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ALUMINUM HYDRIDE PROPELLANT SHELF LIFE [®]

Lockheed Propulsion Company
Redlands, California

TECHNICAL REPORT AFRPL-TR-72-6

JANUARY 1972

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

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FOREWORD

This is the first Interim Technical Summary Report issued under Contract No. FO4611-71-C-0018 covering the period 15 December 1970 through 15 December 1971. This contract is assigned to Lockheed Propulsion Company, Redlands, California, and is monitored by Capt. Fred Clark, Air Force Rocket Propulsion Laboratory, Edwards, California.

The technical effort under this program is being performed under the supervision of Dr. W. E. Baumgartner (Program Manager) by Y. A. Tajima (Project Engineer) and L. K. Asaoka (Senior Chemist) within LPC's Chemistry Department. Work on the Mechanistic Analysis was carried out at the Midwest Research Institute under Subcontract 27-10072 by Dr. A. D. McElroy (MRI Program Manager) and Dr. R. E. Foscante (MRI Project Engineer).

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

The report contains information on the formulations and properties of advanced propellants, and it is therefore classified CONFIDENTIAL.

ABSTRACT

The 70°F shelflife of aluminum hydride propellants can be improved by adding small quantities of noble metal catalysts for condensing internally generated hydrogen gas. This program is designed to provide the factual demonstration that the resulting degree of stabilization is sufficient to assure a minimum five years shelflife under silo conditions.

To effect such demonstration, samples of a ballistically optimized AlH_3/NF_2 propellant have been placed into storage at 20, 30, 40 and 50°C to determine time-to-failure as a function of propellant composition and storage temperature to verify the models. Propellant off-gas composition and changes in the propellants' mechanical behavior are being monitored to derive the capability for shelflife predictions.

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GLOSSARY

AlH ₃	Aluminum hydride
Alon C	Aluminum oxide, submicron size, Cabot Corporation
AP	Ammonium perchlorate
COT	Cyclooctatetraene
DBTDA	Dibutyltindiacetate
Freon MF	Fluorotrichloromethane (Du Pont)
HT	1,2,6-hexanetriol
MRI	Midwest Research Institute
nBA	n-butylamine
NF ₂	Difluoramino
R-18	Multron R-18, poly(diethyleneglycol-adipate), hydroxy terminated, Mobay Chemical
S-4	R-18/TVO TM A based aluminum hydride propellant
SS-4	Same as S-4 except scavenger system added
Taliani	Gas evolution reactor/manometer
TDI	Toluene diisocyanate
TVOPA	Tris vinox propane bis(difluoramino) adduct (C)

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SECTION I

INTRODUCTION

I. GENERAL BACKGROUND

(C) Under Contract FO4611-70-C-0067, Hercules, Inc., is engaged in the scale-up of an advanced propellant that combines a hydritic fuel (aluminum hydride) with an energetic difluoramino plasticized (TVOPA); this propellant will have a delivered specific impulse approaching I_{1000}^{15} 270 lbf sec/lbm at a density of 0.061 lb/cu in., thus provide payload or range increases if used in the upper stage motor of a multistage ballistic missile. Concurrently with these scale-up efforts, Lockheed Propulsion Company (LPC), under this program, will demonstrate that this propellant, if processed into a large grain, will meet a minimum five years shelflife requirement under silo conditions.

(U) This demonstration of shelflife adequacy has been a major problem with aluminum hydride containing propellants. Aluminum hydride, even in its best available form, undergoes slow thermal decomposition with generation of hydrogen gas. Especially in a large grain where the diffusional loss of internally generated gas is slow, this will result in a gradual internal gas pressure build-up, thus grain failure by void or crack formation. The problem is aggravated if the aluminum hydride fuel is used in combination with difluoramino compounds, such as TVOPA, that give rise to additional gaseous degradation products, including HF that can degrade the binder polymer, thus impair the propellant's ability to withstand internal gas pressure.

a. Fluorine Free AlH_3 Propellants

(U) With fluorine free AlH_3 propellants failure results predominantly from hydrogen gas pressure build-up, and the rate of pressure build-up is strictly a function of hydride decomposition rates. This situation is amenable to theoretical and experimental analysis for the purpose of relating propellant shelflife to hydride decomposition rates. Such an analysis was performed by LPC under Contract FO4611-68-C-0023, and it was shown that even with the best available aluminum hydride the thermal degradation rates were in excess of what could be tolerated to assure a five years shelflife for large grains stored at 70°F.

(C) LPC then evaluated various principles for condensing the internally generated hydrogen gas, and it was found that carbon supported palladium catalysts in combination with suitable hydrogen acceptors could be used to react the internally generated hydrogen gas, thus retard failure, as long as hydride decomposition rates remained within the pre-acceleratory rate period. At 70°F this pre-acceleratory rate is maintained for periods of years, and since it was shown that the catalytic activity did not degrade significantly over periods of measurement extending to approximately one year, the test data gave reason to expect that with a fluorine free system a minimum five years 70°F shelflife can be achieved.

b. TVOPA Containing Systems

(C) If the aluminum hydride fuel is used in combination with TVOPA, propellant shelflife becomes a complex function of AlH_3 and TVOPA stability and interaction, as well as interaction of resulting decomposition products (e. g., HF) with the binder polymer. The mechanism and kinetics of the various processes that can result not only in internal gas (H_2 , CO, CO_2 , N_2 , NO_x , HF, H_2O , HCN, $(\text{CN})_2$) generation, but also in mechanical properties degradation, which have not been established sufficiently to enable the use of mathematical models for shelflife predictions or even temperature extrapolations. However, gas generation rate measurements and propellant surveillance studies performed under Contract FO4611-69-C-0038 show that the activity of the palladium catalysts is not impaired by TVOPA, or TVOPA degradation products. Even after a one year storage at 70°F hydrogen gas remained absent in the off gases, the latter being composed primarily of nitrogen, carbon dioxide and water. These gases, which derive from TVOPA decomposition appear to be formed at a sufficiently slow rate so that the critical pressure is not expected to be reached within a five years storage period at 70°F. This data extrapolation assumes that the "steady-state" gas evolution rates that are established after an initial three-four months storage at 70°F will persist to allow linear extrapolation, and it further assumes that there is no significant deterioration in the propellants mechanical properties.

c. Residual Problems

(C) The results that have been obtained under Contracts FO4611-68-C-0023 and FO4611-69-C-0038 provide theoretical and experimental evidence that R-18/TVOPA/ AlH_3 /AP propellants can be stabilized sufficiently to afford a minimum five years shelflife at 70°F. There remain uncertainties, however, that cannot be resolved on theoretical grounds, nor by short term laboratory tests, but require empirical resolution by long term surveillance studies. This pertains to the long term constancy in the overall rate of internal gas production, the ability of the catalytic scavengers to maintain their activity over a minimum three-five years time period, and the maintenance of adequate mechanical properties. These uncertainties will be resolved under this present Contract No. FO4611-71-C-0018.

(U) Another problem that must be considered upon initiating a long term surveillance program is the need for reliable quality control procedures; specifically, there is need to develop laboratory test methods that have a sufficiently short turn-around time to enable quality checks upon propellant mixes prior to casting. Alternatively, there must exist excellent awareness of critical material, formulation and processing variables so that close batch-to-batch reproducibility can be achieved.

(U) 2. PROGRAM OBJECTIVES

The purpose of this program is to effect the factual demonstration that a ballistically optimized AlH_3 / NF_2 propellant will afford a minimum five years shelflife at $70 \pm 20^\circ\text{F}$. This demonstration is to be accomplished by placing suitably sized samples into surveillance at 20, 30, 40 and 50°C ,

and by determining the time to failure as a function of temperature and sample size, and selected material, formulation and processing variables. Moreover, by collecting throughout the surveillance period data on gas evolution rates, changes in scavenger activity and changes in propellant mechanical properties, the program provides the data basis for the refinement of the mathematical models, thus the basis for quality control.

The propellants to be used in this investigation are to be similar or identical to the propellant being scaled and characterized under the concurrent program at Hercules, Inc.

