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(Unclassified Title)

SURVEILLANCE STUDIES OF LMH-1

E. J. Wilson

ANNUAL TECHNICAL SUMMARY REPORT AFRPL-TR-68-35

(1 JANUARY TO 31 DECEMBER 1967)

JANUARY 1968

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

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(Prepared under Contract Nr. F04611-67-C-0067 by
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ANNUAL TECHNICAL SUMMARY REPORT (U)

(1 January 1967 - 31 December 1967)

SURVEILLANCE STUDIES OF LMH-1 (U)

January 1968

Dow Report Nr. FS-1A-68

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FOREWORD

This report was prepared by The Dow Chemical Company, Midland, Michigan, under USAF Contract Nr. FO4611-67-C-0067. The contract was initiated under Air Force BPSN Nr. 623148, AFSC Project Nr. 3148. The work was administered under the direction of the Rocket Propulsion Laboratory, Edwards Air Force Base, with Captain Robert Bargmeyer as Air Force Project Officer.

This is the first annual technical summary report, covering the work performed during the period of 1 January 1967 through 31 December 1967. The Dow Report Number is FS-1A-68.

Management direction at Dow is under Dr. D. A. Rausch, Assistant Laboratory Director, Chemicals Laboratory, and the work supervised by Dr. E. T. Niles, Group Leader. Mr. E. J. Wilson is the principal investigator with Mr. K. Roberson making major contributions.

This report has been reviewed and is approved.

W. H. Ebelke, Colonel, USAF
Chief, Propellant Division

(U) ABSTRACT

(C) Surveillance has continued on various samples of aluminum hydride. The types of aluminum hydride which are being studied include: unstabilized macrocrystalline, magnesium doped, and magnesium doped, DPA (diphenylacetylene) treated AlH_3 -1451. These samples are providing valuable decomposition rate data both neat at -15° , 25° and 40°C ., and in propellant at 25° , 40° and 60°C . Recently, neat surveillance studies have begun on high stability magnesium doped, DPA treated hydride and n-butylamine treated material which have 60°C . stabilities of greater than 30 days to reach 1% decomposition.

(C) Results of surveillance of neat aluminum hydride continue to indicate greater improvement in 25°C . stability than was observed with these samples at 60°C . One sample which reached 1% decomposition in 25.5 days at 60°C . has decomposed only 0.019% after 1 year at 25°C . as determined by a modified Taliani method. Another sample with a 60°C . stability of 12.3 days to 1% decomposition also showed excellent 25°C . stability, reaching only 0.05% decomposition after 1 year storage. Other samples with 60°C . stabilities of up to 40 days to 1% decomposition are now being evaluated at 25°C .

(C) Results of surveillance of aluminum hydride propellant are continuing to show decreasing gas generation rates with time. At 25°C ., double base propellant containing unstabilized macrocrystalline hydride decomposed approximately 0.28% during the first year, 0.09% during the second year, and 0.04% during the first half of the third year study. Decomposition figures assume all gas is generated by decomposition of the aluminum hydride and gas generation measurements are by a modified Taliani method. Stabilized aluminum hydride, magnesium doped and DPA treated samples, decomposed only approximately 0.10% during the first year and very little over the first half of the second year in double base propellant at 25°C .

(C) Small amounts of water in the environment which the aluminum hydride is subjected to have been found to prevent the typical "autocatalytic" shaped decomposition curve. Instead, an "initial gassing" period is observed followed by excellent stability. The shape of these gas generation curves markedly resembles the shape of the decomposition curves previously discussed for propellant at 25°C .

(C) Techniques are being developed to utilize small amounts of hydrolysis to stabilize aluminum hydride. A seventeen-day treatment of the hydride in wet (2.0% water) n-butylamine at 60°C . has produced a two- to fourfold improvement in the stability of various hydride samples.

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(C) The stability of neat aluminum hydride has been found to correlate inversely with the amount of lithium, other than lithium chloride, present as an impurity in the hydride. Excess lithium aluminum hydride is required in production of AlH_3 -1451 to enable desolvation of aluminum hydride etherate. The mechanism of the desolvation is not known, nor has the state in which the lithium resides in the AlH_3 -1451 crystal been determined. Studies are under way under Contract FO4611-68-C-0021 to define and eliminate the stability problems associated with the lithium impurity.

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SECTION I**(U) TECHNICAL RESULTS AND DISCUSSION**

(C) Major advancements have been made in processing aluminum hydride to produce material of improved stability. Figure 1 shows the magnitude of advancements made in the 60°C. stability of aluminum hydride during the past few years. Whereas not long ago average pilot plant aluminum hydride reached 1% decomposition in 5 days at 60°C., today, with the incorporation of magnesium doping and DPA surface treatment along with process improvement, many pilot plant aluminum hydride samples exceed 20 days to reach 1% decomposition at 60°C. Development of a post-treatment procedure with wet n-butylamine has resulted in at least doubling of the 60°C. stability of samples received from the pilot plant.

(C) Although these stability improvements have been made, it is believed that additional improvements are necessary before aluminum hydride can be used in large storable solid rocket propellant motors due to the internal pressures caused by small amounts of decomposition of the hydride. It is the purpose of this program to study and characterize the decomposition of various samples of aluminum hydride over a range of temperatures to enable a better assessment of the decomposition rates to be expected during long term storage of the hydride, neat and in propellant. At a future date these data will be very valuable in predicting long term stability and shelf life.

A. SURVEILLANCE OF NEAT ALUMINUM HYDRIDE-1451 (U)

(C) The decomposition and stability determinations of various samples of aluminum hydride have been continued and expanded to include: unstabilized macrocrystalline, magnesium-doped, magnesium-doped DPA-treated, and more recently n-butylamine treated material. Results indicate that the newer stabilized material not only maintains the factor of stability improvement observed at 60°C., but actually increases the 25°C. stability by an appreciably greater amount. The results presented in this section demonstrate this important phenomenon.

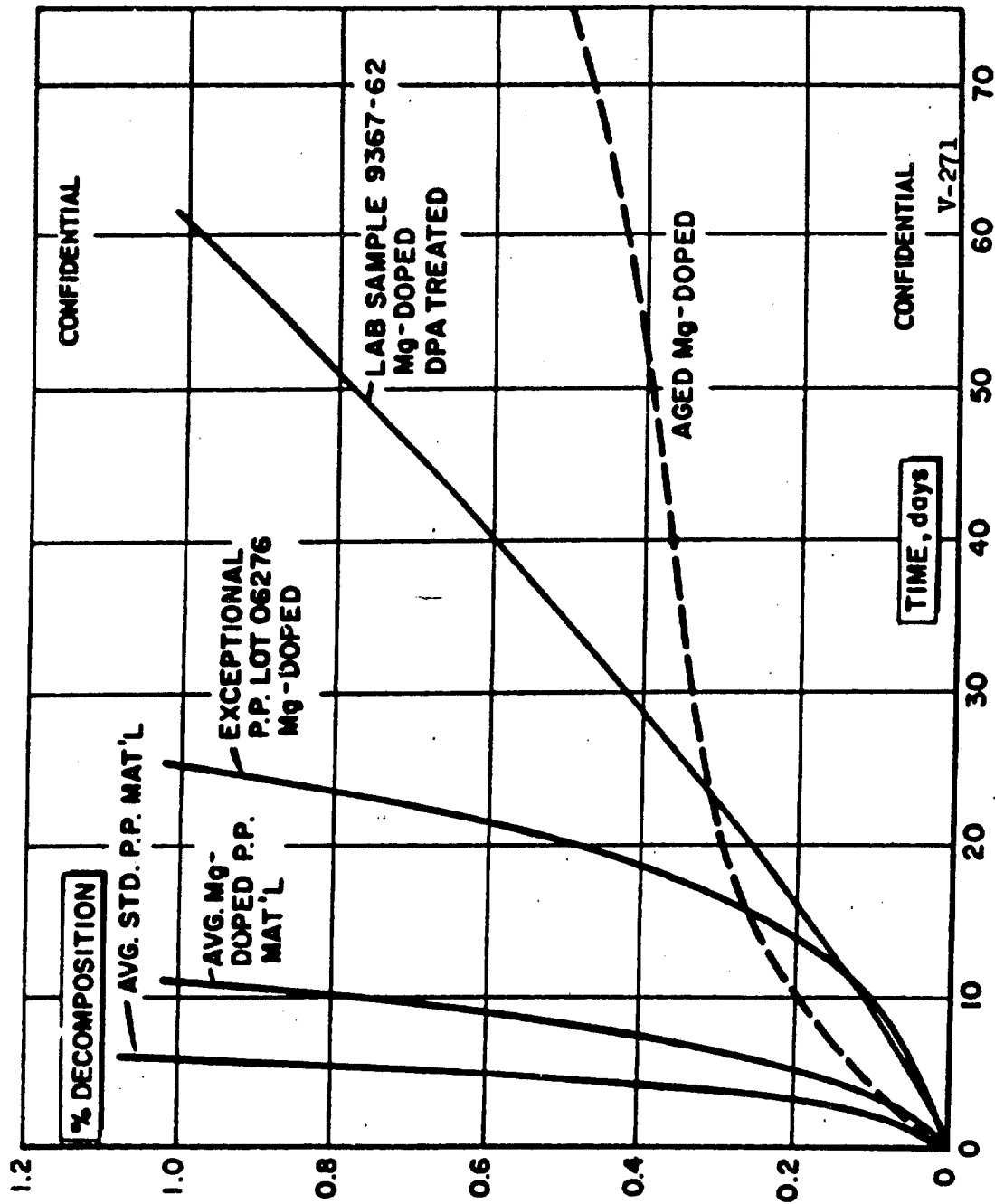
1. Microcrystalline Hydride (U)

