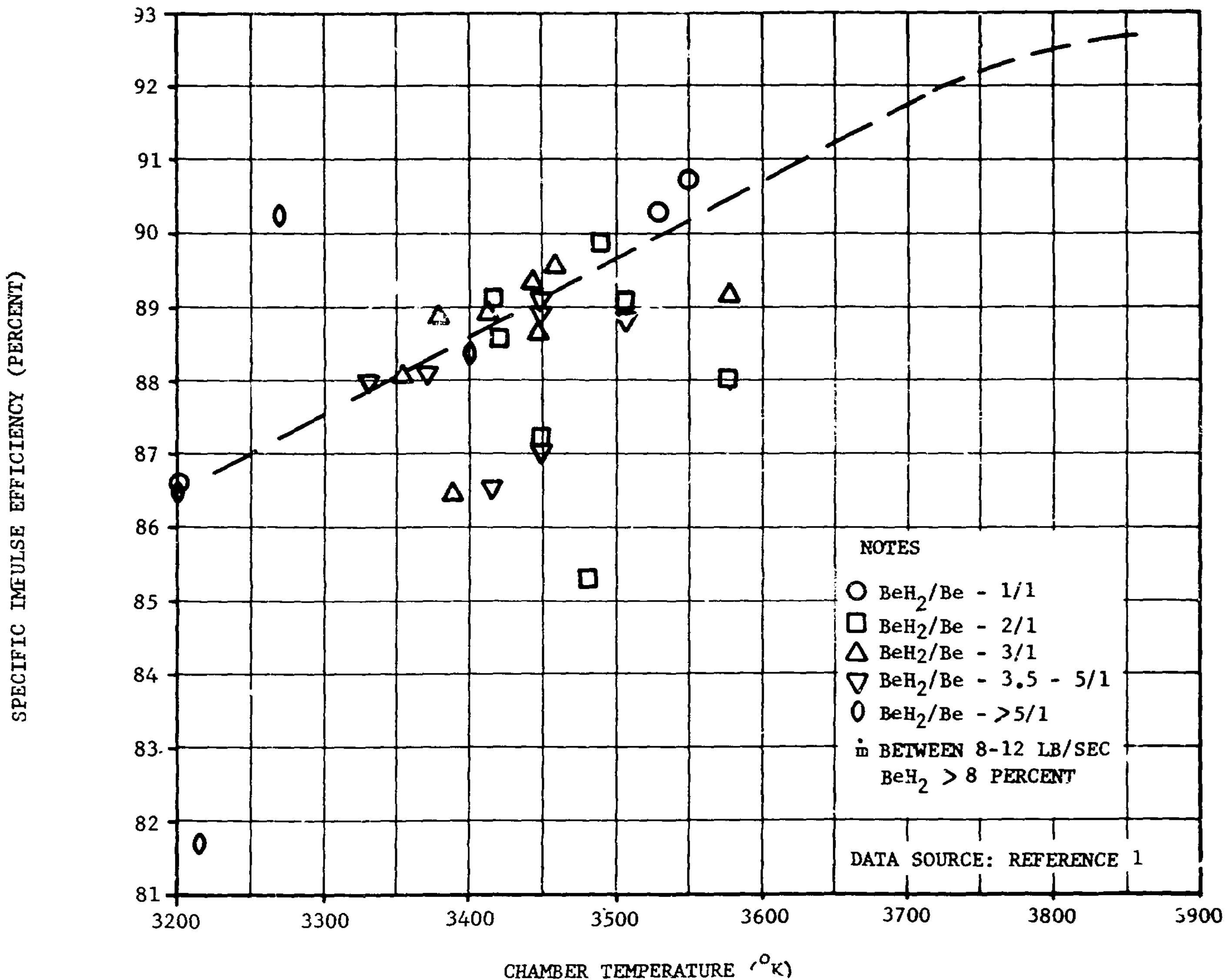


Figure 1. Effect of Chamber Temperature and Oxidation Ratio on Specific Impulse Efficiency of Beryllium Propellants



[1] Refer to References

Figure 2. Effect of Chamber Temperature on Specific Impulse Efficiency for LMH-2 Propellants

(3) More intimate contact between oxidizer and LMH-2

(4) Addition of fluorine to LMH-2 as a combustion aid (CONFIDENTIAL)

The Task II effort is designed to clarify these areas for beryllium and LMH-2 propellants. (CONFIDENTIAL)

Table I shows a list of the beryllium formulations chosen for testing to better define the effect of propellant parameters on performance. VIH propellant was designed to have the same metal level and oxidation ratio as the proposed 19-percent LMH-2/AP formulation. VII was formulated to demonstrate the effect on efficiency due to decreasing oxidation ratio in a high metal level propellant. VIJ was formulated to show the effect on efficiency of the AP:Be ratio at a constant metal level, flame temperature, and oxidation ratio. VIJ was also formulated with 45μ and 180μ AP to demonstrate the effect of AP particle size on impulse efficiency thus providing the performance trade-off between AP particle size and LMH-2 loadings necessary to optimize delivered impulse. VIK, VIL, VIM, and VIN represent the beryllium analogs of the 17 percent LMH-2, AP; 15 percent LMH-2, AP, HMX; 19 percent LMH-2, AN; and 17 percent LMH-2, AN formulations, respectively, in both metal level and oxidation ratio. VIO was designed to determine the effect of oxidation ratio on efficiency at low metal levels, and VIG was formulated to evaluate TAGNO3 as an oxidizer. (CONFIDENTIAL)

Based on the theoretical calculations and using the temperature correlation derived in Figure 2, six candidate LMH-2 formulations were chosen which are designed to confirm the expected efficiency envelope and to attain the program objective of a demonstrated $I_{sp} \geq 280$ sec. Table II shows the LMH-2 propellants along with the theoretical and predicted performance values. (CONFIDENTIAL)

Additional testing to determine the effect of motor parameters on the performance of beryllium and LMH-2 propellants will also be accomplished in Task II as follows:

Motor Parameter	Formulation	Number of Firings
Nozzle Approach Angle		
15° Approach	VCP	2
15° Approach	VIJ	4
5° Approach	VCP	2
5° Approach	VIJ	4
High L*	VCP	2
High L*	--(a)	2
High L*	VIY	10

(a)This formulation will be an inefficient beryllium propellant chosen after completion of the formulation screening phase (CONFIDENTIAL)

TABLE I

BERYLLIUM PROPELLANTS FOR FORMULATION SCREENING

Formulation	Be (Wt %)	Oxidizer Type	I_{sp} (sec)	T_c (°K)	O.R.*	Number of Firings**		Purpose
						1000 psi	500 psi	
VCP	10	HMX/AP	283	3840	1.22	3	3	Control
VIH	15.5	AP	278	4173	1.17	3	3	LMH-2 19% Analog with respect to TM*** and O.R.
VII	15.5	AP	279	3971	1.05	3	3	LMH-2 19% Analog with respect to TM; lower O.R.
VIJ	15.5	AP	277	4145	1.17	3	--	Total AP effect
VIK	14.0	AP	277	4109	1.27	3	3	LMH-2 17% Analog TM and O.R.
VIL	12.0	HMX/AP	281	4008	1.24	3	3	LMH-2 15% Analog AP/HMX with respect to TM and O.R.
VIM	15.5	AN	281	3820	1.17	3	3	LMH-2 19% Analog with respect to TM and O.R.

*, **, ***, See end of table

TABLE I (Cont)

BERYLLIUM PROPELLANTS FOR FORMULATION SCREENING

Formulation	Be (Wt %)	Oxidizer Type	I_{sp} (sec)	T_c (°K)	O.R.*	Number of Firings** 1000 psi	Number of Firings** 500 psi	Purpose
VIN	14.0	AN	279	3744	1.28	3	3	LMH-2 17% Analog with respect to TM and O.R.
VIO	8.0	HMX/AP	281	3745	1.38	3	--	O.R. effect at low TM
VIG	12.5	$TAGNO_3$/AP	291	3633	1.02	3	--	Evaluate $TAGNO_3$
						30	21	

*C.R. = Oxidation Ratio

**Optimum expansion ratio at Bacchus ambient

***TM = Total Metal Level

TABLE II

IMH-2 PROPELLANTS FOR FORMULATION SCREENING

Propellant Type	Binder Level (Wt %)	Oxidizer (Wt %)	IMH-2 (Wt %)	I^{o}_{1000} (sec)	T_c (^{o}K)	(.*	Impulse Efficiency Range	Predicted I_{sp} Del (H)**	Predicted I_{sp} Del (L)***
VIX	62	AP	15	304	3678	1.34	90.7-91.6	279	275
VIY	62	AP	17	309	3679	1.22	90.7-91.6	283	280
VIZ	62	AP	19	314	3671	1.11	90.6-91.5	287	285
--	62	AP/HMX	15	307	3636	1.23	90.2-91.2	280	277
VJA	62	HMX	15	309	3621	1.15	90.0-91.1	282	278
--	62	AN	17	309	3529	1.25	88.6-90.4	279	274

*Oxidation Ratio

**(H) High value expected

***(L) Low value expected

It should be noted that some minor changes have been made to the test design as originally reported in the first quarterly report.[1] (Unclassified)

D. PROGRAM DATA ANALYSIS

Only limited data are available from the Task II firings to date, and no attempt was made to update the developed correlations. However, the data available on the high temperature VIH and VIJ formulations indicate that impulse efficiency is still increasing with increased flame temperatures above 3900° K and that good impulse efficiencies can be obtained at metal levels equivalent to 19 percent LMH-2 loadings. (CONFIDENTIAL)

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[1] Ibid

SECTION 3

TASK II, FORMULATION AND BALLISTIC EVALUATION

A. SCOPE

This task will comprise the majority of the program effort. The objective of this task is to formulate and test candidate high-performance propellant systems selected under task I. This task will utilize both beryllium control and analog formulations as well as LMH-2 formulations. Approximately 37 LMH-2 and 90 beryllium motors will be evaluated under this task. The test motors shall contain a nominal 10- to 15-lb propellant charge and exhibit mass flow rates of approximately 5-lb/sec or greater. The task will be divided into two phases. In phase A, a formulation screening will be conducted, with the bulk of the motors being fired at approximately 1000 psia exhausted to Bacchus ambient pressure (~ 12.2 psia) with optimum expansion ratio. Phase B will more extensively characterize selected high performance formulations. Table III contains a breakdown of various areas to be investigated under task II. (CONFIDENTIAL)

TABLE III

TASK II, FORMULATION SCREENING

Subtask	Purpose	No. Firings (Be)	No. Firings (LMH-2)
Phase A			
II-6	Efficiency correlations with Be propellants	51	--
II-7	Oxidizer particle size studies with Be propellants	9	--
II-8	Optimum nozzle geometry	12	--
II-9	Increased L* studies	4	10
II-10	LMH-2/AP propellants	--	6
II-11	LMH-2/AN or HMX propellants	--	10
Phase B			
II-12	Characterization of selected Be and LMH-2 propellants	4	11

B. LABORATORY FORMULATION AND PROCESSING STUDIES

1. Beryllium Propellants

Several minor changes were made to the beryllium formulations shown in the first quarterly progress report.[1] These changes are as outlined below:

(a) The VIJ formulation was substituted for VIH in the AP particle size and nozzle contouring studies.