(U) 3. PROGRAM ORGANIZATION

To accomplish its purpose the program must establish the various rate processes that control AlH_3/NF_2 propellant shelflife, specifically the rate processes that govern internal gas pressure rise, binder degradation, thus the ability of the propellant to withstand pressure, and it must provide further information on the physical failure mechanism so that the critical parameters can be adequately accounted for in the mathematical definition of shelflife. This is reflected in the program organization.

(U) a. Phase I - Long Term Surveillance

Samples (cylinders and elliptical tubes) of varying size, and representative of four formulations (control and scavenger containing propellants) are to be placed into surveillance at 20, 30, 40 and 50°C to determine the time to failure (crack or void formation, softening) as a function of formulation, storage temperature and sample size. Throughout the storage period the samples are to be monitored for gas evolution rates, changes in scavenger activity, and changes in mechanical behavior (uniaxial tensile properties, creep, tear behavior).

This will provide a factual demonstration of AlH_3/NF_2 propellant shelflife capability, and it will furnish the analytical data that are needed for relating time-to-failure to gas generation rates and changes in propellant mechanical behavior.

(U) b. Phase II - Shelflife Reproducibility

Under this phase of the program the effect of various material, formulation and process variables upon gas evolution rates, mechanical properties and time-to-failure will be determined. This will include the evaluation of changes in scavenger (catalysts) composition and method of preparation, of variability in aluminum hydride properties and methods of passivation, and a study of the effects of varying propellant processing conditions.

These data will form a basis for the establishment of material and process specification.

(U) c. Phase III - Mechanistic Analysis

While working with propellant only the end products of a complex sequence of chemical aging processes become detectable, and it is not always possible to relate the observed effect to a specific chemical event. Such ability for more detailed data interpretation is needed to develop practical quality control procedures; it is also needed to effect the necessary refinements in the mathematical models.

Under this phase an effort will be made to determine the mechanism and the kinetics of the major gas producing reactions, and of reactions causing binder degradation.

(U) d. Phase IV - Failure Modes

The existing mathematical models use the Lawson equation to define the critical gas pressure as a function of the propellant's uniaxial mechanical properties. This affords reasonably good agreement between predicted and observed time-to-failure if the internal gas pressure rise is comparatively fast, but it would not be adequate for defining the 70°F shelf-life of gas scavenger containing systems.

With most AlH_3/NF_2 systems studied to-date there is a significant time lag between a first indication of swelling (usually less than 5% volume change with no further change thereafter) and the appearance of cracks as evidenced by X-ray.

The Phase IV efforts will be concerned with the establishment of a more detailed description of the dependency of the failure process upon physical and chemical parameters to enable necessary refinements in the mathematical models.

(U) e. Phase V - Data Integration

Under this phase of the program the mathematical definition of aluminum hydride propellant shelflife that was originally derived for fluorine free propellants under Contract FO4611-68-C-0023 will be expanded to account for the more complex situation that exists with the R-18/TVOPA/ AlH_3 /AP systems. The specific goal here is to restore ability for temperature extrapolations to enable better use of accelerated surveillance tests.

(U) f. Phase VI and VII - Extended Surveillance

These additional program phases provide for continued surveillance of propellant remaining intact at the conclusion of the technical efforts in 1972, and they provide for the continued monitoring of off gases with propellants placed in 70°F storage under Contract FO4611-69-C-0038.

SECTION II

SUMMARY

1. OVERALL PROGRAM STATUS

(U) As a result of an incident which occurred in April 1971, and which caused considerable damage to the High Energy Propellant Laboratory besides resulting in the loss of the Phase I long term surveillance samples, the technical efforts under this program were considerably slowed down between April and October. Work on Phase III, Mechanistic Analysis, continued under a subcontract at the Midwest Research Institute (MRI), and reprocessing of the Phase I propellants was resumed in October and completed in November.

2. PHASE I, LONG TERM SURVEILLANCE

(U) All the Phase I long term surveillance samples have been processed, placed into storage at 20, 30, 40 and 50°C, and the analytical monitoring program was initiated.

(U) The samples (elliptical tubes ranging from 6 to 80 cm, cylinders ranging from 12 to 18 cm diameter and height) represent one scavenger free control propellant identical in composition to the Hercules HI-53018 propellant, and three gas scavenger containing modifications of this propellant.

(U) The propellant was processed in one-gallon mixes without using an inert diluent, vacuum cast into the appropriate size samples, and cured at 32-35°C. The cured specimens were X-rayed, and zero-time mechanical properties and IR reflectance spectra were obtained.

(U) The processing of this Phase I propellant proceeded smoothly, and there were no unusual events.

3. PHASE III, MECHANISTIC ANALYSIS

(C) A slurry type reactor system was used to study the individual rate processes if R-18, TVOPA and AlH₃ are heated singly or in various combinations. This work was performed at MRI under a subcontract, and the major conclusions to be derived from these studies are as follows:

- (U) (a) At temperatures below 40-50°C the total gas evolved by the the various mixtures is not significantly greater than the sum the gases evolved by individual components; it implies that there does not exist a significant incompatibility problem. Above 40-50°C interaction between the materials becomes more pronounced. This is reflected in an increase in the apparent activation energy as the reaction temperature is increased above 40-50°C, and it renders temperature extrapolation difficult.

- (U) (b) The initial gassing rates vary significantly with the conditions (e. g., mix time) that were used in preparing the slurries, and it would be very difficult to formulate kinetic expressions characterizing these early rate processes.
- (C) (c) Catalytic quantities of materials like DBTDA were shown to exert a significant effect upon the composition of the off gases, and pertinent matters should be studied further for the purpose of suppressing the formation of non-condensable gases, notably CO (potential catalyst poison).

4. PHASE VII, EXTENDED SURVEILLANCE

- (U) Propellant samples prepared under Contract FO4611-69-C-0038 and continued in 70°F storage since January 1970 have been analyzed for off-gas composition using gas chromatography. Data reduction is in progress.

5. ANALYTICAL METHODS DEVELOPMENT

- (U) The mass spectrometer method used previously for analyzing the composition of the off-gases has been replaced by gas chromatography. This became necessary since the mass spectrometer lacks precision in determining CO, CO₂ and N₂ concentration (interference by small air leaks or air contamination).
- (C) Multiple reflectance infrared analysis has been shown to be applicable to determine R-18 polymer degradation in R-18/TVOPA based propellants.

6. HAZARDS ANALYSIS

- (U) Data were produced to indicate that Freon MF if used as an inert diluent for the mix process could cause a high transient friction sensitivity, and the matter deserves further attention.

SECTION III

RESULTS AND DISCUSSION

1. PHASE I, LONG TERM SURVEILLANCE

(U) The surveillance and test specimens for this phase of the program have been processed and placed in storage at 20, 30, 40 and 50°C. The samples are being monitored by periodically performing dimensional measurements, X-ray, off-gas analyses, multiple reflectance infrared analysis, and by determining the time-temperature dependent changes in mechanical properties (uniaxial stress-strain, creep, and crack propagation). With the scavenger containing formulations any changes in catalyst activity and scavenger capacity will be determined by micro-hydrogenation tests.

a. Propellant Formulations

(U) The program calls for the surveillance of one control propellant and of three scavenger containing modifications, the basic formulation to be closely similar or identical to propellant being scaled at Hercules.

(C) Initially the Hercules VKW formulation had been used. This formulation has been replaced by the HI-53018 (Table I) formulation that does not contain HMX besides having a somewhat higher R-18 concentration.

(C) Omission of HMX was found desirable since in a scavenger containing system HMX can undergo reduction to amines that will then catalyze TVOPA decomposition. An increase in the R-18 prepolymer level was considered desirable as binder polymer degradation could prove to be a shelflife limiting parameter.

(C) The scavenger containing systems (A55-62-2, A55-62-3, A55-62-4) vary in the catalyst's palladium content, catalyst concentration and acceptor (COT) concentration.

b. Ingredient Preparation and Characterization

(C) The aluminum hydride was passivated by the Dow Chemical Company with n-butylamine/2% H₂O, and it was used as received. Analytical data supplied by Dow are quoted in Table II.