(C) Many samples of the original microcrystalline (1-5 μ) AlH_3 -1451 have been stored at ambient temperature and -15°C. and have been periodically checked for decomposition by hydrogen analysis under previous programs (1). Results indicated significant decomposition rates even at ambient temperature, with many samples exceeding 50% decomposition after one-year storage. No further work has been conducted with these materials.

¹The Dow Chemical Company, Annual Technical Report, AFRPL-TR-67-37, January, 1967, Contract Nr. AF 04(611)-11400, Dow Report Nr. FS-4Q-66.

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(U) Fig. 1 - Improvements in the 50°C. Stability of Aluminum Hydride-1491

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2. Unstabilized Macrocrystalline Hydride (U)

(U) A number of macrocrystalline AlH_3 -1451 samples has been stored in taped glass vials under an inert atmosphere at ambient temperature and -15°C . for several years. Samples were also stored at 40°C . for 222 days. The samples were periodically sampled for hydrogen analysis and 60°C . stability testing.

(C) The results of the periodic hydrogen analysis of the samples stored at -15°C . and ambient temperature are listed in Table I. These results indicate very little decomposition in many of the samples. It is evident that the low levels of decomposition these samples have reached are within the experimental error of the hydrogen analysis technique used.

(C) The results of periodic hydrogen analysis of the samples stored at 40°C . are listed in Table II. The decomposition levels observed in these samples are significant and the average decomposition curve for the six samples studied is shown in Figure 2.

(C) The periodic 60°C . stability test results for the fourteen unstabilized samples being studied at -15°C . and ambient temperature are listed in Tables III and IV. No additional testing of the -15°C . samples has been conducted on this program. Previous results showed very little change in the 60°C . stability of these samples as a result of storage at -15°C . The samples stored at 25°C . have been re-examined and found to have decreased considerably in 60°C . stability after approximately 36 months storage. Of the fourteen samples listed in Table IV, the first five have been somewhat erratic in stability change whereas the last nine have decreased in stability gradually. The average of the stability of these two groups of samples is shown in Figure 3 as a function of storage time. The increase in stability of the first five samples during the first year storage may have been caused by contamination of the samples with water. The decrease in stability of the second group of samples is more representative of the expected behavior of samples stored under an inert atmosphere at ambient temperature.

3. Magnesium-Doped Hydride (C)

(C) Samples of eight Texas Pilot Plant lots of magnesium-doped AlH_3 -1451 are being studied at -15° , 25° and 40°C . storage. Decomposition is being measured by a modified Taliani procedure at 25° and 40°C ., and also by periodic hydrogen analysis of samples stored at -15 , 25 and 40°C . Periodic 60°C . stability testing is being conducted on all stored samples to determine stability changes. Analyses and the original 60°C . stability of the samples being studied are given in Table V.

Table I

(C) Summary of Hydrogen Analyses* for MacrocrySTALLINE Aluminum Hydride-1451 Sample Stored at Ambient Temperature and -15°C.

Sample Number	Time, Months	Wt. % Hydrogen Storage Temp.		Sample Number	Time, Months	Wt. % Hydrogen Storage Temp.	
		Ambient	-15°C.			Ambient	-15°C.
9655-85	0	10.12	10.12	02034A	0	9.75	9.75
	20	9.70	9.74		3	9.59	9.58
	25	9.78	9.76		10	9.68	9.78
	31	9.84	9.92		15	9.67	9.87
	37	9.60	9.92		21	9.84	9.81
	45	9.78			27	9.78	9.83
		Average 9.97				9.72	
02044	0	9.56	9.86	02044A	0	9.83	9.83
	3	9.38	9.60		3	9.33	9.85
	10	9.82			10	9.87	9.91
	15	9.82	9.90		15	9.95	10.01
	21	9.86	9.85		21	9.92	10.00
	27	9.83	9.88		27	9.77	9.82
		Average 9.63				9.84	
02134A	0	9.54	9.74	02244	0	10.00	10.00
	3	9.74	9.76		3	10.00	10.00
	10	9.53	9.87		10	9.91	10.00
	15	9.53	9.77		15	10.01	10.06
	21	9.84	9.91		20	9.97	10.05
	27	9.93	9.90		24	9.94	
		Average 9.56				Average 10.00	
04194	0	10.27	10.27	06094	0	9.88	9.88
	8	9.94	9.96		6	9.85	9.92
	13	9.99	10.03		11	9.97	9.99
	19	9.93	9.97		17	10.02	10.03
	25	10.02	10.10		23	9.95	10.11
	33	10.03			31	9.94	
		Average 10.00				Average 9.94	
06104A	0	9.83	9.77	06104AT	0	9.90	9.90
	6	10.05	10.02		6	10.01	9.99
	11	9.94	10.03		11	10.03	10.04
	17	9.90	9.96		17	9.97	10.07
	23	10.02	10.10		23	10.06	9.94
	31	10.08			31	10.05	
		Average 10.00				Average 9.99	
07084	0	10.04	10.04	07084AT	0	9.99	9.85
	5	10.05	10.00		5	10.07	10.05
	10	9.97	9.99		10	9.95	10.02
	16	9.94	9.99		16	9.98	9.96
	22	10.03	10.02		22	9.94	10.05
	30	10.02			30	9.95	
		Average 10.00				Average 9.99	

*0.1% Hydrogen.

Table II

(U) Stability of Aluminum Hydride-1451 at 40°C.

<u>Sample Number^a</u>	<u>Decomposition^b, %</u> <u>Storage Time, days</u>			
	<u>80</u>	<u>97</u>	<u>202</u>	<u>222</u>
04185A	1.3	1.3	23.0	42.4
04195	1.8	2.4	16.9	35.0
04265B	1.1	2.2	16.7	33.3
06255	0.2	1.7	14.3	35.0
06275	1.2	2.5	20.4	38.0
06285	<u>1.0</u>	<u>2.2</u>	<u>15.9</u>	<u>36.2</u>
Average % Decomp.	1.1	2.1	17.9	36.6

^aSamples stored 3 months in cold storage prior to evaluation.

^bDetermined by carbon and hydrogen analysis; hydrogen $\pm 1\%$ of total.