(b) The effects of oxygen source (total AP level) and higher oxidation level will be determined in VIH and VIK containing 45μ AP in place of 90μ AP.

(c) The VII formulation was modified to eliminate the use of toluene diisocoyanate (TDI) while maintaining the same oxidation ratio and metal level.

(d) The $TAGNO_3$ formulation VIG was substituted for VIQ. The VIJ, VIH, and VIK formulation changes were made to reduce in-process hazards associated with coarse AP particles in high NG systems. The VII change was made to improve physical properties and to eliminate the need for rigid moisture control required with TDI. The VIG formulation will be used to allow benefit of a limited number of lower pressure firings of this formulation under contract AF 04(694)-127.

(CONFIDENTIAL)

All laboratory work was completed on the beryllium analog propellants for the formulation screening phase of the program. The pertinent data are summarized in Table IV. (Unclassified)

2. LMH-2 Propellants

Laboratory formulation and process studies continued on the LMH-2 propellants selected for characterization based on the Task I correlations. Major emphasis was placed on the four propellants shown in Table V. Laboratory work consisted of evaluating the processing characteristics of "as received" and posttreated LMH-2 in the candidate formulations. The following LMH-2 materials were evaluated:

(a) As received, ground LMH-2

(b) Ground and vacuum baked, 4 hours at 100° C

(c) Wax coated

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[1] Ibid

TABLE IV

BERYLLIUM PROPELLANTS FOR IMPULSE EFFICIENCY STUDIES

	Propellant Type										
	VIH	VII	VIJ	VIJ	VIJ	VIK	VIL	VIM	VIN	VIO	VIG
Formulation (wt %)											
NC (PNC, 10μ)	10.0	15.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
NG	38.5	27.5	32.5	32.5	32.5	38.5	38.5	30.0	30.0	34.0	33.0
AP	32.5(45μ)	32.5(90μ)	37.5(45μ)	37.5(90μ)	37.5(180μ)	34.0(45μ)	18.0(45μ)	--	--	10.5(45μ)	8.0(90μ)
HMX (Class A)	--	--	--	--	--	--	18.0	--	--	33.0	--
AN (Hercules No. 3)	--	--	--	--	--	--	--	37.5	39.0	--	--
TAGNO3	--	--	--	--	--	--	--	--	--	--	31.5
Be (FP-17)	15.5	15.5	15.5	15.5	15.5	14.0	12.0	15.5	14.0	8.0	12.5
Res	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
2-NDPA	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
TA	1.5	7.5	2.5	2.5	2.5	1.5	1.5	5.0	5.0	2.5	--
ADN	--	--	--	--	--	--	--	--	--	--	2.0
Theoretical											
I_{sp}-100/14.7 (sec)	278.0	278.7	277.4	277.4	277.4	276.5	281.1	281.2	278.6	281.1	290.7
Tc (°K)	4173	3971	4145	4145	4145	4109	4008	3820	3744	3758	3633
ρ (gm/cc)	1.718	1.679	1.727	1.727	1.727	1.720	1.715	1.639	1.638	1.718	1.608
O.R.*	1.169	1.050	1.171	1.171	1.171	1.269	1.235	1.177	1.278	1.379	1.017
Sensitivity**											
Impact (cm/2kg)	11 (33)	11 (17)	3.5 (21)	3.5 (41)	6.9 (11)	3.5 (64)	3.5 (26)	3.5 (51)	11 (64)	3.5 (3.5)	3.5 (33)
Friction (lb @ ft/sec)	52 @ 8 (37 @ 8)	32 @ 6 (156 @ 8)	23 @ 6 (86 @ 8)	23 @ 8 (52 @ 8)	40 @ 6 (48 @ 8)	40 @ 8 (54 @ 8)	40 @ 8 (48 @ 8)	29 @ 8 (230 @ 8)	48 @ 8 (280 @ 8)	30 @ 8 (156 @ 8)	17 @ 8 (300 @ 8)
Electrostatic discharge (Joules)	>6.25 (>6.25)	>5.0 (>5.0)	0.250 --	>5.0 (>6.25)	0.250 --	>6.25 (1.25)	>6.25 (>6.25)	>6.25 (>6.25)	>6.25 (>6.25)	0.25 (0.25)	0.25 (>5.0)
Autoignition (°C)	204 (208)	211 (199)	212 (201)	214 (193)	218 (201)	200 (198)	207 (189)	200 (223)	204 (223)	213 (213)	214 (210)
Differential thermal analysis, ignition (°C)	172 (170)	174 (174)	162 (170)	173 (171)	176 (163)	170 (173)	173 (172)	170 (173)	175 (175)	166 (172)	162 (149)
Physical properties											
Tensile (psi)	103	96	--	98	--	96	66	59	88	--	125
Elongation (%)	32	34	--	23	--	39	43	22	22	--	20
Modulus (psi)	304	377	--	526	--	398	280	340	425	--	1000
Burning rates											
r @ 1000 psia	0.97***	0.57	0.75	0.75***	0.45	0.77	0.63	--	0.35	0.55	0.60
Exponent	0.49***	0.81	--	0.47***	--	0.50	0.59	--	0.65	0.61	0.50

*Oxidation ratio

**First number is for uncured propellant, number in parenthesis is for cured propellant

***Motor data, other data were obtained from strands

(d) AP treated

(e) AP treated with a wax overcoat

(CONFIDENTIAL)

TABLE V

LHM-2 FORMULATIONS

	Propellant Type			
	VIX	VIY	VIZ	VJA
Formulation (wt %)				
NC	10.0	10.0	10.0	10.0
NG	50.0	50.0	50.0	50.0
AP	23.0	21.0	19.0	--
HMX	--	--	--	24.0
LMH-2	15.0	17.0	19.0	15.0
Res	1.0	1.0	1.0	--
2-NDPA	1.0	1.0	1.0	1.0
Theoretical				
I_{sp} (1000/14.7) (sec)	303.7	308.9	313.8	309.3
Tc (^oK)	3678	3679	3672	3621
ρ (gm/cc)	1.356	1.319	1.284	1.355
Oxidation ratio	1.342	1.219	1.113	1.146

Previous work with conventional LMH-2 double-base propellants has shown that processibility becomes marginal at 15 percent LMH-2 loadings for most LMH-2 lots. As a result, post-treatment methods were investigated as a means to increase LMH-2 loadings. Of the various methods tried, wax coatings and recrystallization of AP/LMH-2 from an AP saturated water dispersion showed the most promise. Additional details of these posttreatments are given in Section 4. Table VI shows a summary of data obtained from 300 gram mixes of the candidate propellants. Analysis of the effect of posttreatments on processibility was complicated because of the necessity to use several LMH-2 lots each with its own processing characteristics. Figure 3 shows viscosity as a function of shear rate for three LMH-2 lots all ball-milled by the Ethyl Corporation for 30 minutes. In addition, all lots with the exception of lot 275-7 gave serious gassing in propellants made with "as received" material. The porosity problem was eliminated to a large extent in propellants containing AP by vacuum baking the LMH-2 for 4 hours at 100^o C and by using the wax and AP posttreatments. Vacuum baking and wax treating were not as successful with HMX propellants since the treatments only reduced the degree of porosity. Additional details of this anomaly are given in Section 4. (CONFIDENTIAL)

TABLE VI

LMH-2 PROCESS STUDIES

	Propellant Type								
	VIX	VIX	VIX	VIX	VIX	VIX	VIX	VIX	VIX
Mix No.	37-97	83-2	83-4	94-20	69-46	69-47	94-32	94-51	83-31
Mix size (gm)	300	300	300	300	300	300	300	300	300
LMH-2 lot	275-7G	275-7G	275-7	93-1G, 93-2G	93-1G	93-2G	93-1G	95A	275-7G
Special treatment*	G	G	--	G	G	G	G & B	BE	G, AP treated
Mix temperature (°F)	76	81	76	77	104	104	108	81	78
Viscosity @ 3 rpm	225,000	166,000	couldn't mix	303,000	116,000	129,000	200,000	49,800	100,000
Cure conditions (days/°F/psia)	5/120/30	5/120/30	--	5/120/30	5/120/0	5/120/0	5/120/30	3/120/30	5/120/0
Comments	nonporous	nonporous	--	porous	porous	porous	nonporous, but was poorly consolidated	few scattered voids	nonporous
Sensitivity**									
Impact (cm/2Kg)	3.5 --	3.5 --	-- --	1.0 --	1.0 --	1.0 --	3.5 --	-- --	-- (13.0)
Friction (lb @ ft/sec)	6 @ 8 --	10 @ 8 --	-- --	23 @ 6 --	30 @ 8 --	59 @ 8 --	40 @ 3 --	-- --	-- (17 @ 8)
Electrostatic discharge (Joules)	0.075 (0.625)	0.25 (0.25)	-- --	-- --	-- --	-- --	-- --	-- --	-- 0.625
Autoignition (°C)	205 (230)	225 (229)	-- --	204 --	203 --	202 --	200 --	-- --	-- 15
Taliani	42 (14)	-- (12)	-- --	18 (9)	21 (11)	43 --	12 --	-- --	-- (18)
Differential thermal analysis, ignition (°C)	-- (176)	178 (178)	-- --	172 178	177 --	179 --	176 --	-- --	-- (176)