(C) The TVOPA was purified by the silica gel column procedure used by Hercules to afford a material with an NCO equivalence higher than 100,000.

(U) The AP was dried at 106°C for at least 48 hours in a circulating oven.

(U) The Multron R-18 and the 1,2,6-HT crosslinker were dried for at least four hours at 60°C in vacuo.

TABLE I
TEST PROPELLANT COMPOSITIONS
(Weight-Percent)

Ingredients	HI-53018	A55-62-2	A55-62-3	A55-62-4
12% R-18/88% TVOPA 3.44%/25.21%	28.65	28.65	27.695	27.695
TDI	1.08	1.08	1.044	1.044
1,2,6-HT	0.27	0.27	0.261	0.261
AP, Type II (0.3% Alon C) ¹	42.00	42.00	42.000	42.000
AP, 8 micron	9.80	9.30	9.300	8.800
AlH ₃ ²	18.00	18.00	18.000	18.000
CW-105 ³	0.20	0.20	0.200	0.200
30-Catalyst ⁴	----	0.50	----	----
Spiegler Catalyst ⁵	----	----	0.500	1.000
COT	----	----	1.000	1.000

1. Alon C coated on AP
2. nBA/2% H₂O passivated, The Dow Chemical Company
3. Cure catalyst, 10% DBTDL on molecular sieve; Linde Division, Union Carbide Corporation
4. 27.2% Pd/2.8% Pt/70% Shawinigan carbon black
5. 3.68% Pd/0.38% Pt/3.42% Fe⁺³/92.52% Shawinigan carbon black

TABLE II
ANALYTICAL DATA, DOW, AlH₃

LPC Lot No.	Dow Lot No.	Chemical Analysis							Decomposition Thermal Stability	
		% Mg	% Al	% Cl	% Li	% C	% H	% O	Days to 0.1%	Days to 1%
B 11	0062-Q3	1.18	85.9	0.20	0.18	0.23	9.73	2.25	14	36
B 13	0062-Q7	1.16	85.1	0.21	0.15	0.10	9.78	1.61	13	38
B 14	0028-Q20	2.18	84.2	0.05	0.47	0.64	9.64	2.39	23	150

(U) The catalysts were prepared as described earlier.* Microhydrogenation tests were performed to test for catalytic adequacy (see page 19).

c. Optimization of Cure

(U) Check-out mixes of the HI-53018 propellant were processed to optimize the TDI/HT cure system for mechanical properties. The miniature tensile specimens were cured at 90°F for ten days. The stress capability and strain at maximum stress optimized at an NCO equivalence ratio of approximately 1.20, Figure 1, and a triol/R-18 ratio of 0.6/0.4, Figure 2. The optimal physical properties were maximum stress of 103 psi and 24 percent strain.

d. Propellant Processing

(C) To produce the necessary surveillance samples (Table III) five one-gallon mixes had to be processed for each formulation. The mix schedule that was used is shown in Table IV. The mix procedure was as follows:

- (C) (1) The Multron R-18/TVOPA (12/88 wt %) is stripped of the CH_2Cl_2 by purging with nitrogen overnight and then evacuating at 60°C for four hours in a solvent stripper. The premix is added to the mix pot.
- (C) (2) The AP and the 1,2,6-HT crosslinker are added, and the system is mixed for five minutes without vacuum, then for 20 minutes at 2 mm Hg.
- (C) (3) The TDI curative and the CWX-105 cure catalyst are added, and the system is mixed as under (2).
- (C) (4) The aluminum hydride and the scavenger is added, and the system is mixed as under (2).
- (U) (5) The mix is vacuum cast into the appropriate molds using vibration.

Mixing, casting and cure are performed at 90-95°F, and all operations are performed in a facility whose atmosphere is controlled at a -20 to -40°F dew point.

(U) To date in excess of 50 one-gallon mixes of this type of propellant have been performed without experiencing difficulties.

(U) The individual surveillance samples were sealed in polyethylene bags containing silica gel drying bags. Separate samples were cast from each mix for determining zero-time mechanical properties; subsequent mechanical property measurements will be performed from samples cut from elliptical tubes.

(U) * Spiegler, U.S. Patent No. 3,265,636 (assigned to E. I. du Pont de Nemours Co.).