Table III

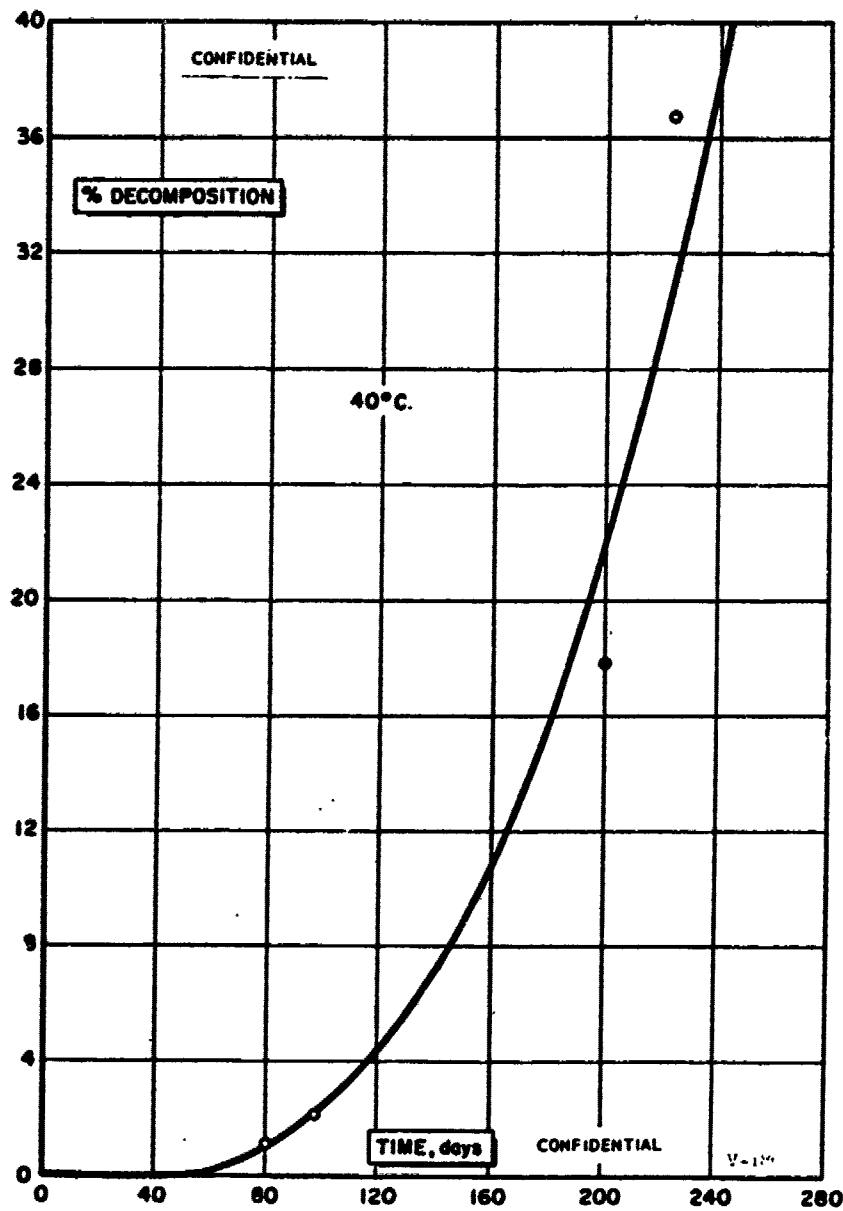
(U) Effect of Storing Time at -15°C. on Thermal
Stability of Unstabilized Aluminum Hydride

<u>Sample No.</u>	<u>Days to Reach 1% Decomposition at 60°C.^a</u> <u>Storage Time at -15°C., months</u>					
	<u>Original</u>	<u>3</u>	<u>8</u>	<u>11</u>	<u>16</u>	<u>20</u>
3655-95-2	4.2	--	--	--	--	--
02034A	5.3	4.7	4.4	4.8	5.3	5.4
02044	3.9	3.7	6.7	7.0	8.9	5.6
02044A	4.5	4.4	5.1	6.8	8.3	9.2
02134A	5.3	4.6	10.5	11.6	13.9	12.6
04194	5.5	7.6	9.0	8.9	--	--
03294	8.0	7.7	7.9	7.6	--	--
06014A	6.2	5.0	5.7	7.1	--	--
06024A	6.3	5.7	5.8	6.8	--	--
06054	8.0	6.2	6.8	7.1	--	--
06104A	10.0	8.7	10.2	9.4	--	--
06104AT	10.2	8.8	8.7	9.6	--	--
07084	7.2	6.7	7.0	7.4	--	--
07084T	6.3	7.1	6.6	7.0	--	--

^aTaliani test

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(U) Fig. 2 - Average Decomposition Curve
for Macrocrystalline Aluminum Hydride-1451 at 40°C.

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Table IV

(U) Effect of Storing Time at Ambient Temperature
on Thermal Stability of Unstabilized Aluminum Hydride

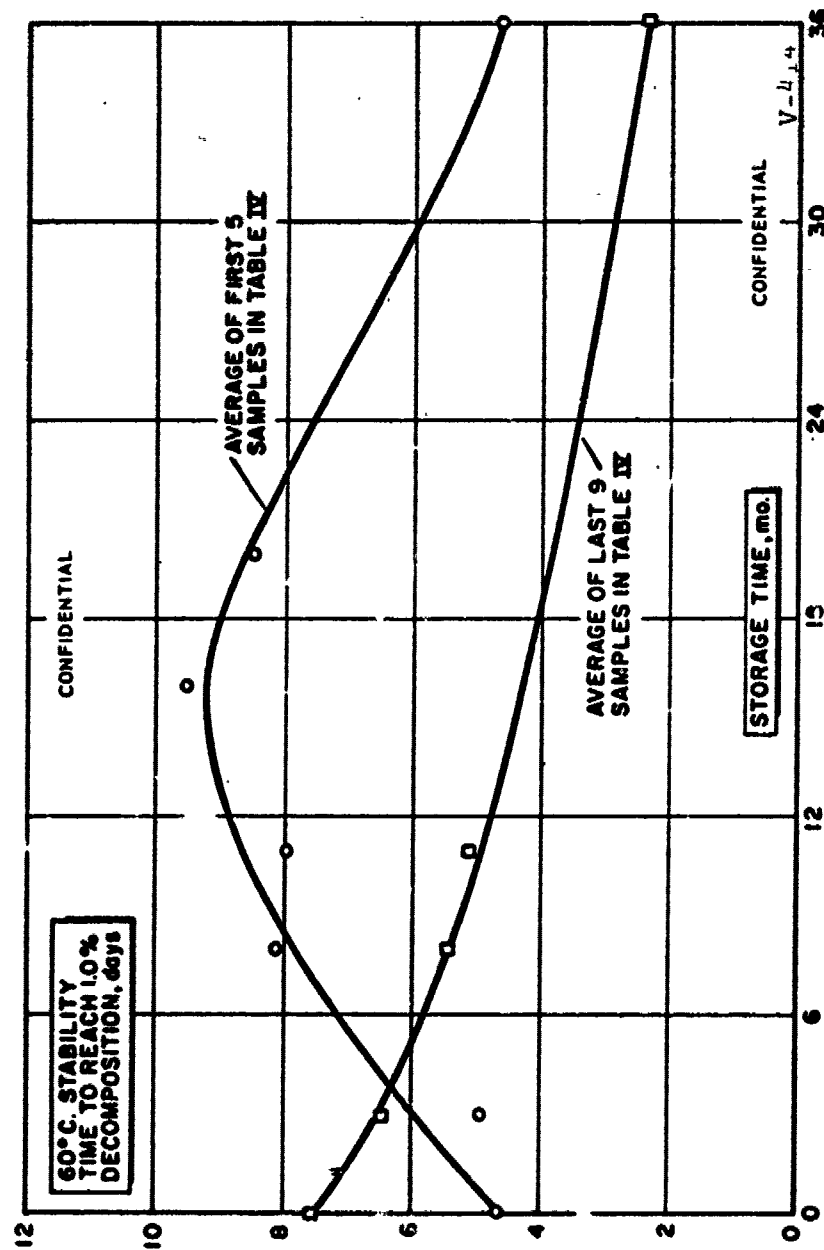
Sample No.	Days to Reach 1% Decomposition at 60°C. ^a Storage Time at Ambient Temperature, months						
	Original	3	8	11	16	20	~36
3655-95-2	4.2	5.8	3.7	4.5	9.6	8.7	--
02034A	5.3	3.3	7.2	8.0	9.2	8.6	5.1
02044	3.9	3.9	6.6	6.3	6.8	8.3	4.0
02044A	4.5	2.8	11.7	12.5	13.0	9.6	5.5
02134A	5.3	8.7	11.2	8.5	9.0	7.0	3.4
04194	5.5	7.2	6.3	6.0	--	--	2.0
03294	8.0	6.9	5.4	4.6	--	--	0.9
06014A	6.2	5.0	4.2	4.2	--	--	2.7
06204A	6.3	4.7	4.0	4.2	--	--	2.8
06054	8.0	5.9	4.5	4.1	--	--	1.2
06104A	10.0	8.3	7.4	7.3	--	--	3.2
06104AT	10.2	8.5	7.1	6.2	--	--	2.1
07084	7.2	6.2	6.3	5.8	--	--	3.1
07084T	6.3	5.4	4.1	3.6	--	--	1.1

^aTaliani test

Table V

(C) Magnesium-Doped Aluminum Hydride-1451
Surveillance Samples

Lot No.	60°C. Stability days to 1% Decomp.	Elemental Analysis, %					
		C	H	Mg	Al	Li	Cl
01206	13.2	0.22	9.98	0.63	88.8	0.12	<0.1
01216	14.4	0.14	10.03	0.67	88.9	0.12	<0.1
01286	12.3	0.18	10.05	0.48	89.1	0.12	<0.1
01316	12.0	0.16	10.02	0.81	88.7	0.14	<0.1
06206	11.7	0.08	10.01	1.26	88.6	0.20	0.05
06276	25.5	0.07	9.96	1.35	88.5	0.12	0.01
06286	18.8	0.17	10.00	0.90	87.8	0.16	0.01
06306	16.8	0.17	10.09	0.86	88.1	0.17	0.05



(U) Fig. 3 - Effect of Storage Time at Ambient Temperature on Thermal Stability of Unstabilized Aluminum Hydride-1451

a. -15°C. Surveillance (U)

(C) The results from storage of these samples at -15°C. are presented in Table VI. Of the eight samples the first four, which contain <0.85% magnesium, have been stored for 20 months, and the last four, which contain >0.85% magnesium, have been stored for 14 months. All eight samples are scheduled for examination during April, 1968.