* Special treatments
B = Baked 4 hr @ 100° C under 2 mm Hg
BE = Baked by Ethyl Corporation
G = Ground

**Plain number is for uncured propellant, number in parenthesis is for cured propellant

TABLE VI (Cont)

LMH-2 PROCESS STUDIES

	Propellant Type								
	VIY	VIY	VIY	VIY	VIY	VIY	VIY	VIY	VIY
Mix No.	83-52	83-56	94-44	67-57	94-55	94-1	94-47	69-58	94-54
Mix size (gm)	300	300	300	300	300	300	300	300	300
LMH-2 lot	90A	90A	93-1	93-1	93-1	90A	93-1	93-1	90A
Special treatment*	G	G, AP treated (X-110-54 / X-110-56} blend)	G & AP treated (X-110-74)	G, B, & AP treated (X-110-73)	G, B, & AP treated (X-110-73 / X-110-74} blend)	G, B, with 0.5% wax	G, B, with 1% wax	G, B, with 1% wax	G, B, with 0.5% wax
Mix temperature (°F)	91	76	86	105	72	80	74	100	72
Viscosity @ 3 rpm	>333,000	116,000	83,000	60,000	116,500	139,000	190,000	608,000	266,400
Cure conditions (days/°F/psia)	6/120/30	5/120/30	5/20/30	4/120/30	3/120/30	5/120/30	6/120/30	5/120/30	3/120/30
Comments	porous	few small voids	few small voids	nonporous	nonporous	few small voids	nonporous	residual solvent on wax, discarded mix	nonporous
Sensitivity** Impact (cm/2Kg)	1.0 --	1.0 --	1.0 (3.5)	3.5 (1.0)	1.0 (6.9)	3.5 (21)	1.0 (3.5)	-- --	1.0 (6.9)
Friction (lb @ ft/sec)	13 @ 8 --	68 @ 6 --	30 @ 4 (88 @ 8)	40 @ 6 (34 @ 8)	23 @ 4 (68 @ 8)	23 @ 4 (70 @ 8)	17 @ 6 (88 @ 8)	-- --	23 @ 3 (88 @ 8)
Electrostatic discharge (Joules)	0.625 --	0.625 --	-- --	-- --	0.075 (0.625)	0.25 --	-- --	-- --	0.25 (1.25)
Autoignition (°C)	217 (195)	236 (195)	205 (212)	204 (215)	203 (229)	219 (207)	203 (208)	-- --	207 (226)
Taliani	21 (40)	22 (45)	29 (18)	37 (14)	-- --	19 (21)	9 (15)	-- --	-- --
Differential thermal analysis, ignition (°C)	-- --	-- --	172 (176)	172 (175)	169 --	-- (175)	170 (178)	-- --	164 --

*Special treatments
B = Baked 4 hr @ 100° C under 2 mm Hg
BE = Baked by Ethyl Corporation
G = Ground

**Plain number is for uncured propellant, number in parenthesis is for cured propellant

TABLE VI (Cont)

LMH-2 PROCESS STUDIES

	Propellant Type					
	VIZ	VIZ	VJA	VJA	VJA	VJA
Mix No.	33-62	94-2	69-44	69-49	94-49	94-52
Mix size (gm)	300	300	300	300	300	300
LMH-2 lot	90A	90A	90A	93-1, 93-2	93-1	95A
Special treatment*	G, AP treated	G, AP treated with 0.5% wax	G, B with 0.5% wax	G	G, B with 1% wax	G, BE
Mix temperature (°F)	80	78	78	110	64	83
Viscosity @ 3 rpm	263,000	238,000	51,200	29,400	60,600	32,000
Cure condition (days/°F/psia)	6/120/30	5/20/0	5/120/0	5/20/0	5/120/0	5/120/0
Comments	Deaeration voids & cracks	Deaeration voids & cracks	porous	porous	few small voids & cracks	porosity & cracks
Sensitivity**						
Impact (cm/2Kg)	1.0 (3.5)	1.0 --	1.0 --	1.0 --	3.5 --	-- --
Friction (lb @ ft/sec)	10 @ 6 116 @ 8	23 @ 8 --	23 @ 6 --	13 @ 4 --	23 @ 8 --	-- --
Electrostatic discharge (Joules)	0.025 >5.0	0.625 --	-- --	-- --	-- --	-- --
Autoignition (°C)	231 (217)	217 --	223 --	219 --	222 --	-- --
Taliani	-- (37)	23 --	24 (10)	45 --	20 --	-- --
Differential thermal analysis, ignition (°C)	-- (179)	-- --	269 (263)	185 --	254 (267)	-- --

*Special treatments
B = Baked 4 hr @ 100° C under 2 mm Hg
BE = Baked by Ethyl Corporation
G = Ground
**Plain number is for uncured propellant, number in parenthesis is for cured propellant

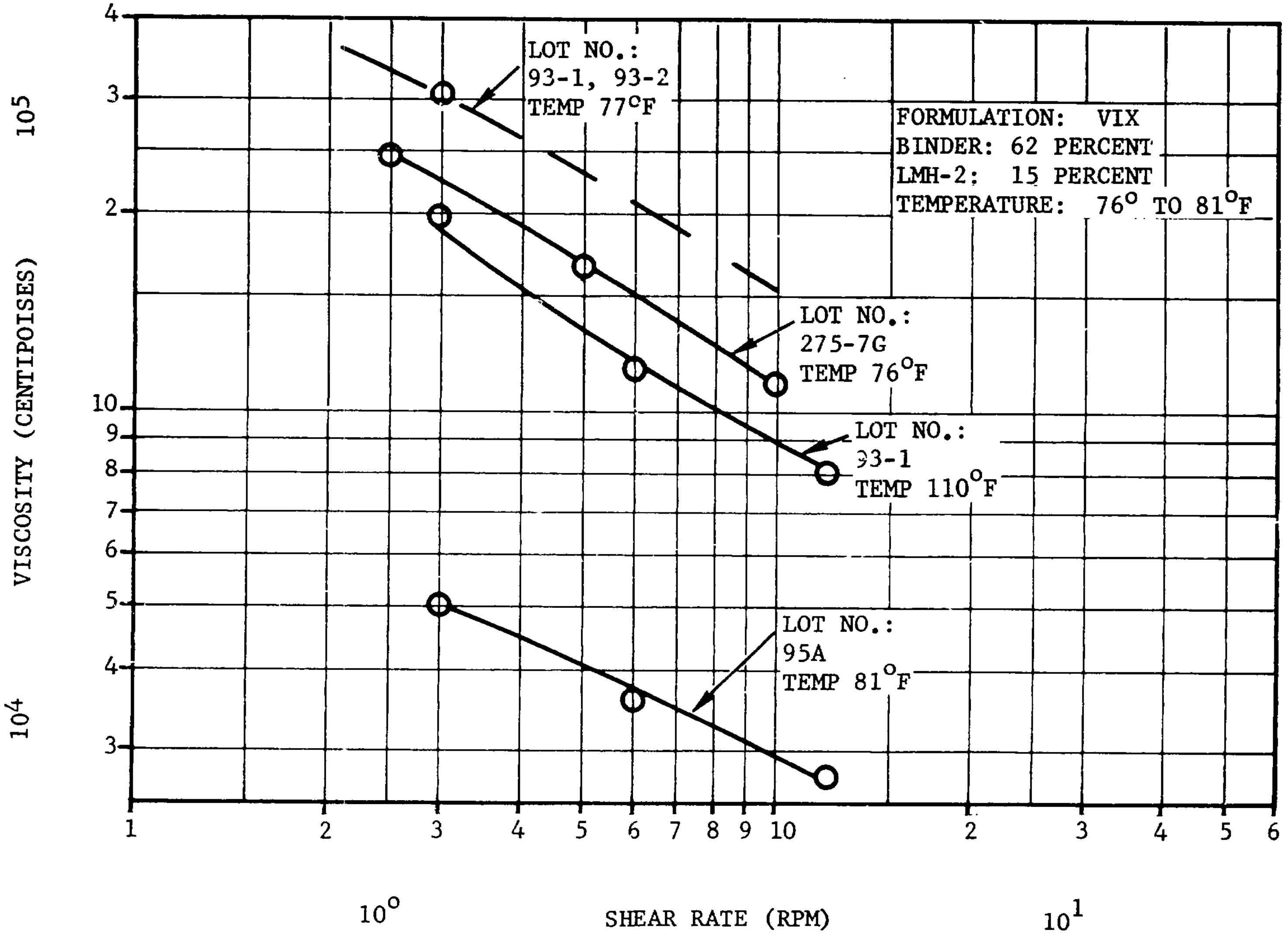

Figure 3. Viscosity as a Function of Shear Rate For Various Ball-Milled LMH-2 Lots

Figure 3 also shows that higher mix temperatures are effective in reducing viscosities for the VIX type formulation. The effect of wax and AP posttreatments for two LMH-2 lots in the VIY formulation (17 percent LHM-2) is shown in Figures 4 and 5. The AP treatment reduces viscosity by a factor of three while the wax treatment reduces viscosity by approximately a factor of two. Figure 3 shows that the AP treatment in VIY reduces viscosity much below that of VIX (15 percent LMH-2) probably closer to the level of 12 percent LMH-2 loadings. (CONFIDENTIAL)

LMH-2 loadings as high as 19 percent (VIZ) were successfully incorporated by a combination of wax and AP treatments. (See Table VI, Mix 83-62 and 94-2.) These mixes were very thixotropic, and additional techniques such as vibration under vacuum will be necessary to remove occluded air during casting for these high LMH-2 levels. (CONFIDENTIAL)