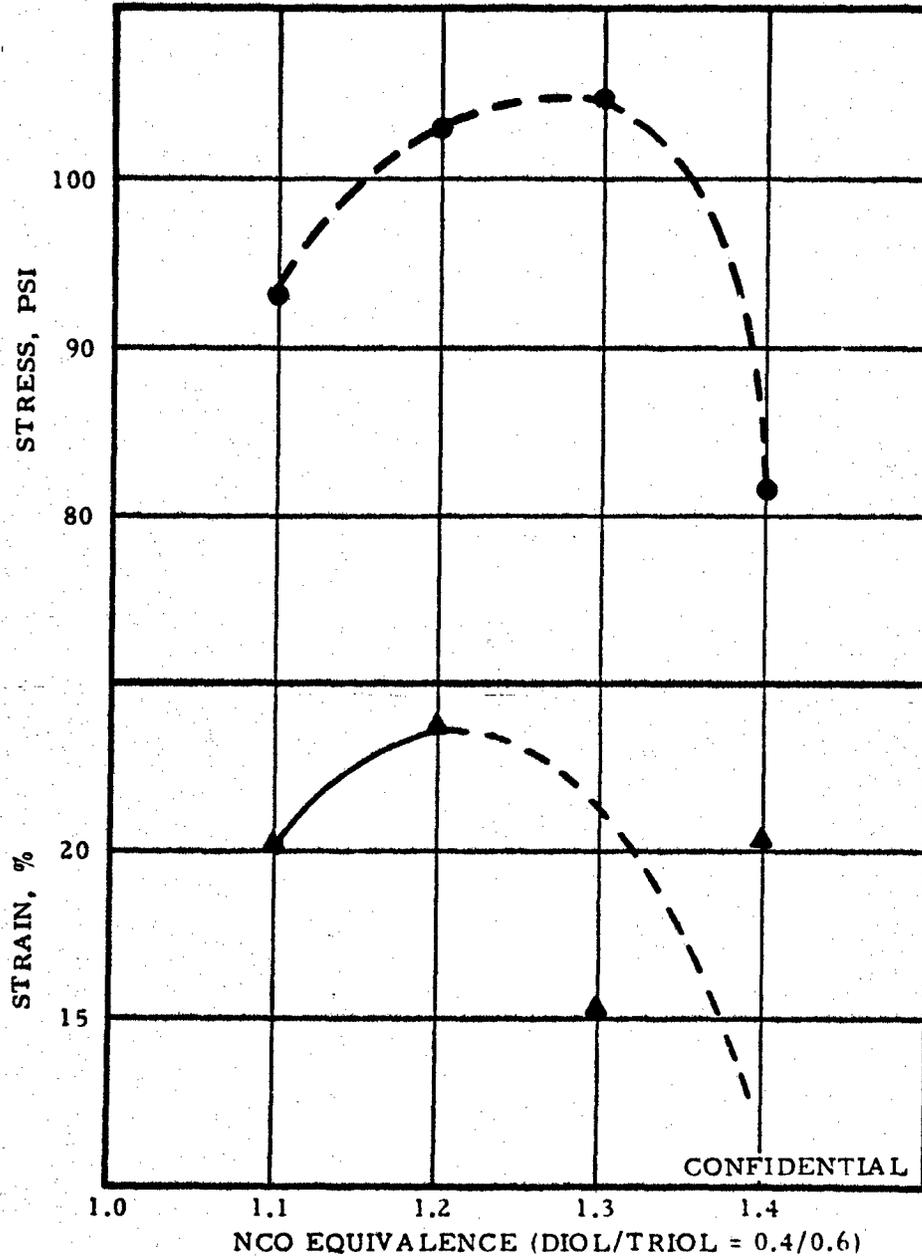


Figure 1 HI-53018 Propellant, Silica Gel Treated TVOPA TDI Curative Study

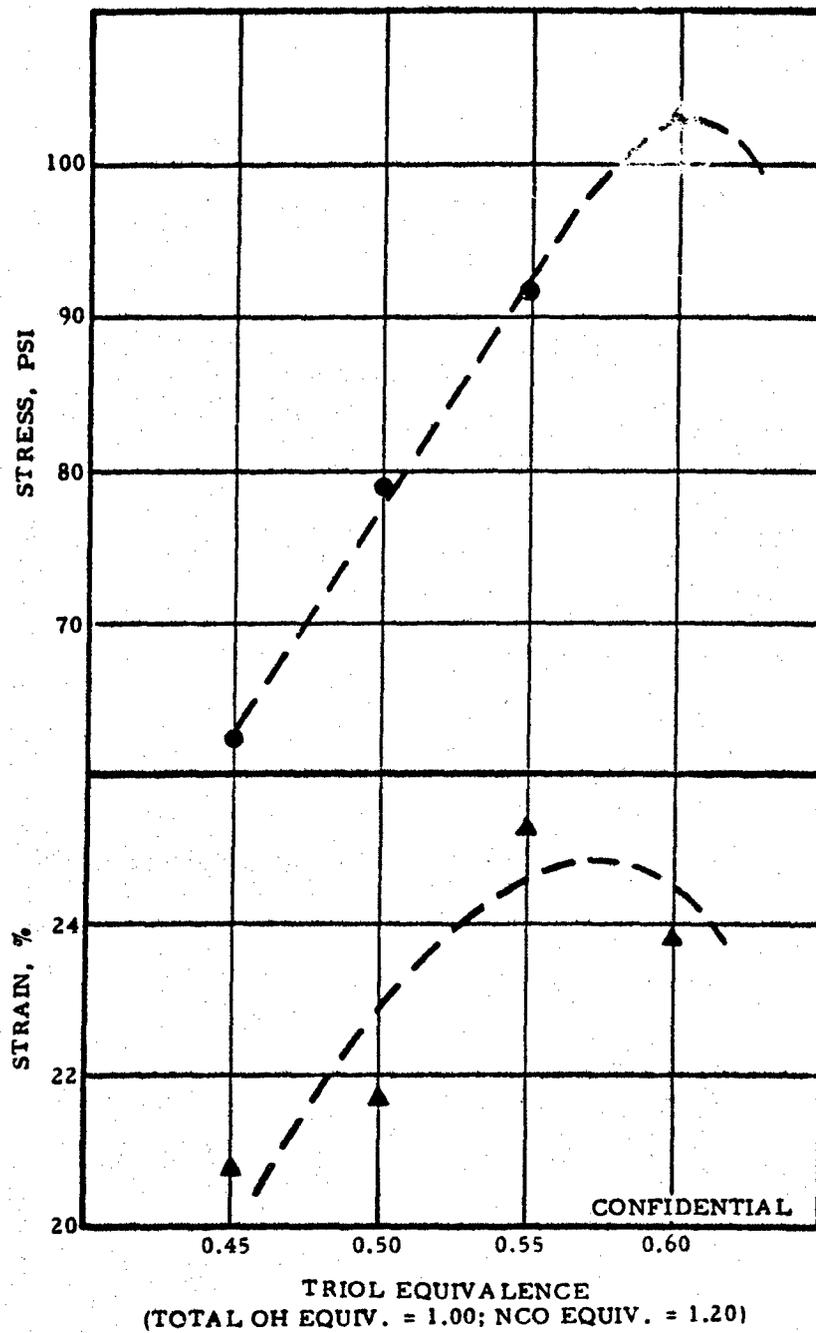


Figure 2 HI-53018 Propellant, Silica Gel Treated TVOPA 1, 2, 6 Hexane Triol Crosslink Study

TABLE III
 SCHEDULE OF SURVEILLANCE AND TEST SPECIMENS
 PER PROPELLANT COMPOSITION

Surveillance Temperature (°F)	Surveillance Specimens	
	Elliptical Tubes (cm)	Cylinders (cm)
68	40, 50, 60, 80	
86	18, 24, 30, 40	
104	12, 18, 24, 30	12, 18
122	6, 12, 18, 24	

TEST SPECIMENS

Mechanical Properties:	18 cm elliptical tubes at 68, 86, 104, 122°F
Scavenger capacity and catalyst activity:	18 cm elliptical tubes at 68, 86, 104, 122°F
Gas solubility and diffusivity:	18 cm elliptical tubes at 68, 86, 104, 122°F
Crack propagation and creep:	30 cm elliptical tubes at 68, 86, 104, 122°F

TABLE IV

PROPELLANT MIX SCHEDULE PER FORMULATION

Specimens	No.	Mix No.				
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
Elliptical Tubes, cm						
6	2	1	1			
12	4	2	2			
18	40	11	11		9	9
24	6	3	3			
30	22	5	5	12		
40	4	2	2			
50	2	1	1			
60	2	1	1			
80	2	1	1			
Block, 2" x 2" x 3"		1	1	1	1	1
Cylinders, cm						
17					← 1 →	
12				1		

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e. Elliptical Tube Preparation

(U) To conduct a long term surveillance study with aluminum hydride propellants it is necessary to use sample dimensions that simulate large grains as regards the diffusional path length. This calls for excessive quantities of propellant if cubes or cylinders are used, and it forced adoption of the Rohm and Haas elliptical tube sample configuration as a means of economizing on propellant.

(U) With these elliptical tube specimens the quality of the propellant-liner-tube bond is critical as debonding will drastically reduce the diffusional path length, thus lead to erroneous results.

(U) Several liner systems were evaluated using peel tests and the failure mode (cohesive, adhesive) was determined. Without liner the failure was cohesive/adhesive, and peel time increased with 50°C storage up to 150-200 days, then dropped drastically (Figures 3 and 4). With all liners studied the failure was adhesive, and none of the systems afforded more permanency in bond strength upon 50°C storage. As a result, no liner was used in processing the elliptical tube specimens. Considerable care, however, was taken in preparing the tubes. The method that is being used is as follows:

- (U) (1) The tubes used are Alcoa aluminum tubes with an outside diameter of one inch, and a wall thickness of 0.016 inch. They are cut to length and then are forced through a mandrel to produce an elliptical shape with minor x major axes of 0.62" x 1.24".
- (U) (2) The individual tubes are sand blasted, rinsed, then etched with ten percent NaOH at 120°F and rinsed with water.
- (U) (3) They are then dipped into dichromate sulfuric acid solution, rinsed again with water and dried at 110°C for two days.
- (U) For filling the tubes with propellant, a number of the tubes is bundled with tape and placed into an appropriate container for vacuum casting.

f. Sample Monitoring**(1) Test Procedures**

(U) The tests to be performed and the test frequencies are outlined in Table V.

(a) Gas Evolution Rates

(U) To determine the gas evolution rates at 40 and 50°C, replicate samples of the various propellant mixes are placed into standard Taliani manometers to monitor the pressure rise. For the 20 and 30°C samples sealed manometers are used to circumvent problems with leaks.