(C) The data presented in Table VI show no significant change in the hydrogen content of the samples, another indication that decomposition does not occur at a noticeable rate during storage at -15°C. The periodic 60°C. stability testing of these samples shows a slight increase in stability as a function of storage time. Periodic oxygen analysis, by neutron activation, are being included in the surveillance program to determine if these increases in 60°C. stability are related to water contamination. Not enough data are available at this time to ascertain whether moisture contamination is occurring.

b. Ambient Temperature (~25°C.) Surveillance (U)

(C) The results from the ambient temperature (~25°C.) storage of the eight magnesium-doped AlH_3 -1451 samples during 20 and 14 months storage are listed in Table VII. At this temperature the decomposition of the samples is being continuously measured by a modified Taliani procedure as well as by periodic hydrogen analysis, and 60°C. stability determinations are being conducted.

(C) The data shown in Table VII are of considerable importance since this is the most detailed long term study of the decomposition of AlH_3 -1451 which has been conducted. The most important results are the Taliani measurements of decomposition at 25°C. These results are plotted for representative samples in Figure 4. The differences between samples in 25°C. stability is much larger than the differences in the 60°C. stability of these samples. This will be discussed further in Section I.D.1. The periodic hydrogen analysis data are scattered and are not valid for determining decomposition in this low range. The periodic 60°C. stability results show a gradual decrease in stability as a function of storage time. Not enough oxygen analysis data are available at this time to determine if contamination with water is occurring.

c. 40°C. Surveillance (U)

(C) The results from the 40°C. surveillance of the eight magnesium-doped AlH_3 -1451 samples are listed in Table VIII. The decomposition rate at 40°C. is high enough to be easily followed by hydrogen analysis although considerable scatter of the data is evident. For this reason portions of the samples were transferred to a modified Taliani apparatus midway through

Table VI
(C) Effect of Storage of Magnesium-Direct Aluminum Hydride-1451 at -15°C.

Gravimetric, wt. %		60°C. Stability of Various Sized Samples																	
		Time to Reach 0.5% Decomposition, days					Time to Reach 1.0% Decomposition, days												
Al	Si	Al	Si	Al+Si	Al	Si	Al+Si	Al	Si	Al+Si	Al	Si	Al+Si	Al	Si	Al+Si			
9.36	--	--	--	9.36	5.5	6.2	5.3	6.6	6.9	7.2	11.2	10.3	10.6	12.2	12.2	14.4	13.6	14.4	15.5
10.05	--	--	--	10.05	5.5	6.5	7	6.5	7.9	8.7	12.2	11.5	13.3	14.1	14.4	15.5	15.2	17.1	15.5
10.05	--	--	--	10.05	5.2	6.3	5.3	6.0	6.6	7.2	11.2	10.1	11.5	11.3	12.3	13.5	13.1	14.9	14.2
10.02	--	--	--	10.02	5.0	6.5	5.3	6.6	6.4	9.2	11.3	10.4	11.8	11.2	12.0	13.9	13.4	14.9	14.2
10.01	--	--	--	10.01	5.2	5.3	5.2	--	--	9.5	10.0	10.7	--	--	11.7	12.2	13.2	--	--
9.36	--	--	--	9.36	5.6	9	9	--	--	14.5	17.2	20.3	--	--	25.5	21.6	25.1	--	--
10.00	--	--	--	10.00	5.3	7.3	7.9	--	--	15.3	14.6	17	--	--	19.8	17.8	21.1	--	--
10.05	--	--	--	10.05	6.3	6	6.8	--	--	13.6	12	14.4	--	--	16.8	14.7	17.0	--	--

Table VII

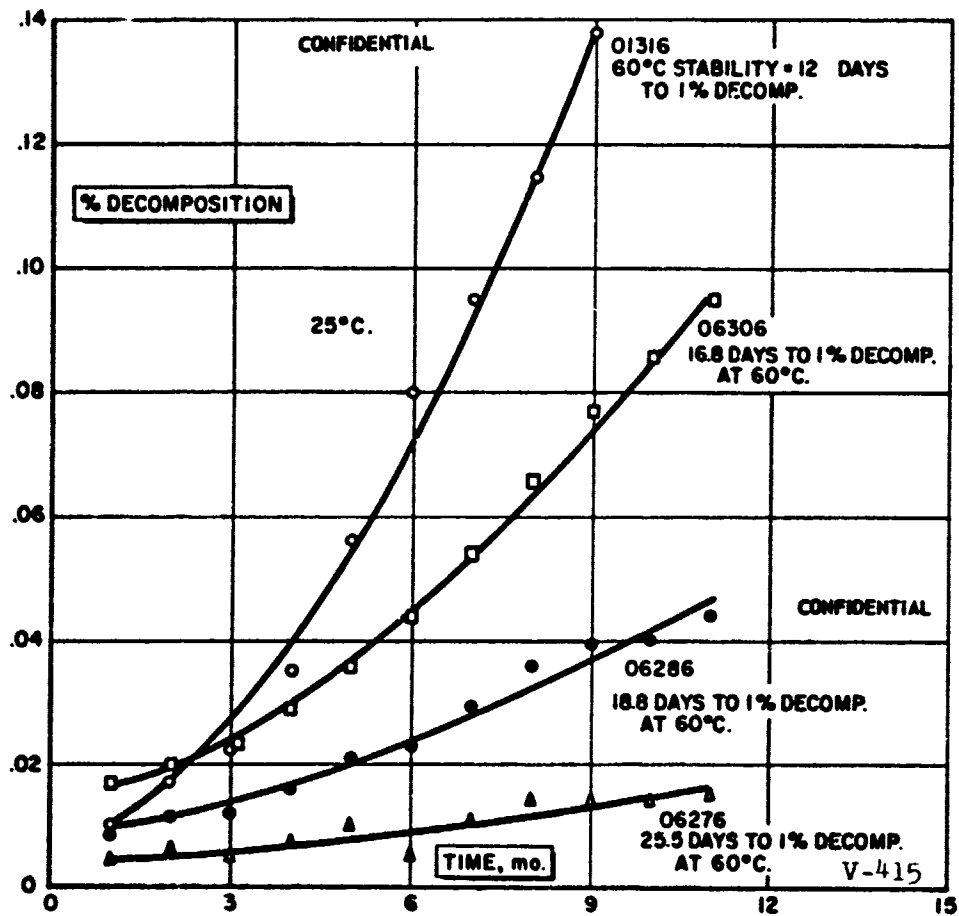
(c) Effect of Storage of Magnesium-Doped Aluminum Powder-1471 at Ambient Temperature