C. BALLISTIC TESTING

1. Motor Design

Modifications to the basic 15PC motor design were completed to maintain a mass flow rate of 5 lb/sec on all firings. The following grain designs are used in the program:

	Grain Design		
	15PC	15PC-1	15PC-2
Length (in.)	11.5	14.0	2(9)*
Web (in.)	1.3	1.0	0.75

* Two 9-in. grains are used
(CONFIDENTIAL

2. Castings and Firings

A total of thirty-nine 15PC beryllium grains were cast and eleven motors were fired during the report period. A summary of the castings and firings made is presented in Table VII. All of the castings were made without difficulties. (Unclassified)

A summary of the ballistic data obtained from the eleven firings is shown in Table VIII. A complete analysis of the ballistic data will not be made until additional firings are completed. However, the firings made to date show increased impulse efficiency over VCP with the high flame temperature propellants, VIJ and VIH. It should also be pointed out that good efficiencies can be obtained at high metal levels equivalent to 19 percent LMH-2 loadings. (CONFIDENTIAL)

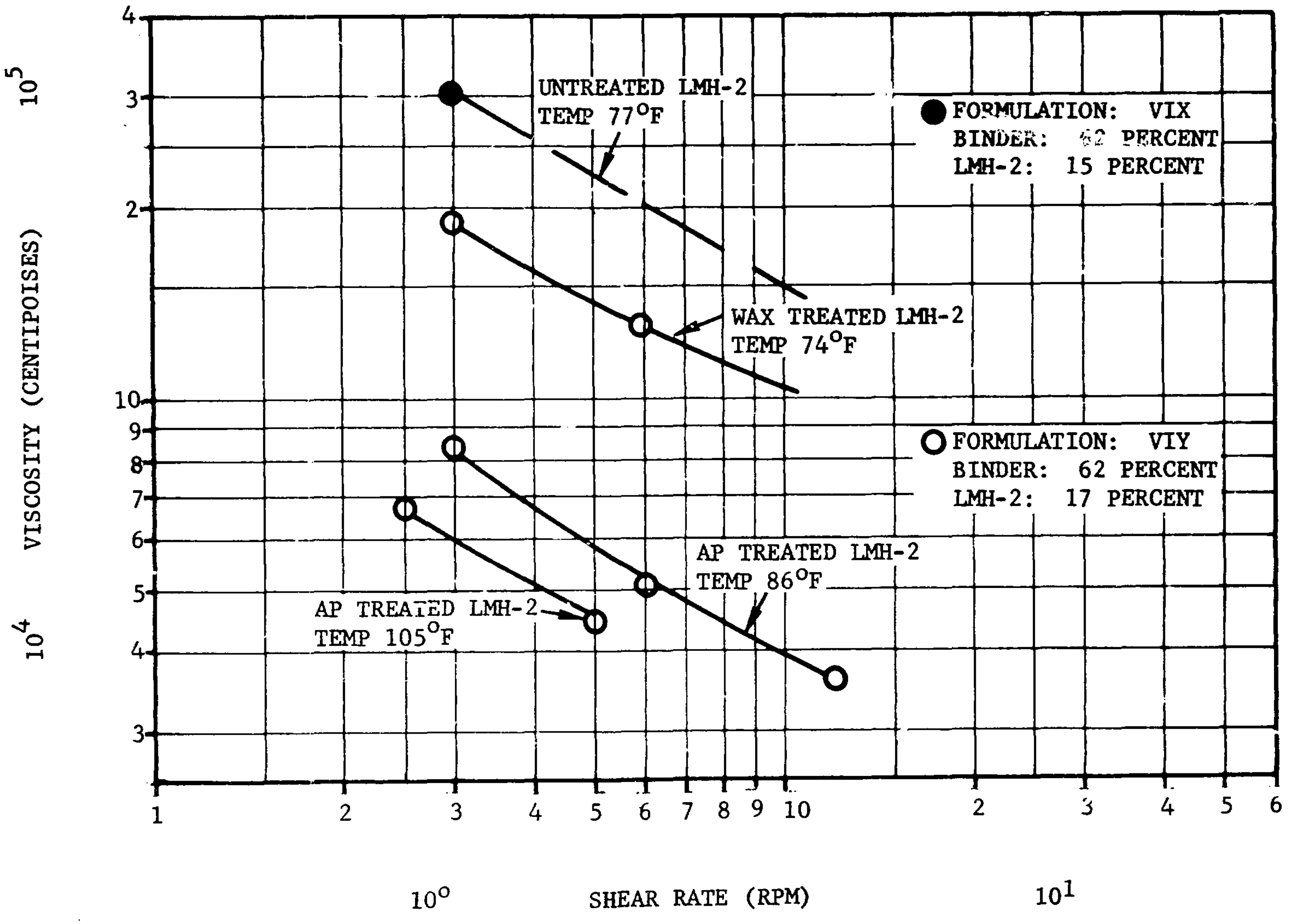

Figure 4. Viscosity as a Function of Shear Rate For Posttreated LMH-2, Lot 93-1

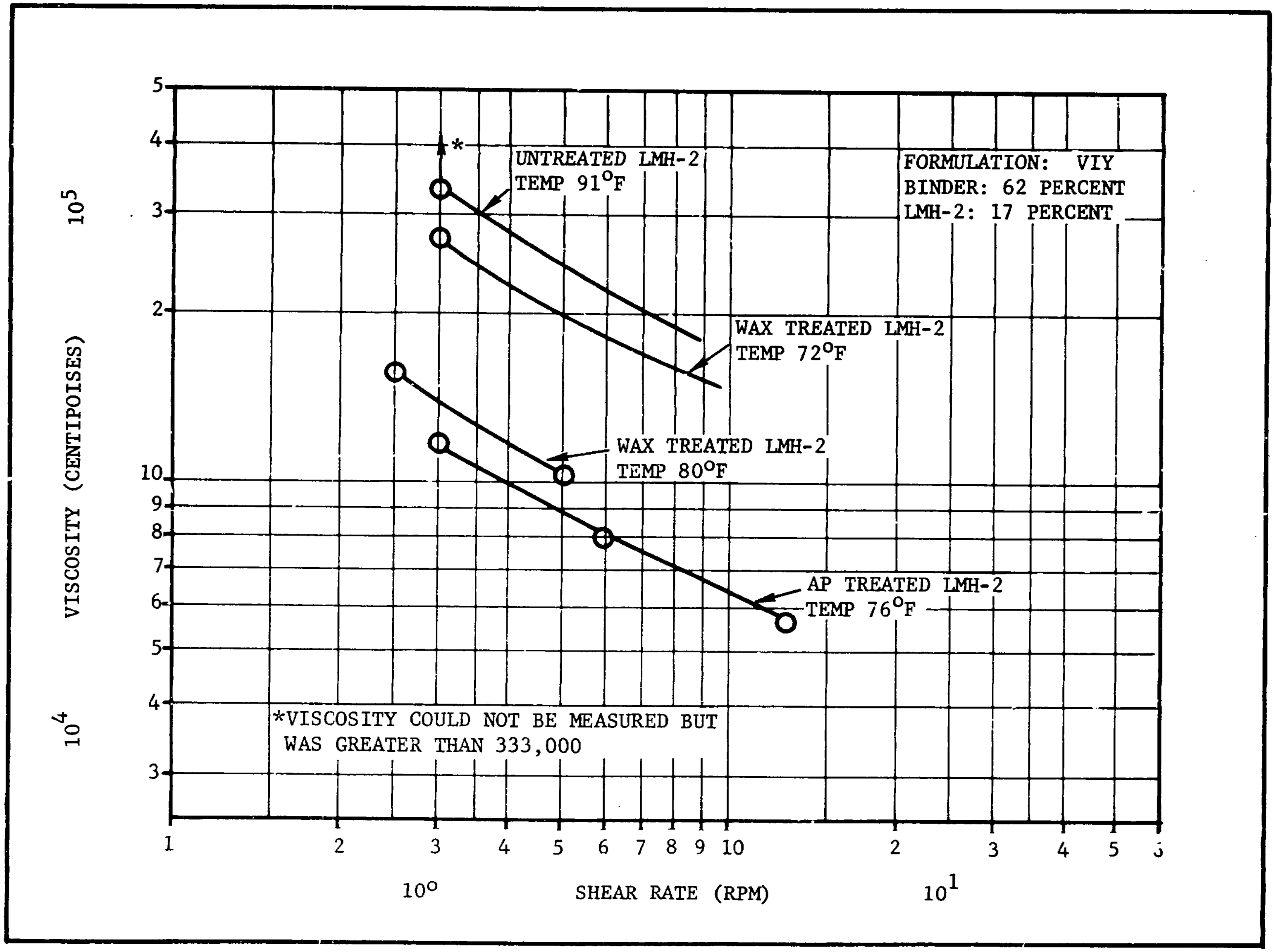

Figure 5. Viscosity as a Function of Shear Rate For Posttreated LMH-2, Lot 90A

TABLE VII

SUMMARY OF CASTINGS AND FIRINGS

Purpose	Propellant Type	Type Grain	No. of Castings	No. of Firings
Beryllium efficiency studies	VCP	Standard	3	1
		Mod-1	3	2
	VIH (45μ AP)	Standard	3	3
	VIJ (90μ AP)	Standard	3	2
		Mod-1	3	2
	VIK	Standard	3	1
		Mod-1	3	
	VIL	Standard	3	
		Mod-1	2	
		Mod-2	1	
	VIN	Mod-2	2	
Optimum nozzle geometry with Be	VCP	Standard	4	
	VIJ (90μ AP)	Standard	6	
Total			39	11

Two problems occurred in these firings. The first firing of the VIH formulation (LM2-3) gave a considerably higher pressure than was predicted based on strand burning rate data. The scale-up in pressure from strand data to motor data was allowed for in subsequent firings and, in general, the actual pressures obtained were close to design. The scale-up from strand to motor data is graphically illustrated in Figure 6 for the four VIJ firings. The large scale-up is believed to be due to the high concentration of metal used in these propellants resulting in poor efficiency in the strand burner. (CONFIDENTIAL)