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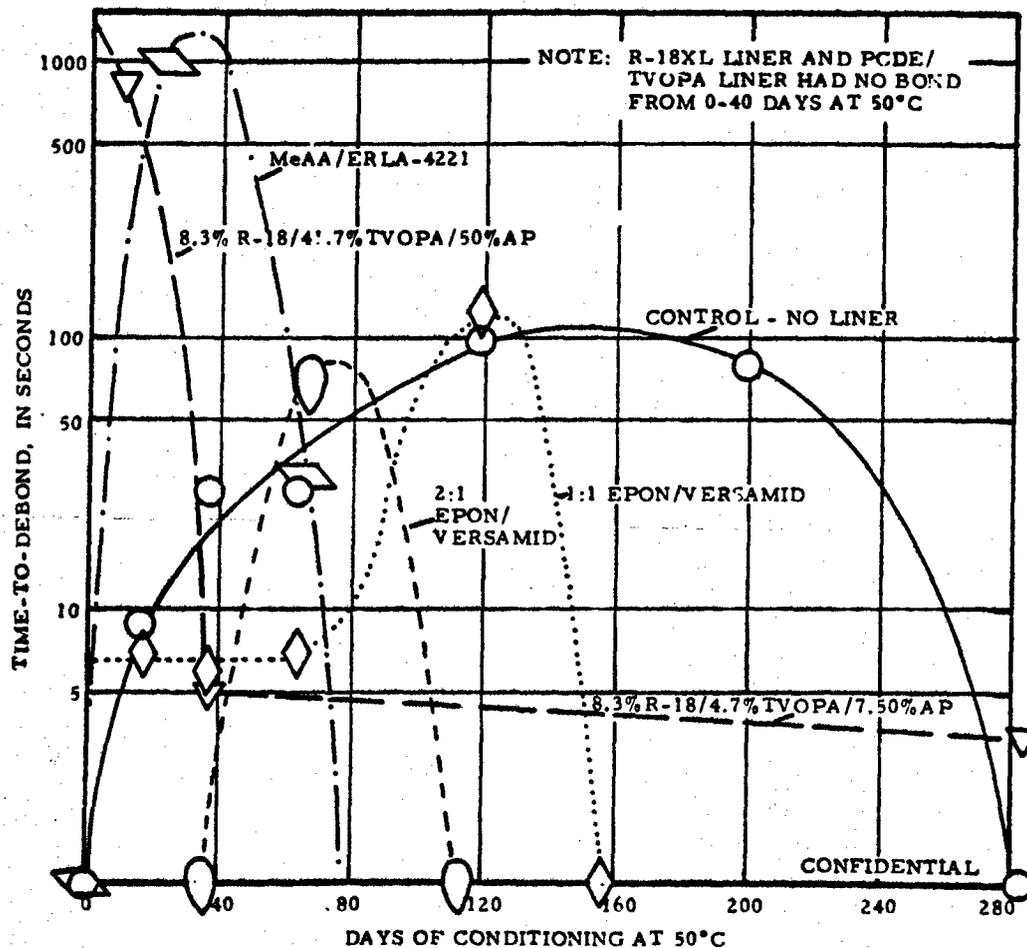


Figure 3 Liner Study: S-4 Propellant/Aluminum

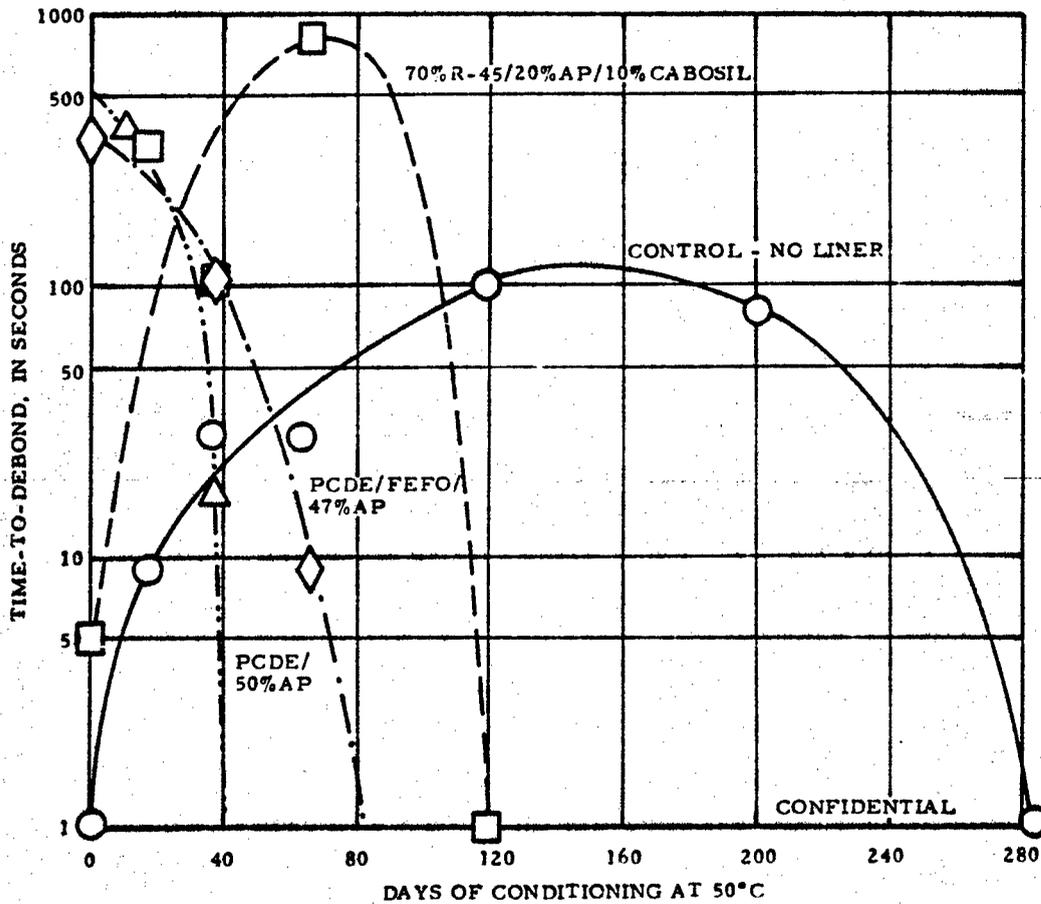


Figure 4 Liner Study: S-4 Propellant/Aluminum

TABLE V
TEST SCHEDULE FOR PHASE I

Type Test	Frequency, days between tests				Comments
	68°F	86°F	104°F	122°F	
a. Gas Evolution Rates	60	30	15	7	These test periods are approximate and depend on zone of gas evolution curve.
b. Off-Gas Analysis					
c. Dimensions & X-Ray	30	30	14	7	
d. Mechanical Properties					To be scheduled
Stress/Strain	300	150	100	75	
Creep	300	1/S/C	1/S/C	1/S/C	
Crack Propagation	300	1/S/C	1/S/C	1/S/C	
Hardness (TMA)					
e. Binder Polymer Degradation					
MRIR	1/C	1/C	1/C	1/C	
f. Gas Solubility and Diffusivity		1/S/C		1/S/C	
g. Scavenger Capacity/Activity	1/C	1/C	1/C	1/C	

NOTE: 1/S/C = initial, at swell and at crack failure time.
1/C = initial and at crack failure time

(b) Off-Gas Analysis

(U) A gas chromatographic method is used to determine the composition of the off-gases, and a combination of gas chromatography - mass spectrometry is used for identifying components of the mixture.

(U) To determine off-gas composition with propellant stored at 40 and 50°C, gas is being sampled from the Taliani sets. For determining off-gas composition with propellant stored at 20 and 30°C, samples of the various propellants were sealed into glass ampules.

(C) The off-gases that are being determined are H₂, O₂, N₂, NO, N₂O, CO, CO₂, H₂O, HF, HCN, CH₄, C₂H₄ and C₂H₆.

(c) Dimensions and X-Ray

(U) Changes in the dimensions of the elliptical tubes are detected by passing the tubes through the standard die; cylinders are tested by measuring circumference.

(U) Standard X-ray procedures are used to determine crack formation.

(d) Mechanical Properties

(U) The uniaxial stress strain values are determined with minithin samples (0.1" sample thickness) that are cut from the center of elliptical tube samples. Center cut propellant samples are also used to determine creep under constant load, tear resistance and changes in hardness.

(e) Binder Polymer Degradation

(U) Work recently completed under Contract FO4611-69-C-0038 has shown that the rate of hydrolysis of the Multron R-18 ester linkages can be monitored in the propellant by multiple reflectance infrared analysis. This technique therefore is being applied under Phase I.

(f) Scavenger Capacity and Activity

(C) Existing data indicate that the R-18/TVOPA binder matrix contains groupings that can be hydrogenated in the presence of palladium catalysts, thus serve as a hydrogen acceptor; it implies that an additional acceptor, such as COT, may not be needed, or may be needed only in small concentration to provide an additional hydrogen sink during the cure process.