No. Doped Sample Number	Storage Time, Months																		
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
0120c																			
H Analysis, %	9.96	--	--	--	--	--	--	--	--	--	--	--	9.67	--	--	--	--	9.98	--
% Recomp. (H Anal.)	--	--	--	--	--	--	--	--	--	--	--	--	1.1	--	--	--	--	811	--
Tallent % Recomp.	--	0.012	0.015	0.010	0.005	0.066	0.028	0.119	0.135	0.173	0.204	0.235	0.270	0.302	0.359	0.413	0.458	0.575	0.623
to C. Tal. Stabil.	--	--	--	--	--	--	--	--	--	--	--	--	3.5	--	--	--	--	--	0.575
Days to 0.1% Recomp.	5.5	--	--	5	--	--	--	--	--	3.6	--	--	3.5	--	--	--	--	--	--
Days to 0.5% Recomp.	10.7	--	--	20.5	--	--	--	--	--	7.1	--	--	3.1	--	--	--	--	--	--
Days to 1.0% Recomp.	12.2	--	--	33.1	--	--	--	--	--	13	--	--	13	--	--	--	--	9.0	--
Oxygen Analysis, %	--	--	--	--	--	--	--	--	--	--	--	--	0.19	--	--	--	--	--	--
0.21c																			
H Analysis, %	10.03	--	--	--	--	--	--	--	--	--	--	--	10.03	--	--	--	--	10.04	--
% Recomp. (H Anal.)	--	--	--	--	--	--	--	--	--	--	--	--	0	--	--	--	--	811	--
Tallent % Recomp.	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
to C. Tal. Stabil.	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Days to 0.1% Recomp.	9.7	--	--	5	--	--	--	--	--	3.6	--	--	3.2	--	--	--	--	--	--
Days to 0.5% Recomp.	10.7	--	--	11.9	--	--	--	--	--	7.2	--	--	7.2	--	--	--	--	--	--
Days to 1.0% Recomp.	14.4	--	--	15.5	--	--	--	--	--	12.7	--	--	13.3	--	--	--	--	10	--
Oxygen Analysis, %	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
0.22c																			
H Analysis, %	10.05	--	--	--	--	--	--	--	--	--	--	--	9.98	--	--	--	--	9.83	--
% Recomp. (H Anal.)	--	--	--	--	--	--	--	--	--	--	--	--	0.7	--	--	--	--	1.2	--
Tallent % Recomp.	--	0.009	0.014	0.028	0.037	0.095	0.082	0.125	0.116	0.200	0.256	0.309	0.373	0.433	0.516	0.626	0.711	0.805	0.977
to C. Tal. Stabil.	--	--	--	--	--	--	--	--	--	--	--	--	2.8	--	--	--	--	--	0.977
Days to 0.1% Recomp.	9.2	--	--	11.3	--	--	--	--	--	3.5	--	--	2.8	--	--	--	--	--	--
Days to 0.5% Recomp.	9.3	--	--	11.3	--	--	--	--	--	6.3	--	--	10.8	--	--	--	--	--	--
Days to 1.0% Recomp.	12.3	--	--	13.7	--	--	--	--	--	11.3	--	--	10.8	--	--	--	--	--	--
Oxygen Analysis, %	--	--	--	--	--	--	--	--	--	--	--	--	0.15	--	--	--	--	--	--
0121c																			
H Analysis, %	10.02	--	--	--	--	--	--	--	--	--	--	--	9.7	--	--	--	--	9.94	--
% Recomp. (H Anal.)	--	--	--	--	--	--	--	--	--	--	--	--	0.5	--	--	--	--	0.6	--
Tallent % Recomp.	--	0.010	0.017	0.022	0.035	0.096	0.080	0.095	0.115	0.136	0.167	0.207	0.237	0.281	0.344	0.475	0.542	0.671	0.805
to C. Tal. Stabil.	--	--	--	--	--	--	--	--	--	--	--	--	2.3	--	--	--	--	--	--
Days to 0.1% Recomp.	9.2	--	--	9.5	--	--	--	--	--	2.9	--	--	2.3	--	--	--	--	--	--
Days to 0.5% Recomp.	9.1	--	--	9.5	--	--	--	--	--	8.1	--	--	7.1	--	--	--	--	--	--
Days to 1.0% Recomp.	12	--	--	13	--	--	--	--	--	11.6	--	--	9.15	--	--	--	--	--	--
Oxygen Analysis, %	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--

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(C) FIG. 4 - Decomposition of Magnesium-Doped Aluminum Hydride 1451 at 25°C.

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Table VIII
(C) Effect of Storage of Magnesium-Doped Aluminum Hydride-1451 at 40°C.

Sample	Storage time, months															
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
01206																
H Analysis, wt. %	9.25	--	--	10.15	9.34	9.70	9.72	--	--	--	9.36	Terminated				
Decomp. Based on Anal.	--	--	--	0.11	0.4	2.4	2.0*	--	--	--	10.2					
Tallent Decomp.	--	--	--	--	--	--	2.0	--	4.45	6.71	7.98					
60°C. Stabil., days to 15	15	15	12	6	5	--	--	--	--	--	--					
01210																
H Analysis, wt. %	10.05	--	--	10.05	9.89	9.76	9.69	--	--	--	8.5	Terminated				
Decomp. Based on Anal.	--	--	--	0.11	1.4	2.1	3.4*	--	--	--	10.5					
Tallent Decomp.	--	--	--	--	--	--	5.4	--	4.51	7.03	8.85					
60°C. Stabil., days to 15	44	15	11	7	7	--	--	--	--	--	--					
01200																
H Analysis, wt. %	10.06	--	--	10.02	9.45	9.57	9.73	--	--	--	9.18	Terminated				
Decomp. Based on Anal.	--	--	--	0.4	1.5	1.9	3.5*	--	--	--	8.5					
Tallent Decomp.	--	--	--	--	--	--	5.5	4	5.05	7.64	9.35					
60°C. Stabil., days to 15	12	11	10	6	5	--	--	--	--	--	--					
01316																
H Analysis, wt. %	10.02	--	--	10.00	9.5	9.50	9.40	--	--	--	7.14	Terminated				
Decomp. Based on Anal.	--	--	--	0.2	0.7	2.2	2.2*	--	--	--	22.5					
Tallent Decomp.	--	--	--	--	--	--	2.2	--	4.15	7.11	6.85					
60°C. Stabil., days to 15	11	11	9	6	5	--	--	--	--	--	--					
01206																
H Analysis, wt. %	10.01	--	--	10.0	9.70	9.5	9.53	--	--	--	--	9.51	--	--	--	--
Decomp. Based on Anal.	--	--	--	0.5	0.5	2.3	4.3	--	--	--	--	4.2	--	--	--	--
Tallent Decomp.	--	--	--	--	--	--	1.5	--	1.6	2.0	3.65	4.06	4.6	5.74	6.36	--
60°C. Stabil., days to 15	11.7	9.5	5.4	--	5.7	--	--	--	--	--	--	--	--	--	--	--
01276																
H Analysis, wt. %	9.45	--	--	10.00	9.40	9.48	9.56	--	--	--	--	9.50	--	--	--	--
Decomp. Based on Anal.	--	--	--	0.11	0.6	0.2	1.0*	--	--	--	--	4.0	--	--	--	--
Tallent Decomp.	--	--	--	--	--	--	1.0	--	1.02	1.09	1.15	1.20	--	1.53	1.85	--
60°C. Stabil., days to 15	22.5	20.2	19.8	--	--	--	--	--	--	--	--	--	--	--	--	--
01200																
H Analysis, wt. %	10.00	--	--	10.0	9.77	9.4	9.40	--	--	--	--	9.0	--	--	--	--
Decomp. Based on Anal.	--	--	--	0.11	1.2	0.4	1.0*	--	--	--	--	7.0	--	--	--	--
Tallent Decomp.	--	--	--	--	--	--	1.0	--	1.12	1.17	1.22	2.01	2.51	2.74	2.95	--
60°C. Stabil., days to 15	18.9	16.7	15.8	--	15.9	--	--	--	--	--	--	--	--	--	--	--
01306																
H Analysis, wt. %	10.03	--	--	10.05	9.68	9.56	9.27	--	--	--	--	8.95	--	--	--	--
Decomp. Based on Anal.	--	--	--	0.5	2.1	1.5	8.2*	--	--	--	--	11.4	--	--	--	--
Tallent Decomp.	--	--	--	--	--	--	2.2	--	2.4	5.19	4.45	4.44	5.30	6.87	7.21	--
60°C. Stabil., days to 15	16.8	12.6	11.5	--	10.8	--	--	--	--	--	--	--	--	--	--	--

*Tallent study started at this point. In final de-composition based on H analysis.

the program. Since that time only one hydrogen analysis has been run on each sample. However, at the point of analysis good agreement between hydrogen content and Taliani data is observed only for three samples, numbers 01216, 01286, and 06206. The remaining five show poor agreement and the hydrogen analysis values seem to be out of line. Additional analytical determinations of hydrogen content are planned to determine the nature of the discrepancy in the data.

(C) Figure 5 shows the average decomposition curve for the two groups of magnesium-doped samples (<0.85% magnesium and >0.85% magnesium). The data plotted is taken from the hydrogen analysis results for the first six months and subsequently from the Taliani measurements.