A second problem occurred with a hangfire in the initial low pressure VIJ firing. No explanation other than the larger free volume of Mod-1 grain design, can be given for this anomaly. Future firings of the Mod-1 and Mod-2 grains will use a slightly larger igniter. (CONFIDENTIAL)

TABLE VIII

MOTOR FIRING SUMMARY

Propellant type	VCP-2111	VCP-2111	VCP-2111	VIH-5061	VIH-5061	VIH-5061	VIJ-5031	VIJ-5031	VIJ-5031	VIH-5031	VIK-5061
Motor design	15PC-1	15PC-1	15PC	15PC	15PC	15PC	15PC-1	15PC	15PC	15PC	15PC
Grain No.	S82B	S87B	S75B	S80B	S81B	S78B	S84B	S91B	S92B	S97B	S72B
Firing No.	LM2-1	LM2-6	LM2-7	LM2-3	LM2-8	LM2-9	LM2-2*	LM2-4	LM2-5	LM2-11**	LM2-10
Propellant wt (lb)	14.88	14.92	15.12	15.11	15.24	15.04	14.95	15.20	15.24	15.00	14.92
Powder wt (lb)	0.04	0.05	0.05	0.05	0.05	0.06	0.06	0.04	0.03	0.05	0.05
K (S/A_t)	104.0	103.3	143.2	139.8	110.5	109.2	101.8	140.5	141.3	128.6	109.6
t_a (sec)	1.884	1.874	1.817	1.148	1.322	1.350	1.824	1.694	1.830	1.927	1.227
t_b (sec)	1.794	1.770	1.715	1.043	1.274	1.280	1.686	1.593	1.721	1.875	1.165
$\bar{P}_a$ (psia)	578	586	1074	1604	1121	1085	566	1103	1019	892	1182
$\bar{P}_b$ (psia)	592	600	1103	1695	1144	1118	595	1141	1054	907	1216
$\bar{P}$max (psia)	657	650	1175	1885	1223	1202	671	1245	1145	999	1303
$\bar{P}$amb (psia)	12.41	12.12	12.10	12.41	10.20	12.19	12.41	12.11	12.10	12.00	12.08
$\bar{r}$ (in./sec)	0.557	0.565	0.764	1.246	1.028	1.023	0.593	0.822	0.761	0.693	1.115
$\dot{m}$ (lb/sec)	7.90	7.96	8.32	13.16	11.53	11.14	8.20	8.97	8.33	7.78	12.81
C_d (sec $^{-1}$)	0.00580	0.00578	0.00581	0.00613	0.00604	0.00603	0.00611	0.00608	0.00612	0.00610	0.00606
ϵ (Ae/A_t)	5.615	5.616	9.607	9.576	9.624	9.607	5.591	9.595	9.609	9.022	9.653
$\bar{F}_a$ (lb_f)	1939	1995	2209	3570	3088	2949	1979	2367	2180	1991	3221
Isp del $\left(\frac{lb_f\text{-sec}}{lbm}\right)$	245.6	250.7	265.5	272.1	268.1	264.8	244.2	263.9	261.9	255.8	265.1
Theoretical Isp at firing conditions	268.7	269.5	288.2	289.6	286.8	283.4	262.9	283.1	281.6	280.6	283.7
Efficiency	91.40	93.02	92.12	93.96	93.48	93.44	92.89	93.22	93.00	91.17	93.44
Isp $^{15}_{1000}$	258.7	263.2	260.6	261.2	259.9	259.8	257.7	258.6	258.0	252.9	258.4
Isp del (c)***	245.0	250.0	264.9	271.4	267.4	264.0	243.4	263.4	261.5	255.1	264.4
Efficiency (c)***	91.18	92.76	91.92	93.71	93.24	93.16	92.58	93.04	92.86	90.91	93.20
Isp $^{15}_{1000}$ (c)***	258.0	262.5	260.1	260.5	259.2	259.0	256.8	258.1	257.6	252.2	257.7

Notes: All efficiency values are calculated at the actual firing conditions

Isp $^{15}_{1000}$ and Isp $^{15}_{1000}$ (c) values were calculated by multiplying efficiency times the theoretical Isp at standard conditions

*4-second hangfire, all data is questionable

**Poor thrust-time trace data is questionable

***Corrected for powder embedment

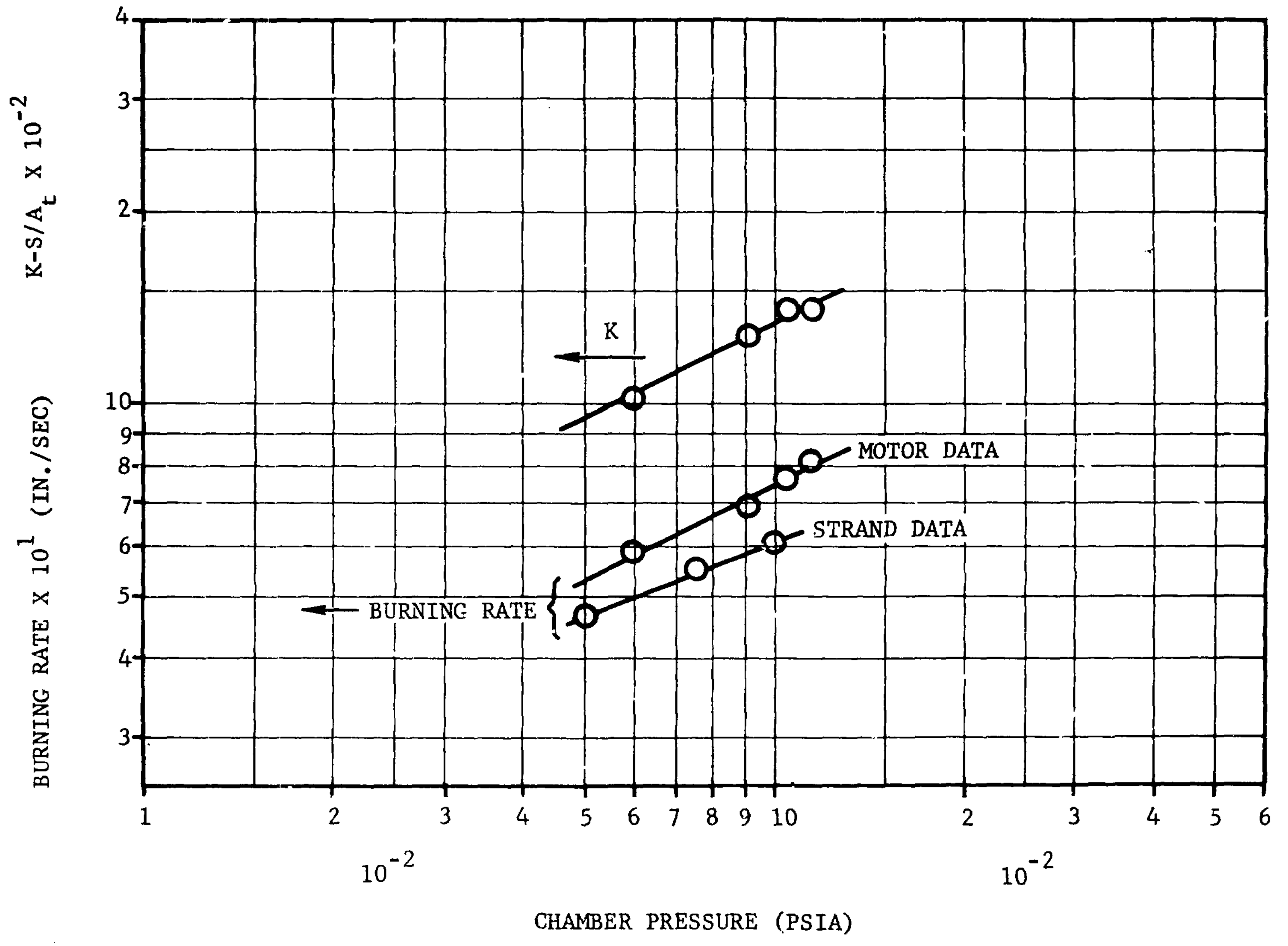

Figure 6. Burning Rate and K Data For VLJ-5031

3. Closed Bomb Tests

A series of closed bomb tests were performed on selected beryllium and LMH-2 propellants. Combustion bomb tests for residue analysis and micro-window bobmb tests for agglomerate size data were performed at Allegany Ballistics Laboratory using techniques previously employed under contract AF 04(611)-10742.[1] Heat of explosion tests were performed at Bacchus in a standard Parr bomb. Results of these tests are summarized in Table IX. Analysis of the data indicates the following:

(a) Based on the VIH and VIJ data, the decrease in combustion efficiency is negligible between 45μ AP and 90μ AP at high pressures. The data for 180μ AP is inconclusive but, based on agglomerate size, both VIH (180μ AP) and VIJ (90μ AP) may show decreased efficiency at lower pressures. In contrast, the efficiencies of VIL and VIO appear to be independent of pressure.

(b) The VIM and VIN data show AN oxidized systems to have a significantly lower efficiency than AP oxidized systems. Based on these data, only a limited number of AN firings will be made to confirm the combustion bomb results.

(c) For the LMH-2 propellants the VIX and VIY formulations gave good efficiencies at high pressures with a trend towards lower efficiencies at low pressures. The AP treated LMH-2 gave slightly lower efficiencies than ground LMH-2 in VIX and VIY. Only heat of explosion data were available on the VIZ and VJA formulations both of which showed lower efficiencies than VIX or VIY.