(U) To determine the long term adequacy of the catalytic scavengers the following tests are being performed periodically:

- (1) Samples of the propellants are cut and placed under hydrogen atmosphere in a microhydrogenator to determine changes in the total volume

of hydrogen taken up, and of related rate phenomena.

- (2) The exhaustively hydrogenated samples (from (1) above) are then immersed into a COT solution in alcohol, and hydrogenation is repeated; this provides data on changes in catalyst activity.

(g) Gas Solubility and Diffusivity

(U) The gas solubility and diffusivity in the propellants will be measured by a permeation method at 30 and 50°C. In this method, the volume of gas passing through the membrane, which has been cut from bulk propellant, is recorded as a function of time. The diffusivity and solubility are calculated from the steady state rate and the intercept of this rate on the time axis. This method is commonly referred to as the Barrer method.

(2) Analytical Monitoring Status and Results

(U) The Taliani gas evolution rate measurements have been started at all four temperatures, and the necessary number of sealed ampules for long term off-gas analysis purposes have been prepared.

(U) Zero-time reflectance infrared spectra were obtained for all four formulations.

(U) All samples for determining zero-time mechanical properties have been cut, and the uniaxial tests have been completed. The test data are summarized in Table VI.

2. PHASE II, SHELF LIFE REPRODUCIBILITY

(C) Efforts under this phase have been limited to studying the shelf life of the palladium catalysts, and to determining the effects of ball milling upon aluminum hydride particle size distribution.

a. Catalyst Shelflife

(U) The hydrogenation rate of a COT solution in alcohol was determined using the standard palladium-on-carbon catalyst, a catalyst having higher palladium content, and a standard catalyst prepared without adding iron. The data are plotted in Figure 5.

(U) Additional tests were performed to determine if the catalyst activity had changed after 150 day storage in a dry state. The plots are also shown in Figure 5. The initial rate of reduction is slightly lower than that of the fresh catalyst. However, the total hydrogen uptake per gram COT at 96 hours was not appreciably changed.

TABLE VI
MECHANICAL PROPERTIES OF PHASE I PROPELLANTS
AT TIME ZERO

Mix No.	Formulation	Stress (psi)/Strain (%) (86 and 104°F*)
C	HI-53018 Control	67/15.2
D		74.1/16.7
E		75.7/17.4
W		90.6/20.4
X		99.5/21.5
F	0.5% Catalyst (30% Pd-Pt)	106.5/22.1
G		101.5/18.9
H		98.1/18.3
I		112.8/21.0
J		117.0/20.5
K	0.5% Spiegler Catalyst/1% COT	77.6/11.3
L		107.8/20.4
M		92.2/17.1
N		102.5/16.3
P		88.2/15.6
R	1% Spiegler Catalyst/1% COT	110.4/22.6
S		117.6/22.7
T		114.1/19.6
U		95.0/14.0
V		118.5/18.8

* No differences at the two temperatures.

b. Hydride Surface Stabilization

(C) The cubic crystalline structure of AlH_3 causes increased viscosity in propellant mixes, limiting solids loading and increasing the chance for attrition of AlH_3 particle. The Al_2O_3 coating on the sharp corners of the crystals may be especially vulnerable to attrition. The crystals themselves are rather fragile and may split into smaller cubic crystals along flaw lines, when stressed.

(C) An attempt was made to improve the shape and to split fragile AlH_3 crystals into more stable species by ball milling untreated Mg-doped AlH_3 in hexane. By sieving the AlH_3 after ball milling, the rounded crystals can be separated from the chips, shattered crystals and Al_2O_3 . In Table VII is sieve data from samples collected from various grinding times. The 44 micron size particles appear to be the strongest and, therefore, the least likely to be fractured or abraded. On the other hand a parallel study using microscopic examination indicated that rounding of the edges and corners, with the least amount of fracturing, occurred with a twenty-minute grind, using a different loading of grinding pellets.

3. PHASE III, MECHANISTIC ANALYSIS

(C) The shelflife of an aluminum hydride propellant can be predicted mathematically if the rate constants for the processes that govern internal gas generation, gas dissipation and any degradation in the system's ability to withstand internal pressure are known. Moreover, knowing the activation energies one can make temperature extrapolations, thus rely upon accelerated testing for quality control purposes.

(C) With the fluorine free AlH_3 propellant that was used under Contract FO4611-68-C-0023 the internal gas generation was caused largely by the thermal decomposition of the hydride, and the latter process could be defined by an empirical rate equation. As a result, the effect of lot-to-lot variations in hydride thermal stability, as determined by short term Taliani tests, upon 70°F propellant shelflife could be evaluated mathematically.

(C) In the AlH_3/NF_2 systems gas is being generated by several rate processes that are likely to involve different activation energies. Consequently much more detailed knowledge than is presently available of the various materials' reactions and interactions is needed if similar quality control capability is to be established for these systems.

(C) To provide this knowledge a subcontract was issued to the Midwest Research Institute for determining the rates of decomposition or reaction of individual ingredients and of various ingredient combinations using a slurry-type chemical reactor set-up. In a parallel effort LPC was to use a series of experimental propellants whose composition would closely duplicate the slurry systems to be used at MRI for comparison purposes.

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TABLE VII

PARTICLE SIZES, BALL MILL STUDY

Mesh Size	Particle Size, Micron Fraction Greater	Ball Mill Time (Minutes)			
		30	60	90	120
100	149	2.4%	0%	0%	0%
150	100	19.6%	1.1%	0.4%	0%
170	88	21.4%	4.4%	0.8%	0.8%
200	74	29.4%	18.2%	3.8%	1.7%
250	58	6.9%	17.6%	5.3%	4.6%
325	44	13.1%	35.7%	42.3%	73.4%
	<44	0.4%	23.1%	47.1%	19.6%*

* This fine fraction is probably in error due to hang-up of fines on walls of ball-mill container during last sample collection.

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a. Experimental Conditions

(C) The ingredients and ingredient combinations to be studied were Multron R-18, Multron R-18 prereacted with phenyl isocyanate for simulating the cure polymer, TVOPA and AlH_3 .

(C) For studying the decomposition of the neat materials, one gram quantities were weighed into manometer equipped glass containers that could be connected to a gas chromatograph for off-gas analysis purposes. For studying gas generation rates with mixtures, notably AlH_3 slurries in R-18, TVOPA and R-18/TVOPA, the ingredients were weighed into a round bottom flask to give a total material weight of ten grams. It was then mixed remotely for 30 minutes under nitrogen purge with a glass rod stirrer. One-gram quantities were then transferred to the manometric equipment for rate studies over the 30-50°C temperature range. Analytical techniques that were used in addition to gas chromatography off-gas analysis were infrared analysis and thin layer chromatography of the liquid phase.

(U) The materials were supplied by LPC to match the materials used under Phase I.

b. Summary of Results

(1) Reproducibility

(C) To determine the within-sample reproducibility and the effects of mix time, three R-18-C/TVOPA/ AlH_3 slurries were mixed for 20, 40 and 60 minutes and six one-gram quantities from each slurry were used to load four bulbs for off-gas analysis by gas chromatography, and two manometric reactors to determine overall gas evolution rates. The results of the analyses are summarized in Table VIII; they show that the volumes of H_2 , CO and CO_2 that are being generated during the initial ten days following mixing tend to decrease as mix time is increased; on the other hand, mix time has no significant effect upon the N_2O gas generation.

(C) The data are in contradiction with observed effects of mix time upon cured propellant gas evolution rates, and the disparity is presumed to reflect the more ready outgassing of the slurry during mixing and transfer. Nevertheless, both slurry and propellant data evidence a relatively high initial rate of gas evolution that may be attributed to AlH_3 surface phenomena (H_2 , CO, CO_2), but also to the presence of residual impurities (N_2O).

(U) Within sample reproducibility is good, and in this regard the data reflect the experience made with propellants.