4. Magnesium-Doped, DPA Treated Hydride (U)

(C) Eight samples of magnesium-doped, DPA treated AlH_3 -1451 have been received from the Texas Pilot Plant. The first four samples have been undergoing surveillance for about four months; the second group has just recently been added to the program and no data are available at this time. A list of the eight samples, showing the 60°C. stability (time to reach 1% decomposition at 60°C.) and the elemental analysis of the samples is given in Table IX.

Table IX

(C) Magnesium-Doped, DPA Treated Aluminum
Hydride-1451 Surveillance Samples

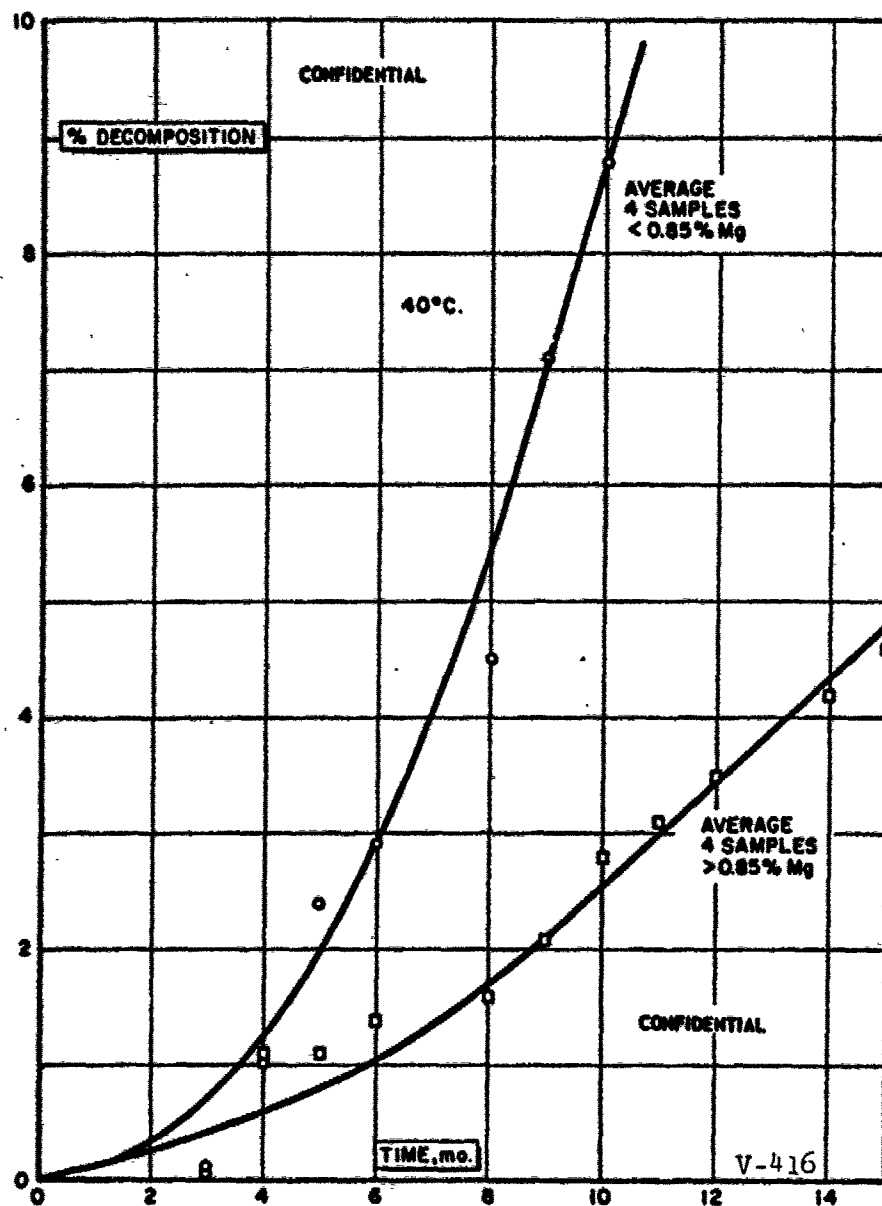
Lot. No.	60°C. Stability days to 1%	Elemental Analysis, %						
		C	H	Mg	Al	Li	Cl	O
10056	20.1	0.58	9.90	1.19	87.4	0.20	0.09	0.19
11286	18.9	1.06	9.95	1.26	87.5	0.17	0.03	0.23
11306	14.3	1.25	10.00	1.17	87.9	0.17	0.03	0.27
12066A	22.7	1.10	10.08	1.18	87.4	0.15	0.03	0.35
08117A	~40	>1.0	9.99	0.93	88.1	0.10	0.05	--
08187A	~30	>1.0	10.04	1.58	87.8	0.12	0.06	--
10107A	~35	>1.0	9.95	0.86	87.9	0.13	0.24	--
10197B	~25	>1.0	10.05	1.30	87.9	0.11	0.04	--

a. -15°C. Surveillance (U)

(U) No data have been obtained from the -15°C. surveillance of the Mg-doped, DPA treated hydride. Both sets of samples will be examined for hydrogen content and 60°C. stability after 6

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(C) Fig. 5 - Decomposition of Magnesium-Doped Aluminum Hydride-1451 at 40°C.

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months' storage time, which will be in February, 1968, and July, 1968 for each set, respectively. The oxygen content of the samples will be closely followed by periodic analysis during storage.

b. 25°C. Surveillance (U)

(U) No analysis or 60°C. stability data have been obtained on these samples stored at 25°C. The first sampling period will come in March and July, 1968, for the two sets of samples. However, continuous measurement of decomposition at 25°C. with a modified Taliani apparatus is being conducted and the results are shown in Table X.

Table X

(C) Surveillance of Magnesium-Doped, DPA Treated
Aluminum Hydride-1451 at 25°C.

Sample	% Decomposition ^a Storage Time, months		
	1	2	3
10056	0.005	0.008	0.013
11286	0.004	0.008	0.015
11306A	0.009	0.020	0.029
12066A	0.005	0.009	0.015
08117A	0.005	--	--
08187A	0.003	--	--
10107A	0.005	--	--
10197B	0.003	--	--

^aModified Taliani method

c. 40°C. Surveillance (U)

(U) The samples stored at 40°C. will be periodically analyzed for hydrogen content and will be checked for 60°C. stability. Continuous measurement of decomposition with a modified Taliani apparatus is also being conducted and available results are shown in Table XI.

Table XI

(C) Surveillance of Magnesium-Doped, DPA Treated
at 40°C.

Sample	% Decomposition ^a			
	Storage Time, months			
	1	2	3	4
10056	0.043	0.114	0.232	0.337
11286	0.053	0.159	0.356	0.524
11306A	0.083	0.215	0.576	1.027
12066A	0.041	0.108	0.217	0.330
08117A	0.005	--	--	--
08187A	0.038	--	--	--
10107A	0.029	--	--	--
10197B	0.015	--	--	--

^aModified Taliani method.

B. SURVEILLANCE OF ALUMINUM HYDRIDE-1451 PROPELLANT (U)

(C) The stability of neat aluminum hydride, discussed previously, is of utmost importance if the hydride were to behave similarly when formulated in propellants. The decomposition rates of the neat hydride could then be applied directly to determining the gas generation within the formulated propellant. However, there is a growing amount of evidence to show that the hydride does not decompose in a manner in propellant which is at all similar to the decomposition of neat aluminum hydride, particularly at ambient (25°C.) temperature.

(C) The gas generation of aluminum hydride propellants has been studied at 25°, 40° and 60°C. Three propellant formulations have been used in these studies, and are listed in Table XII. These formulations were used only to facilitate the study of the behavior of AlH₃-1451 in propellant environment; no other purpose is intended. The suggestions of other propellant contractors in our propellant studies are sincerely appreciated.

Table XII

(C) Aluminum Hydride-1451 Propellant Formulations

Ingredient	Weight, %		
	Propellant		Composite Thiokol ^c
	Double Base ABLA ^a	LPC ^b	
AlH ₃ -1451	25.0	20.80	25.00
AP	26.0	30.69 ^d	49.50
PGNC	12.0	--	--
NG	26.2	--	--
TEGDN	4.4	13.02	--
BDNPA	4.4	--	--
2-NDPA	1.0	--	--
Resorcinol	1.0	0.50	--
TMETN	--	21.63	14.00
ZL-437	--	--	10.00
HX-874	--	--	1.39
NPP-PNC	--	--	12.86
Ethyl Centralite	--	--	0.50

^a Formulation and assistance by Dr. John Alabashi,
Allegany Ballistics Laboratory.