(CONFIDENTIAL)

At the present stage of development, the absolute values obtained from closed bomb data are questionable but they may normally be relied upon to establish reliable trends. Based on the bomb data to date, the optimum delivered impulse may occur with either the VIY formulation or the VJA formulation. As a consequence these two formulations will be investigated initially. Due to the differences in combustion efficiency noted between untreated or waxed LMH-2 and AP treated LMH-2, both posttreatments will be explored in motor firings. (CONFIDENTIAL)

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[1] Combustion of High Energy Solid Propellants, First Quarterly Report, Volume II, Report No. RPL-TDI-65-142, Contract AF 04(611)-10742, Hercules Powder Company (ABL), July 1965

TABLE IX

CLOSED BOMB DATA

	Beryllium Propellants								LMH-2 Propellants						
	VIH (45μAP)	VIH (90μAP)	VIH (180μAP)	VIJ (90μAP)	VIL	VIM	VIN	VIO	VIX	VIX(1)	VIY	VIY(2)	VIY(1)	VIZ(3)	VJA
Mix No.	37-40	37-93	70-92	37-95	37-99	70-73	70-87		37-97, 83-2	83-31	83-52	94-1	83-56		
Residue analysis(4)															
Pressure (psia)															
1000	100	100	--	100	100	77.0	83.3	99.8	100, 100	98.2		99.0	96.4		
400	99.7	99.6	--	100	100	43.8	50.9	99.5	96.4,97.5	93.3		95.8	89.4		
Window bomb agglomerate															
Size (mils)															
Pressure (psia)															
1000	<2	< 2	--	2.8	< 2	8.0	6.5	< 2	2.7, 3.4	3.6		4.1	4.2		
400	<2	< 2	4.5	4.6	< 2	15.8	14.1	< 2	3.3, 4.7	4.3		4.5	5.2		
200	Small Flaking	Small Flaking	6.0	Flaking	< 2	--	12.8	--	4.5, 4.3	4.6		--			
Heat of explosion (cal/gm) experiment(5)				2628, 2606		2172	1992, 2024	1955	2311, 2280	2324, 2296	2320	2353, 2330	2303	1866, 2337	2001, 2142, 2158
Heat of explosion (cal/gm) theoretical(5)				2654		2331	2205	1867	2270	2270	2384	2384	2384	2500	2217
Percent of theoretical				98.3 to 99.2(6)		93.2	90.5 to 91.6	105	101 to 102	101 to 102	97.3	97.7 to 98.7	96.6	74.6 to 93.5	90.2 to 97.5

Notes:

(1) Contains AP treated LMH-2

(2) Contains 0.5 percent wax on LMH-2

(3) Contains 0.5 percent wax over-coat on AP treated LMH-2

(4) Percent beryllium converted to BeO

(5) Heat of explosion values refer to formulation but not necessarily mix numbers

(6) Values were obtained on VIJ (45μ AP) and VIJ (180μ AP) respectively

SECTION 4

TASK III, ADVANCED CONCEPTS

A. SCOPE

The objective of Task III is to use advanced formulation or motor techniques to study and/or improve LMH-2 combustion. Six LMH-2, three beryllium, and one aluminum five-pound motor will be fired. The experiments in this task will be designed to evaluate the effect of the propellant combustion mode on performance. The task will include the following three major areas of effort:

(1) Characterization of LMH-2

(2) Modification of LMH-2

(3) Fluorine addition

(CONFIDENTIAL)

B. BACKGROUND

Based on the Task I correlations and theoretical calculations, two means of promoting higher efficiencies for LMH-2 propellants were originally selected for investigation under this task. The correlations showed that both decreasing the AP particle size and grinding LMH-2 would increase LMH-2 propellant efficiencies. This led to the belief that more intimate contact between the oxidizer and fuel might lead to additional performance gains. One means investigated was the pressing of LMH-2/AP binary mixtures. Work in the first quarter[1] showed pressing to be unattractive because of the poor physical properties of the pressed material and the correspondingly high pressures required for consolidation. Another means of achieving intimate contact, developed under independent research and development funding, was the recrystallization of AP/LMH-2 from an AP saturated water dispersion. Initial attempts at incorporating the AP treated material in propellant showed a marked improvement in processibility over "as received" LMH-2. As a result, emphasis was transferred from pressing LMH-2/AP mixtures to AP treatment of LMH-2. Other methods of surface treating LMH-2 are also being investigated. (CONFIDENTIAL)

In addition to more intimate contact to improve LMH-2 performance, theoretical calculations show that in fluorine/LMH-2 systems significant amounts of BeF_2 gas are formed, which may improve thrust efficiency by reducing particle lag effects. The possibility also exists that a

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[1] Ibid

fluorine environment may improve LMH-2 combustion efficiency. To study these possibilities, means of introducing fluorine into LMH-2 propellants are being investigated. (CONFIDENTIAL)

To provide support to the Task II and Task III efforts, a limited amount of work is also being done to characterize the chemical and physical properties of LMH-2 being used on this and associated contracts. (CONFIDENTIAL)

C. CHARACTERIZATION OF LMH-2

Three different lots of LMH-2 were used in the formulation phase of this program during the report period. In addition, data were available from two other lots used on associated programs. Table X shows a summary of data furnished on these LMH-2 lots by Ethyl Corporation. All of these lots, except 275-7, gave gassing in propellant mixes made with "as received" material. Because mixes made with Lot 91 had a strong, pungent, odor, a high volatiles content was suspected and confirmed by analysis. Subsequent lots also had this strong odor to varying degrees but did not, however, have exceptionally high volatile contents. Vacuum baking and/or post treatment by wax coating and AP treating eliminated the gassing for most lots. Due to the beneficial effect of vacuum baking, it was assumed that volatiles were the source of the gas. However, differential thermal analysis followed by gas chromatograph analysis (DTA/GC) of the various LMH-2 lots has failed to isolate the source of the gas or odor. The GC analysis of Lot 91 indicated the volatiles were mainly water. This was later confirmed by the Ethyl Corporation whose analysis gave the following volatile content:

Volatile	Content (%)
Water	70
Kerosene	5
Ethanol	20
2-Butanol	5
Air	Trace

(CONFIDENTIAL)

Further analysis by Ethyl Corporation has shown the strong odor to be associated with residual kerosene from the LMH-2 pyrolysis and has indicated the odor is probably caused by a sulfide or mercaptan. A strong odor would then be indicative of a high residual kerosene content. It is difficult to associate the gassing problem in propellants with any of the identified volatiles, except absorbed air, and additional work is needed before any definite conclusions can be made. To expedite handling in the future, all LMH-2 lots will be vacuum baked by the Ethyl Corporation prior to shipment. (CONFIDENTIAL)

TABLE X

ANALYSIS OF LMH-2 LOTS EVALUATED COMPOSITION (wt %)

Lot No.	LMH-2	Beryllium Metal	Beryllium Alkyls	Beryllium Alkoyides	Total Chloride	Volatiles	True Density
275-7	94.2	2.7	5.7	0.33	0.17	0.06	0.63
90A	92.9	2.2	2.5	0.14	0.38	0.23	0.62
91	91.9	1.3	2.9	0.33	0.81	1.0	0.62
93	92.6	2.3	3.1	0.15	0.42	0.09	0.63
94	94.0	2.0	3.0	0.73	0.67	0.05	0.64

Particle size analysis of ground as compared to unground LMH-2 is shown in Figure 7. Both ground lots were ball milled by the Ethyl Corporation for approximately 1/2 hour. As shown, lot 93-1 had an average particle size of 11 microns as compared to 16 microns for lot 90A. Typical unground material has a particle size of 35 microns. There was little difference in the processibility detected between the two ground lots. However, additional data are needed to specify the optimum grinding time from processibility considerations. (CONFIDENTIAL)

D. MODIFICATION OF LHM-2

Laboratory studies were conducted to determine the effect of processing variables on the quality and yield of "surface-treated" LHM-2. Three treatments were employed:

(1) AP treating, in which the oxidizer is first dissolved in hot water and then recrystallized in the presence of LMH-2

(2) Wax treating, in which a coating of wax is applied to LMH-2 from a volatile solvent

(3) AP treating, followed by a wax overcoat

(CONFIDENTIAL)

1. AP Treating

The initial preparation of AP-treated LMH-2 was accomplished by recrystallization of AP from a saturated aqueous solution of AP containing dispersed LMH-2. The resulting agglomerates were immediately filtered, and then they were washed with Freon and dried. By applying the temperature-solubility relation of AP in water, the ratio of the recrystallized AP to charged LMH-2 could be approximately predetermined. Laboratory mixes made with this AP-treated material showed significantly improved processing at