(2) Origin of Gases

(U) The data that are summarized below were obtained primarily in tests performed over a 30-60 day time period at 50°C, and by conducting additional tests at different temperatures to determine temperature dependencies.

TABLE VIII
 REPRODUCIBILITY STUDY - R-18-C/TVOPA/AlH₃
 (Ten Days Gas Generation Data)

Mix Time ⁽¹⁾ (min)	No Data Point ⁽²⁾	Total Gas ⁽³⁾ (cc/g)	Species (cc/g) ⁽³⁾				
			H ₂	CO	CO ₂	N ₂ O ⁽⁴⁾	Others ⁽⁵⁾
60	6	0.164±0.004	0.065±0.004	0.009±0.001	0.076±0.004	0.014±0.003	None
40	6	0.236±0.020	0.086±0.003	0.013±0.001	0.113±0.002	0.013±0.001	None
20	6	0.247±0.019	0.092±0.004	0.019±0.003	0.123±0.017	0.013±0.0001	None

- (1) Mix compositions: 60 Minute - 6.57% R-18-C/55.40% TVOPA/38.05% AlH₃
 40 Minute - 6.54% R-18-C/55.48% TVOPA/37.98% AlH₃
 20 Minute - 6.49% R-18-C/55.35% TVOPA/38.14% AlH₃
- (2) One batch mix divided into six samples (four bulbs and two gas generation manometers).
- (3) Error is one standard deviation.
- (4) Evolved initially, not continuous.
- (5) IR analysis.

(a) Hydrogen Gas

(C) At temperatures below 40-50°C the hydrogen gas originates primarily from the thermal degradation of the hydride, AlH_3 /TVOPA and AlH_3 /TVOPA/R-18 systems giving off hydrogen gas at approximately equal rates. At temperatures above 40-50°C additional hydrogen gas appears to be produced by AlH_3 /TVOPA interaction. This is reflected in an increase in the apparent activation energy for the H_2 gas evolution as the temperature increases (Figure 6). The data imply that gas evolution rate measurements performed above 40-50°C will have little value for predicting material behavior; the data moreover indicate that the extrapolation of the results of surveillance tests performed at temperatures above 40-50°C will produce a pessimistic assessment of the system's 70°F shelflife capability.

(b) Carbon Dioxide

(C) At 50°C R-18, R-18-C and TVOPA alone give rise to little or no CO_2 generation during the initial 30-40 days, and the same holds true for R-18/TVOPA mixtures. After 30-40 days CO_2 production of R-18/TVOPA mixtures increases over TVOPA CO_2 production rates and AlH_3 appears to have a stabilizing effect. A more significant increase in CO_2 production occurs if phenyl isocyanate end-capped R-18 (R-18-C) is heated in mixture with TVOPA, and the CO_2 production rates increase further if DBTDA cure catalyst is added. In the latter system (R-18-C/TVOPA/DBTDA) distinct rate acceleration is observed after 20-30 days even in the presence of AlH_3 at 50°C (Figure 7). The data suggest that an initial surge in CO_2 production is prompted by the destruction of residual (unreacted) isocyanate which is in agreement with propellant off-gas analyses. Moreover, CO_2 may be generated by DBTDA catalyzed degradation of the cure linkage, a reaction that would produce free amine that will then catalyze TVOPA degradation; this process seems to be important, however, at temperatures of 50°C or above. Moreover, judging from the off-gas analyses one can conclude that in a propellant DBTDA catalyzed polymer breakdown could become quite significant at high storage temperatures.

(c) Carbon Monoxide

(C) Carbon monoxide originates primarily with the TVOPA, and the rate of CO production is increased by contacting TVOPA with AlH_3 . The CO production is almost completely suppressed by adding DBTDA to the system (R-18-C/TVOPA/ AlH_3 versus R-18-C/TVOPA/ AlH_3 /DBTDA). Consequently, since DBTDA does not catalyze CO_2 production below 50°C and since it suppresses CO production, its net effect upon propellant shelflife could be beneficial; this the more since CO is a non-condensable gas.

(d) Nitrous Oxides

(C) The formation of N_2O from TVOPA is suppressed by admixing TVOPA with R-18-C and there does not seem to be any additional effect upon AlH_3 addition.