^b Formulation and assistance by Dr. J. Martin,
Thiokol Chemical Company, Huntsville, Alabama.

^c Formulation and assistance by Dr. George Myers,
Lockheed Propulsion Company, Redlands, California.

^d AP is 70% 45 μ , 30% + 48 mesh.

1. 25°C. Surveillance (U)

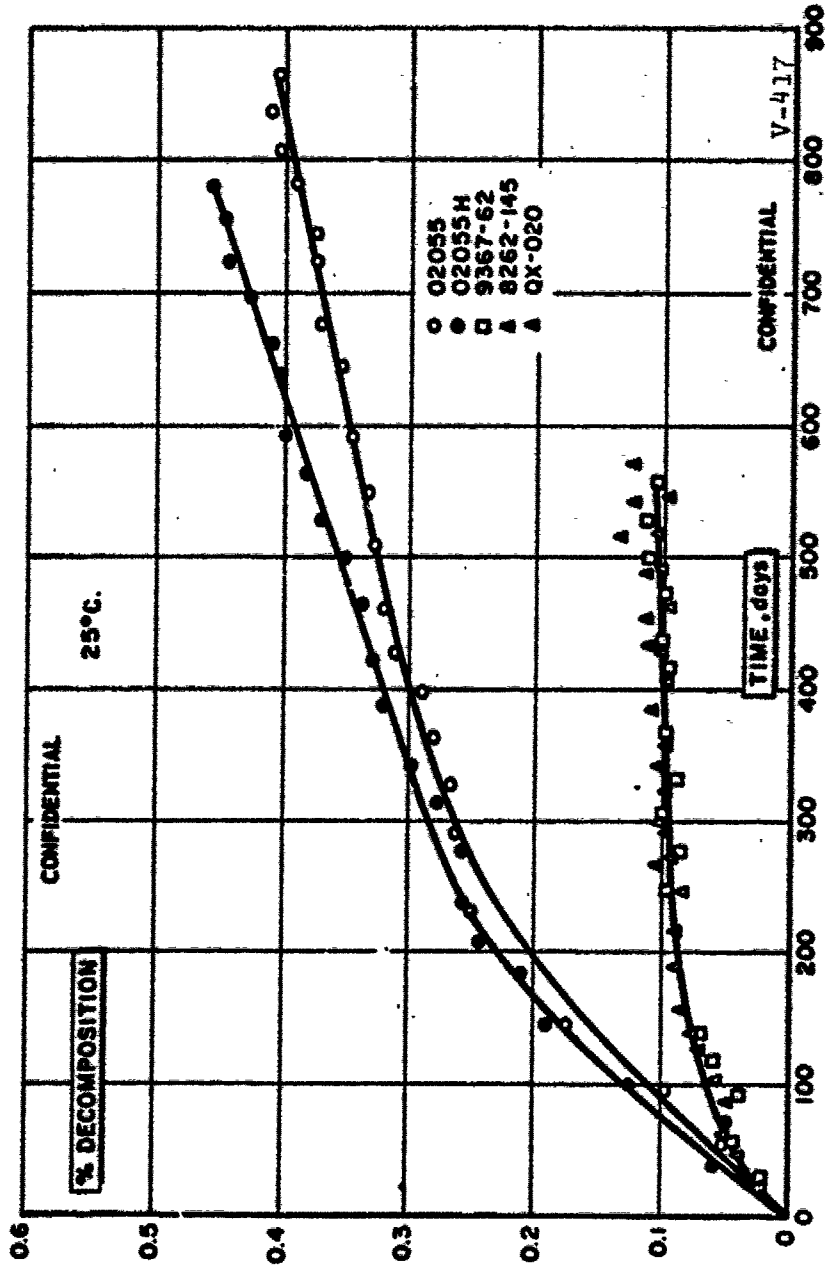
(C) The propellant samples which have been studied at 25°C. during the past year are listed in Table XIII. Five samples of aluminum hydride, including unstabilized macrocrystalline, surface hydrolyzed macrocrystalline, magnesium-doped, and magnesium doped DPA-treated AlH_3 -1451, continue to be studied in double base propellant.

Table XIII(U) Double Base Propellant Samples on 25°C. Surveillance Test

<u>Fuel Type</u>	<u>Date Placed on Test</u>	<u>Days on Test</u>	<u>% Decomposition</u>
Aluminum (reference)	8-04-65	Terminated	Negligible
AlH_3 - Lot 02055	8-04-65	865	0.40
AlH_3 - Lot 02055 - surface hydrolyzed	10-13-65	780	0.46
AlH_3 - Lot QX-020- magnesium-doped	5-20-66	571	0.126
AlH_3 - Lab sample 8262-145, magnesium-doped, <u>in situ</u> DPA-treated	6-06-66	557	0.099
AlH_3 - Lab sample 9367-62, magnesium-doped, <u>in situ</u> DPA-treated	6-07-66	556	0.107

(C) The decomposition of the AlH_3 -1451 propellant samples listed in Table XIII is plotted in Figure 6. The decomposition of the three magnesium doped varieties is shown to be very similar. The unstabilized macrocrystalline and surface hydrolyzed macrocrystalline samples show considerably poorer stability than the magnesium-doped material.

(C) The shape of the decomposition curve of aluminum hydride in double base propellant is significantly different from that of the neat material. A decrease in decomposition rate is observed with time instead of the normal acceleration of decomposition. At 25°C., double base propellant containing unstabilized macrocrystalline hydride decomposed approximately 0.28% during the first year, 0.09% during the second year, and 0.04% during the first half of the third year. Decomposition figures



(C) FIG. 5 - Decomposition of Aluminum Hydride-1451 in Double Base Propellant at 25°C.

assume all gas is generated by decomposition of the aluminum hydride, and gas generation measurements are by a modified Taliani method. Stabilized aluminum hydride, magnesium doped and DPA treated samples, decomposed only approximately 0.10% during the first year and very little over the first half of the second year in double base propellant at 25°C.

(U) The double base propellant samples under surveillance at 40°C. include those containing aluminum (reference), unstabilized macrocrystalline, magnesium-doped, and magnesium-doped, DPA-treated AlH₃-1451. One sample is being evaluated in a Thiokol composite propellant formulation; the others are in the ABL double base formulation. A list of the samples studied at 40°C. during the past year is given in Table XIV.