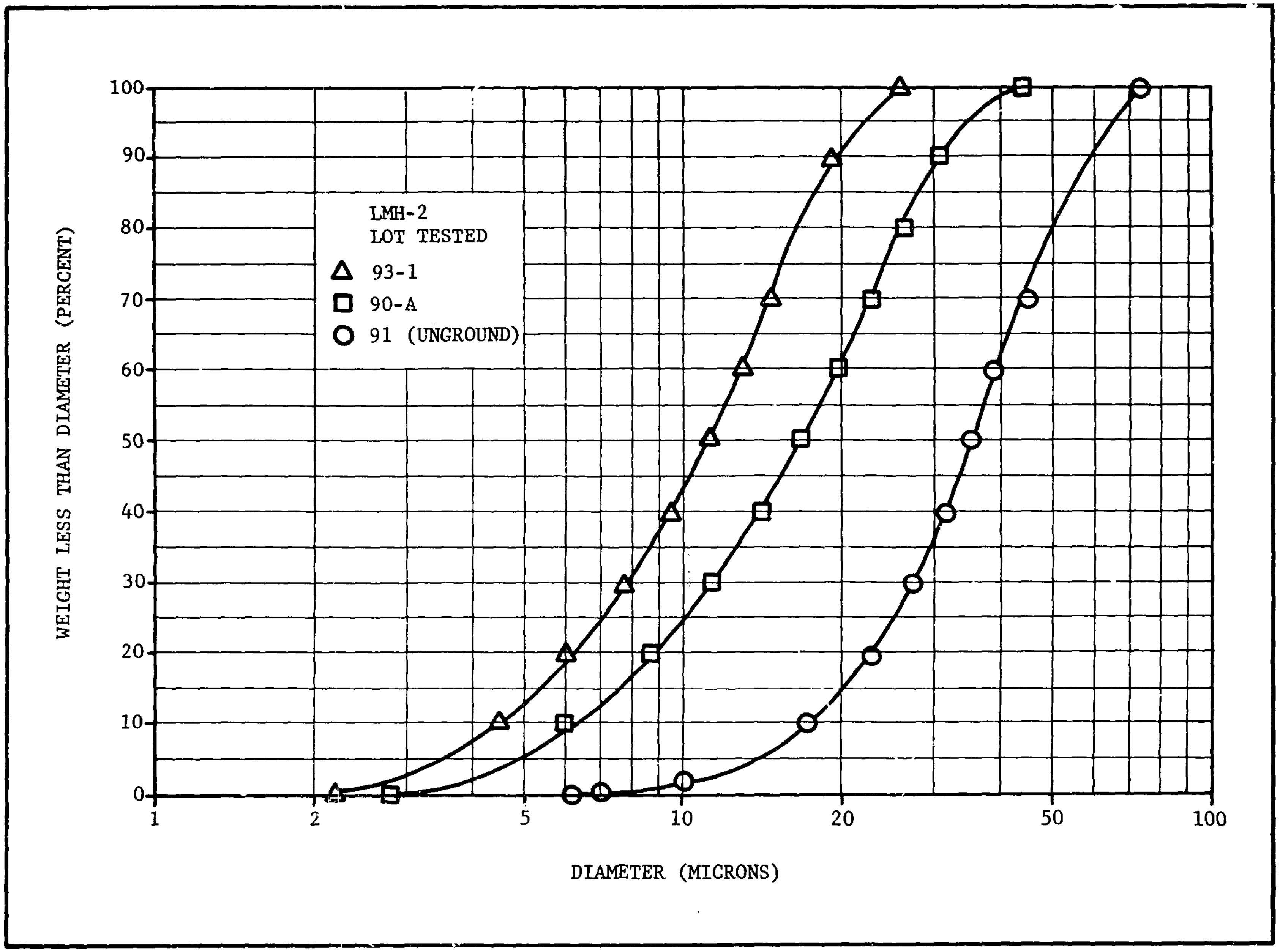


Figure 7. Particle Size Distribution Analysis For IMH-2 Lots by Micromerograph

15 to 19-percent LMH-2 loadings over those made with untreated LMH-2, and mix viscosities were lowered by a factor of three. (CONFIDENTIAL)

Work during the past quarter was oriented towards the following areas:

(a) Preparation and sensitivity testing of AP-Treated LMH-2

(b) Characterization of the AP-treated materials, by liquid separation techniques, chemical analysis, particle size analysis, and photomicroscopy.

(CONFIDENTIAL)

a. Preparation and Sensitivity Testing of AP-Treated LMH-2

Laboratory batches of AP-treated LMH-2 were prepared by temperature recrystallization and by crystallization through vacuum evaporation. A summary of the samples prepared is presented in Table XI. (CONFIDENTIAL)

In the temperature recrystallization method, a saturated aqueous solution of AP containing dispersed LMH-2 was heated to 60° C. The mixture was shaken vigorously to ensure that all the AP had dissolved. The mixture was then cooled as rapidly as possible to 20° C and suction filtered. The filter cake was washed twice with Freon-TA solvent and was dried for 16 hours under vacuum. A 1 percent addition of Triton X-100 surfactant was then applied to the AP-treated LMH-2, and the material was vacuum dried 16 hours at 120° F. Incorporation of this material into propellant mixes has consistently shown a reduction in propellant mix viscosity by approximately a factor of three over that obtained with untreated LMH-2. (See Section 3, Table VI.) (CONFIDENTIAL)

In the crystallization by evaporation method, a laboratory rotating evaporator (Rinco) was charged with an aqueous AP/LMH-2 dispersion. The water was stripped off by heating at 60° C under 14 mm Hg. The AP-treated LMH-2 material made by this method has not shown any improvement in propellant processibility over that obtained with untreated LMH-2. (CONFIDENTIAL)

The sensitivity data show the AP-treated LMH-2 material to be satisfactory for safe handling with impact values between 40 and 115 cm/2 Kg and friction between 10 lb at 6 ft/sec and 40 lb at 8 ft/sec. The sensitivity values show that wax-treated samples are comparable in sensitivity to the AP-treated LMH-2, but isopropanol-wet material may be more sensitive to impact and electrostatic discharge. No significant difference in sensitivity resulted from the two different methods of AP treated LMH-2 preparation. (CONFIDENTIAL)

TABLE XI

AP-TREATED LMH-2 RUNS

	LMH-2 Lot	Material Balance			I (cm/2Kg)	Sensitivity		Autoignition (°C)
		LMH-2 (gm)	AP (gm)	AP/LMH-2		F (lb/ft-sec)	ESD (Joules)	
Treated by Temp. Recrystallization								
X-110-50	275-76	10	12.6	1.26/1	> 115	40/6	0.025	> 300
X-110-51	90A	10	7.4	0.74/1	> 115	10/6	0.025	> 300
X-110-52-1	90A	10	6.5	0.65/1	80	21/6	0.025	> 300
X-110-54	90A	125	127	1.03/1	41	48/8	0.025	> 300
Isopropanol wet					21	40/8	0.00125	> 300
X-110-56	90A	75	111	1.45/1	> 115	38/6	--	291
X-110-58	90A	100	81	0.81/1				
0.5% CEC wax					41	50/6	--	> 300
1.0% CEC wax					> 115	38/8	0.025	> 300
2.0% CEC wax					80	38/8	--	> 300
X-110-66	90A	250	330	1.32/1				
X-110-73	93-1	100	120	1.20/1				
X-110-74	93-1	100	122	1.22/1				
Treated by Vacuum Evaporation								
X-110-62 R	90A	10	12.4	1.24/1	> 115	38/6	--	292
X-110-68 R	90A	10	12.4	1.24/1	> 115	40/8	0.050	> 300

b. Characterization of AP-Treated LMH-2

1) Microscopy

The AP-treated LMH-2 material was examined with a polarizing microscope under this contract and also under an independent research and development program. The photomicrographs are presented in Figure 8. Utilizing the birefringence phenomena of AP, the product was seen to be an agglomerate with the oxidizer forming the center. The LMH-2 particles appeared to be adhered to the surface of the AP crystal. The size of the agglomerates appeared to be approximately 75 to 150 microns. (CONFIDENTIAL)

2) Micromerographs

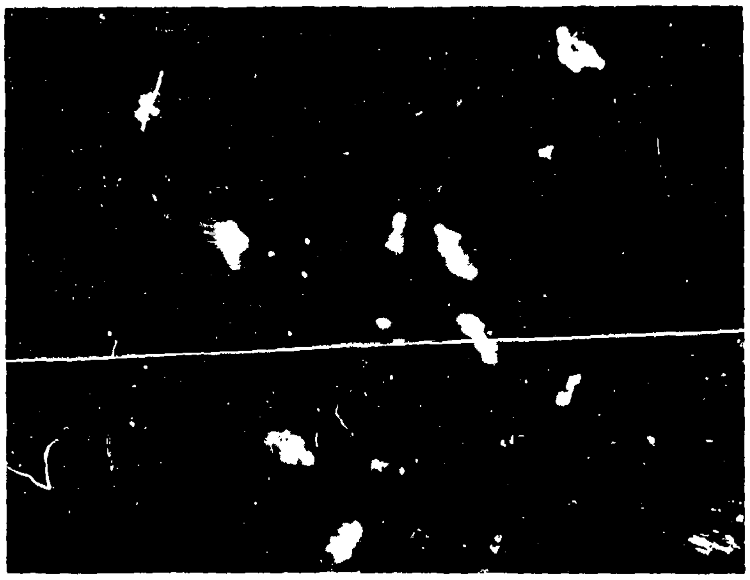
Micromerographs of AP-treated LMH-2 prepared by both the temperature recrystallization method and by crystallization through vacuum evaporation (Rinco method) were taken and are presented in Figure 9. The plots show that the particle size distribution were similar for the two methods, although dissimilar processing properties in propellants were observed, as discussed previously. The micromerograph data for the AP-treated LMH-2 give a somewhat inaccurate picture of particle size distribution, since the material tested consists of free LMH-2, free AP, and LMH-2 adhered to AP crystals and, consequently, results in variable composition and density. To overcome the inaccuracies of the micromerograph test, sieving tests are planned which should permit accurate measurements of the AP-treated LMH-2 particle size distribution. (CONFIDENTIAL)

3) Liquid Separation

Samples of AP-treated LMH-2 were separated with benzene into a light fraction (< 0.88 g/cc) and a heavy fraction of (> 0.88 g/cc). With the temperature-recrystallized material, the light fraction contained approximately 98 percent LMH-2, whereas the heavy fraction contained 5 to 8 percent LMH-2. Any LMH-2 in the heavy fraction would have to be adhered to the AP in some manner, since the density of LMH-2 is 0.65 g/cc. Based on the weight and analysis of each fraction, the separation indicated that the AP-treated LMH-2 is primarily free LMH-2 and free AP with only 5 to 10 percent of the total LMH-2 adhering to the surface of the AP. A run was also made in which the light fraction was recycled three times through the crystallization process, but the final combined product still only showed approximately 10 percent LMH-2 in the heavy fraction. (CONFIDENTIAL)

4) Chemical Analysis

The AP-treated LMH-2 was analyzed for active hydrogen to determine whether any LMH-2 decomposition had occurred during the crystallization process. The analysis showed the treated LMH-2 had the same active hydrogen content as the untreated LMH-2. (CONFIDENTIAL)



VIEW A. TEMPERATURE RECRYSTALLIZED AP/LMH-2 (100X)

VIEW B. VACUUM CRYSTALLIZED AP/LMH-2 (430X)