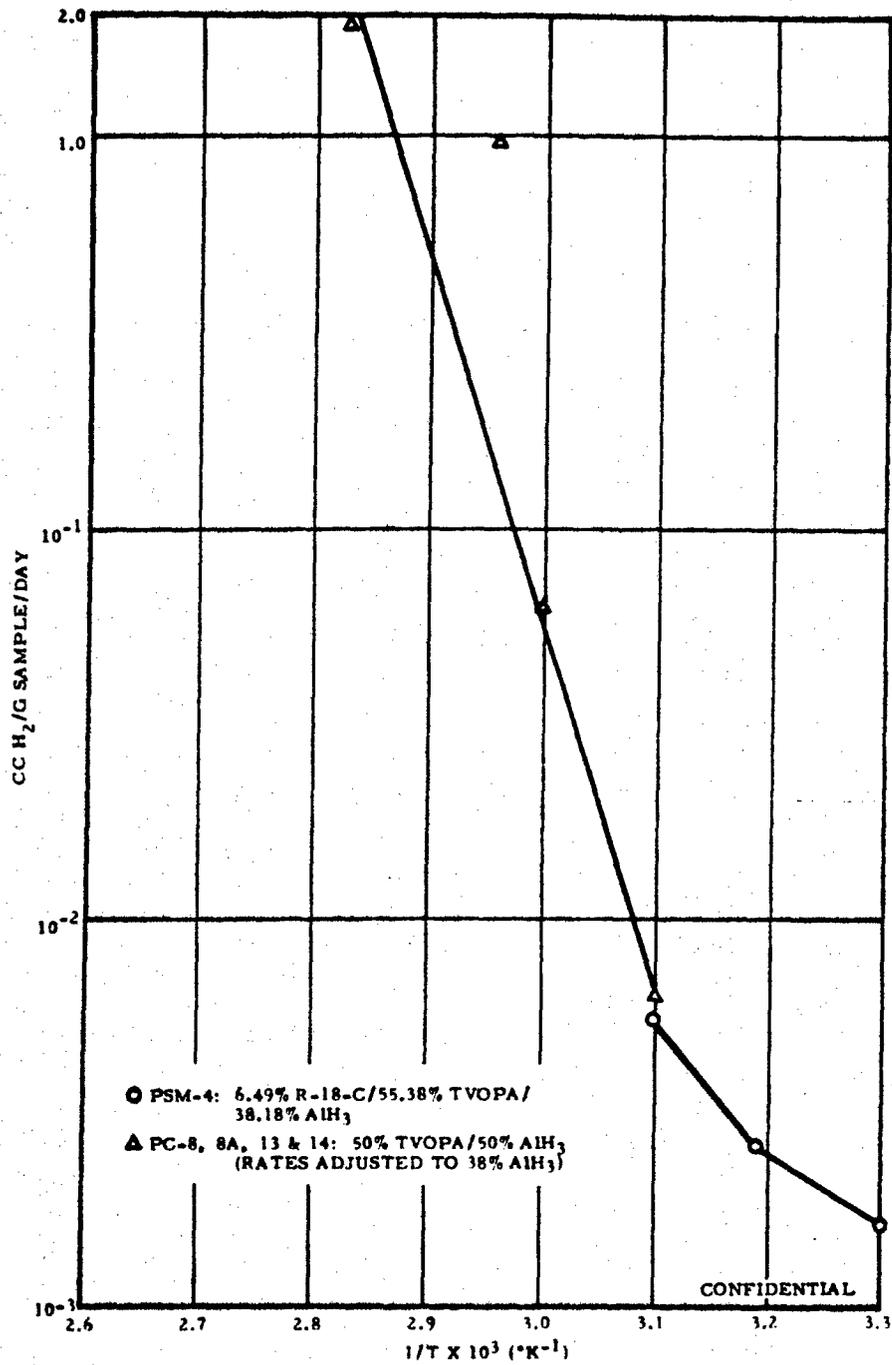


Figure 6 Rate of Evolution of Hydrogen

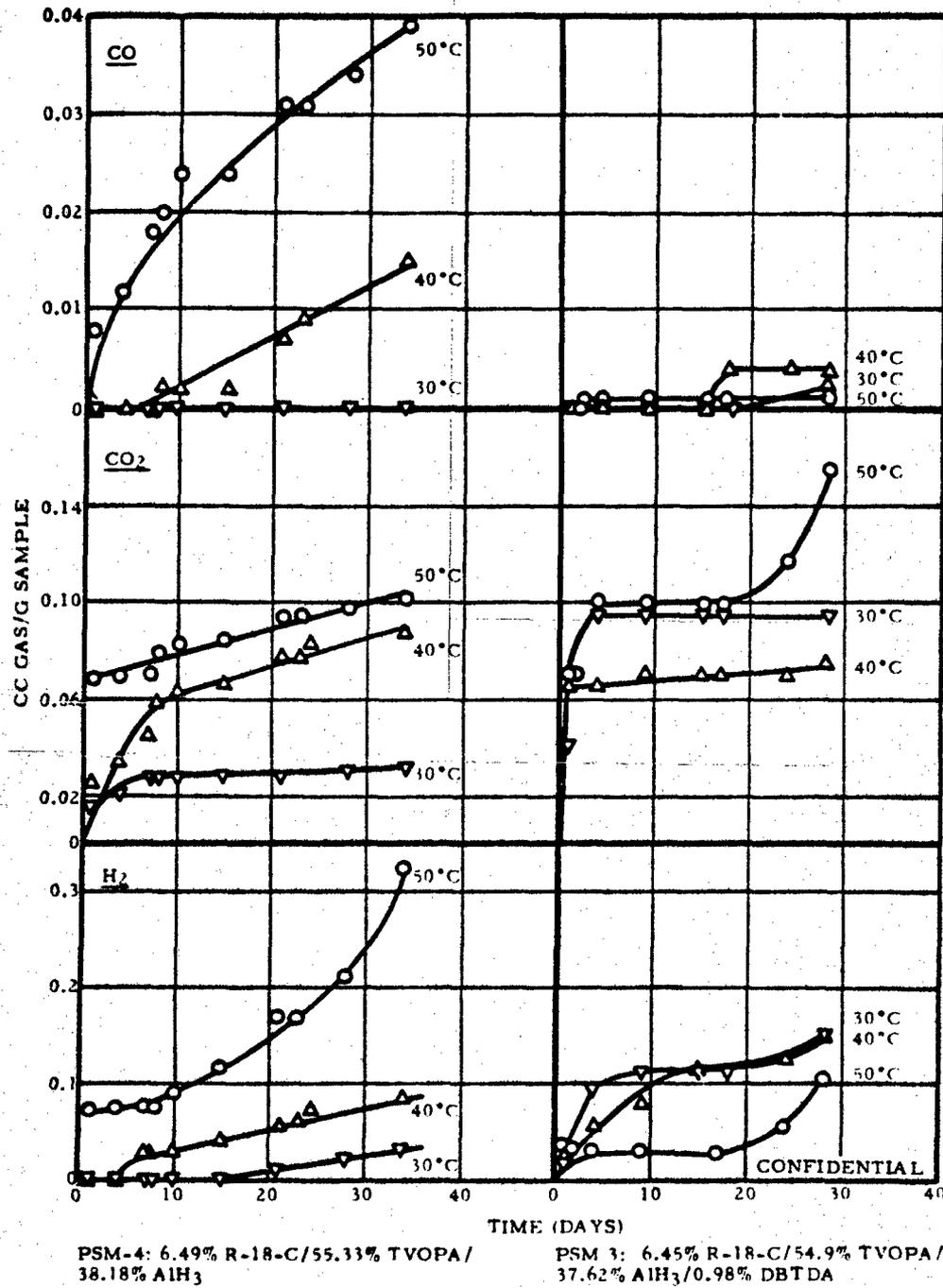


Figure 7 Effect of DBTDA on Off-Gas from R-18-C/TVOPA/AIH₃

(e) Other Species

(C) HCN, C₂H₄, HF (in the form of SiF₄) and species containing carbonyl, C-F and N-F bonds were identified as TVOPA decomposition products by infrared spectroscopy. The formation of these species is suppressed by adding R-18-C to the TVOPA.

(3) Conclusions

(U) The data show that the short term, accelerated testing (e.g., 50°C or higher) of individual ingredients, or specific ingredient mixtures, produces data that are difficult to extrapolate, and it is questionable whether further in-depth studies would render such accelerated testing suitable for quality control purposes. As a consequence, this work was discontinued.

(C) A positive outcome of the studies is the recognition that the composition of the off-gases can be influenced by adding catalytic amounts of materials like DBTDA. This is of interest, and it will be studied further, if via this means the formation of a non-condensable gas can be suppressed.

4. PHASE IV, FAILURE MODES

(U) The method to be used primarily for studying failure modes and storage time dependent changes in failure modes is the measurement of tear properties, and the analysis of parameters that govern tear behavior. The specific method to be used involves the use of biaxial strips to determine the stress levels, at varying strain levels, that result in zero crack propagation.

5. PHASE V, DATA INTEGRATION

(U) There has been no effort as yet under this phase.

6. PHASES VI AND VII, EXTENDED SURVEILLANCE

(U) Propellant samples (sealed samples of S-4 propellant) having been in storage at 70°F or 660-690 days have been analyzed for off-gas composition by a gas chromatographic method. Data reduction is in progress, and the results will be reported in the next technical report.

7. HAZARDS TESTING

(C) Preparatory to using Freon MF as a diluent in the mix process for determining the effects of varying mix processes upon propellant shelflife a hazards evaluation was performed. The data are summarized in Table IX, and they show that the addition of Freon MF can result in a transient high friction sensitivity.

(U) The Freon MF effect was determined as follows: the sample of propellant was placed on the pad of the Esso Friction Screw, and a drop of Freon MF was added. Friction sensitivity tests were then performed after waiting one-half, one and two minutes for the majority of the Freon to evaporate.

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TABLE IX
LABORATORY HAZARD SENSITIVITY TESTS

Ingredients	S-4	VKW	HI 53018		
12% R-18/88% TVOPA	32.18	30.94	28.56		
TDI	1.51	0.82	1.16		
HT	0.31	0.23	0.28		
Catalyst CWX 105	0.20	0.20	0.20		
AP, Type II	30.00	23.08	34.70		
AP, 8 micron	14.00	17.31	17.30		
HMX B	----	9.61	----		
AlH ₃	22.00	18.00	18.00		
Impact Test (kg-cm) (Bureau of Explosives)	4-5	3-4	4		
<u>Esso Friction Test (ft-lb)</u>			Without Freon MF	With Freon MF (1 drop)	
				1 min	1 min 2 min
No Grit	-	-	-	+<5	+75 -
SiC Grit	+<5	+<5	+18	(not tested)	
Pyrex Grit	+ 5	+<5	+5	(not tested)	

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(U) On the basis of these data no "Inert Diluent" mixes will be performed until the situation has been reviewed with Hercules.

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SECTION IV
FUTURE WORK

1. PHASE I, LONG TERM SURVEILLANCE

(U) The analytical monitoring of the samples in storage at 20, 30, 40 and 50°C will continue.

2. PHASE II, SHELF LIFE REPRODUCIBILITY

(C) The effect of grinding AlH_3 upon propellant gas evolution rates and shelflife will be determined.

3. PHASE III, MECHANISTIC ANALYSIS

(U) Emphasis will shift to the determination of rate phenomena that govern propellant mechanical properties degradation.

4. PHASE IV, FAILURE MODES

(U) Propellant tear behavior will be studied using biaxial strip samples to determine tear rates as a function of stress at varying strains.

5. PHASES VI AND VII, EXTENDED SURVEILLANCE

(U) Data reduction on the off-gases produced by S-4 and SS-4 propellants stored since January 1970 at 70°F will be completed.

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