Figure 8. AP Treated LMH-2 Under Polarized Light

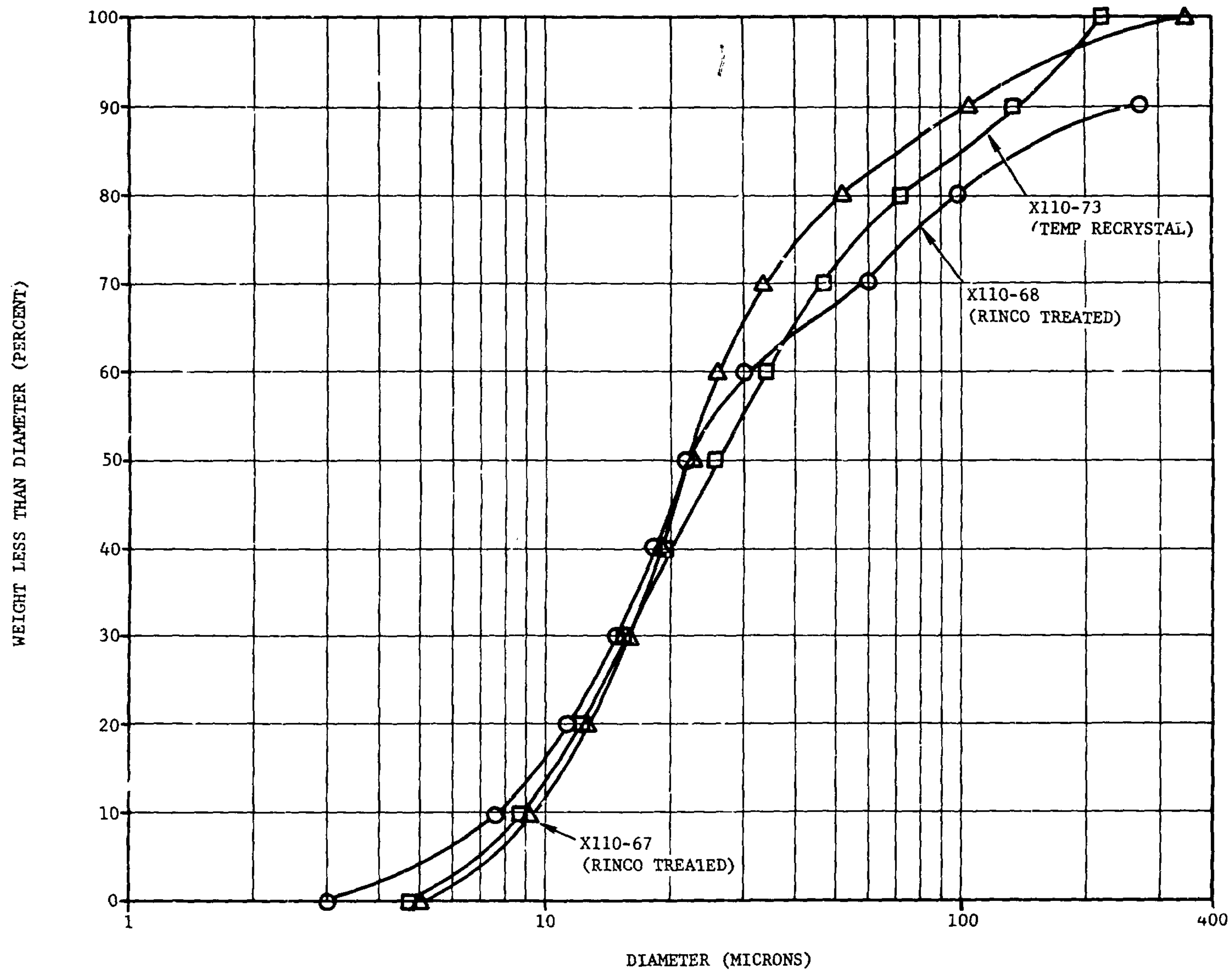


Figure 9. Particle Size Distribution Analysis by Micromerograph

2. Wax Coating Studies

Coating LMH-2 with a wax obtained from Consolidated Electrodynamics Corporation was found to improve propellant processing of LMH-2 propellants. (See Section 3.) The wax consists of shellac, rosin, and dibutyl sebacate. Wax coatings were applied to LMH-2 in which the weight percent of wax on the LMH-2 was varied from 0.01 to 10.0 percent. Optimum wax content for best processing with "as-received" LMH-2 was found to be 1 percent at the 17 percent LMH-2 level. (CONFIDENTIAL)

As-received LMH-2 and also vacuum-baked LMH-2 were wax coated by the addition of an acetone/isopropanol solution of the wax, calculated to give the desired percent of wax after the solvent was stripped off. Extended heated vacuum drying times were required to completely remove all traces of solvent which, when present, caused premature gelation of the nitrocellulose and excessively high mix viscosities. As the wax became coated on the LMH-2 surface, a hard outer shell formed that trapped some process solvent, causing solvent removal to be difficult. Heated vacuum drying removed the last traces of acetone-isopropanol solvent. (CONFIDENTIAL)

In the future, it is planned to investigate wax coatings applied to LMH-2 using a heated, jacketed "Vee-blender" under vacuum conditions. More uniform coatings and faster drying times are expected with the Vee-blender operation. (CONFIDENTIAL)

3. Wax Coatings Applied to AP-Treated LMH-2

Processing was improved for propellants when a wax coating was applied to the AP-treated LMH-2. This method enabled 19 percent LMH-2 levels to be processed satisfactorily in propellants. (See Section 3, Table VI, Mix 94-2.) (CONFIDENTIAL)

The procedure used for wax coating AP-treated LMH-2 was the same as that for wax coating plain LMH-2. It is possible that some dissolution and recrystallization of the AP may occur in the acetone/isopropanol solvent used for dissolving the wax. The solubility is low, however, and injurious effects are not expected from the short solvent contact times that were used with the AP-treated LMH-2. (CONFIDENTIAL)

E. FLUORINE ADDITION

Two methods of adding fluorine as a possible combustion aid will be investigated. Theoretical calculations with LMH-2 propellants containing binders based on interesting fluorine compounds have been performed. Typical formulations containing Hexakis (Difluoramino) Trivinoxy Propane (TVOPA) are shown below:

Ingredient (Wt %)	Formulation A	Formulation B
NC	10.0	10.0
TVOPA	35.0	35.0
LMH-2	15.5	14.0
AP	38.5	40.0
2-NDPA	1.0	1.0

Theoretical	Formulation A (Cont)	Formulation B (Cont)
Isp (sec)	309	307
T_c (°K)	3370	3410
Oxidation Ratio	1.00	1.09
BeO - moles/100 gr @ exit	0.92	0.904
BeF_2 - moles/100 gr @ exit	0.40	0.32

(CONFIDENTIAL)

Because of the availability, cost, and sensitivity, it is not practical to investigate TVOPA during this program. However, by the addition of teflon to LMH-2 propellants (either by pressing or by direct addition to conventional slurry mixes), product specie similar to that of TVOPA propellants can be obtained. Both pressing and direct addition of teflon will be investigated with formulations similar to those shown below:

Ingredient (Wt %)	Pressed Propellants			Direct Addition of Teflon		
NC	--	--	--	10	10	10
NG	--	--	-	50	50	50
Teflon	20	25	30	22	22	24
AP	65	60	55	4	2	2
LMH-2	15	15	15	14	16	14
Theoretical						
Isp	293	291	289	293	297	291
T_c	3602	3536	3461	3427	3381	3387
Oxidation Ratio	1.48	1.37	1.26	1.13	1.03	1.10
BeO moles/100 gm	1.09	1.02	0.926	0.94	1.02	0.90
BeF_2	0.21	0.29	0.38	0.28	0.38	0.33

(CONFIDENTIAL)

Small laboratory pressings and castings will be made and evaluated for physical properties and combustion characteristics. Based on these results, the best method of manufacture will be determined and scaled up to the 5-lb level. The following motors will be cast and fired:

Motor Size	Propellant Type	Number Units
5PC	Al/Teflon	1
5PC	Be/Teflon (~15% Be)	2
5PC	LMH-2/Teflon (~16% LMH-2)	3
5PC	Conventional LMH-2, ~16%)	3

(CONFIDENTIAL)

SECTION 5

INDUSTRIAL HYGIENE

The work associated with contract AF 04(611)-10754 progressed during this reporting period into additional buildings and areas resulting in an increase in the number of personnel under observation by the medical department. The medical examinations failed to reveal any symptoms which could be associated with Be. (Unclassified)

Protective clothing and safety equipment were provided for personnel performing operations involving Be. Specific types of clothing and equipment depended upon the potential hazard involved. A study is being conducted to determine the degree of contamination on clothing and equipment after a specific operation in an effort to adjust to a realistic protection requirement instead of overprotecting in all cases. (Unclassified)

A total of approximately 475 air samples were obtained from work areas in buildings where operations were performed involving Be compounds in the particulate form. These samples were analyzed to determine the concentration of airborne Be material to monitor both personnel exposure and protective equipment performance. (Unclassified)

All sampling data were well below the maximum acceptable concentration as outlined in Exhibit "A", pages 6, 7 and 8. All sample analysis data will be delivered to Captain Owen H. Kittilstad, Industrial Hygienist Officer, Edwards Air Force Base, California. The samples from the plant boundary samplers were sealed and placed in storage for future analysis if desired. The neighborhood sampling system was placed on remote control lines during this period and provides a centralized control point. These samplers will be activated automatically in the event of a fire or explosion on plant or at any time upon direction of the industrial hygienist. (Unclassified)

SECTION 6

FUTURE WORK

Work to be accomplished during the next report period will include the following:

(1) Casting and firing the majority of the remaining 15PC motors of the beryllium analog formulations as well as the 15PC motors for the nozzle approach and L* studies

(2) Casting and firing of initial LMH-2 propellants containing wax-coated and AP-treated LMH-2 at 17 percent LMH-2 loadings

(3) Additional work on surface treated LMH-2 with wax and AP posttreatments

(CONFIDENTIAL)

SECTION 7

CONCLUSIONS AND RECOMMENDATIONS

Conclusions drawn and recommendations made as a result of work done during this report period are listed below:

(1) Initial 15PC firings of high flame temperature beryllium propellants show increased impulse efficiency with increased flame temperatures even at high metal level (~15.5 percent).

(2) A serious gassing problem has occurred with new LMH-2 lots. Vacuum baking and/or surface treating has been effective in eliminating this problem with AP oxidized systems but not with HMX propellants. Additional emphasis should be placed on isolating the source of the gassing.

(3) Wax coating of LMH-2 and AP treating of LMH-2 have shown significant improvements in the processibility of LMH-2 propellants

(CONFIDENTIAL)