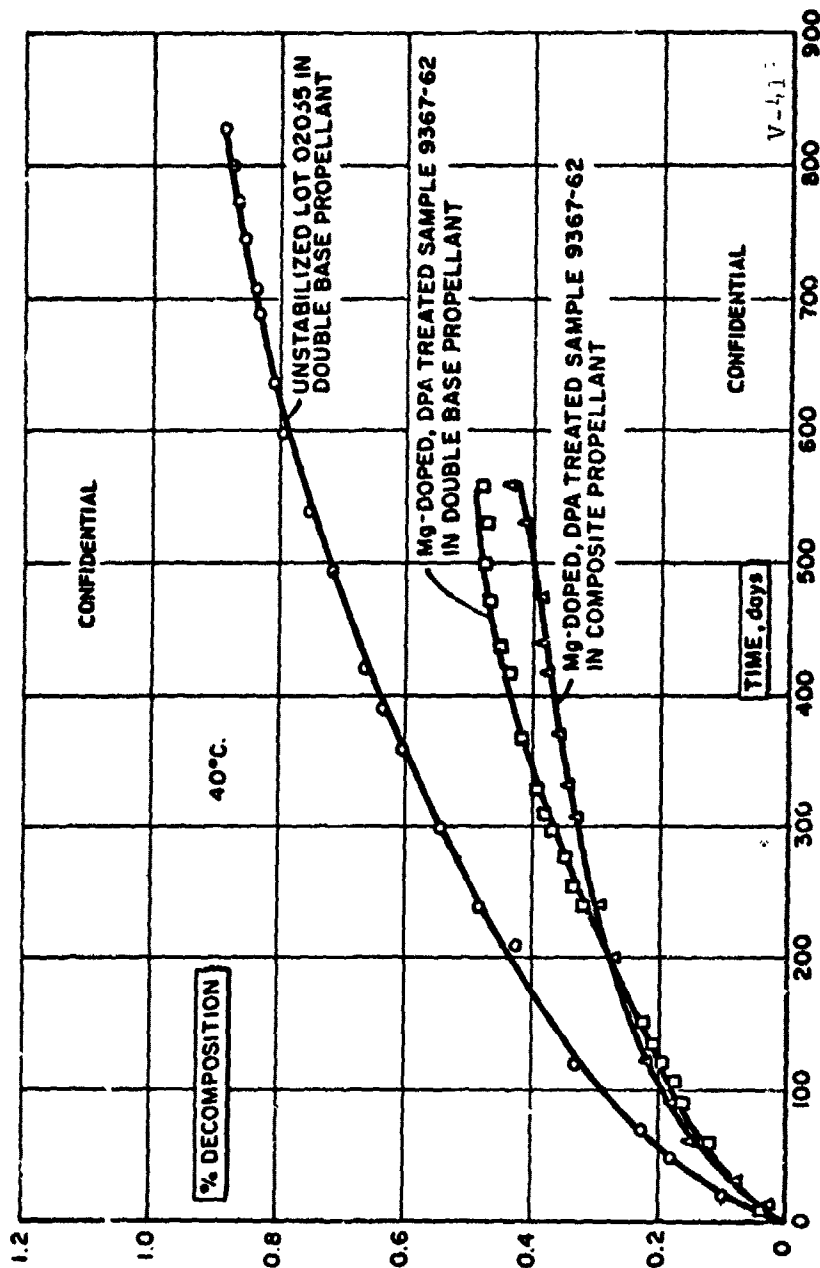
Table XIV

(U) Double Base Propellant Samples
on 40°C. Surveillance Test

Fuel Type	Date Placed on Test	Status, 12-15-67	
		Days on Test	% Decomposition
AlH ₃ - Standard Lot 02055	9-8-65	828	0.89
AlH ₃ - Lot QX-020 - magnesium-doped	4-14-66	Terminated	1% at 359 days
AlH ₃ - Lab Sample 9367-62 - magnesium-doped, <u>in situ</u> DPA-treated ^a	6-08-66	555	0.42
AlH ₃ - Lab Sample 9367-62 - magnesium-doped, <u>in situ</u> DPA-treated	6-07-66	556	0.49
AlH ₃ - Lab Sample 8262-145, magnesium-doped, <u>in situ</u> DPA-treated	6-06-66	557	0.70
Aluminum (reference)	9-08-65	828	Negligible

^aEvaluated in Thiokol composite formulation.

(C) The decomposition rates observed from some of these samples listed in Table XIV are shown in Figure 7. The aluminum (reference) sample shows very little gassing and is not included. Again the magnesium-doped, DPA treated hydride sample 9367-62 is shown to produce much less gassing than unstabilized hydride, in both double base and composite propellant. However, some



(c) Fig. 7 - Decomposition of Aluminum hydride-1451 in Double Base Propellant at 40°C.

unexplainable results have been observed in the 40°C. propellant surveillance program. The magnesium-doped sample QX-020, which was shown to be quite stable in 25°C. tests presented earlier, followed a more typical "autocatalytic" shaped decomposition curve in the propellant at 40°C., reaching 1% decomposition after 359 days. This is similar to earlier results with surface hydrolyzed unstabilized hydride which was reported previously (1) to have reached 1% decomposition at 264 days in double base propellant at 40°C. On the other hand, sample 8262-145 (magnesium-doped, DPA treated lab sample) which looked very good at 60°C. has exhibited a large amount of initial gassing at 40°C., and, although the gassing rate is decreasing with time, the gas generation curve more closely follows the unstabilized sample than the other magnesium-doped, DPA treated aluminum hydride propellant samples.

(C) Although these discrepancies are present it is still evident that aluminum hydride in propellant at 40°C. more often follows a decreasing gas generation rate curve than the accelerating gas generation associated with decompositions of neat hydride. A likely cause of this phenomenon is the presence of some moisture or other reactive impurity in the propellant.

c. 60°C. Surveillance (U)

(C) Several different types of AlH_3 -1451, including unstabilized, magnesium-doped, magnesium-doped, DPA treated, and "aged" magnesium-doped hydride have been evaluated in propellant at 60°C. Remarkable stability has been observed from AlH_3 -1451 propellants containing the stabilized hydride. A few of the most stable samples remained on test at the time of the last report (1) and have continued into this program.

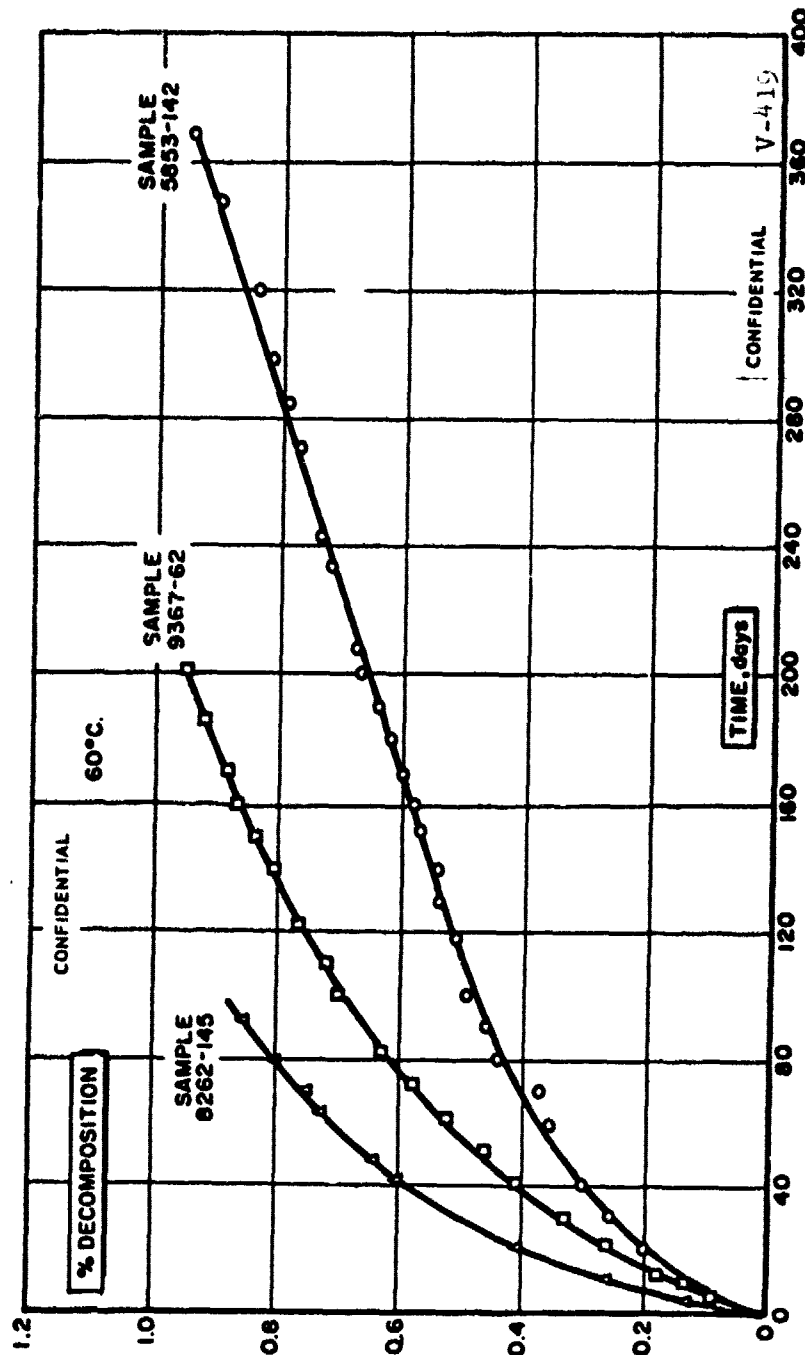
(C) Figure 8 shows the results obtained from these samples in ABL double base propellant at 60°C. Shown in Figure 8 are lab samples 8262-145 and 9367-62, magnesium-doped, DPA treated material, and sample 5853-142 which is a magnesium-doped sample that had been aged at -15°C. prior to formulation. The magnitude of improvement in stability of these formulations containing stabilized aluminum hydride is tremendous as formulations containing unstabilized hydride reached 1% decomposition in less than 10 days at 60°C.

(C) The effect of moisture in the propellant cannot be discounted. Shown in Figure 9 is the decomposition of three samples of ABL double base propellant containing unstabilized hydride. The effect of adding very small amounts of H_2O to the propellant formulation is amazing. The "autocatalytic" process of decomposition is essentially stopped. Unfortunately, however, the initial gassing of hydride exposed to water would be very detrimental to propellant.

¹The Dow Chemical Company, Annual Technical Report, AFRPL-TR-67-37, January, 1967, Contract Nr. AF 04(611)-11400, Dow Report Nr. FS-4Q-66.

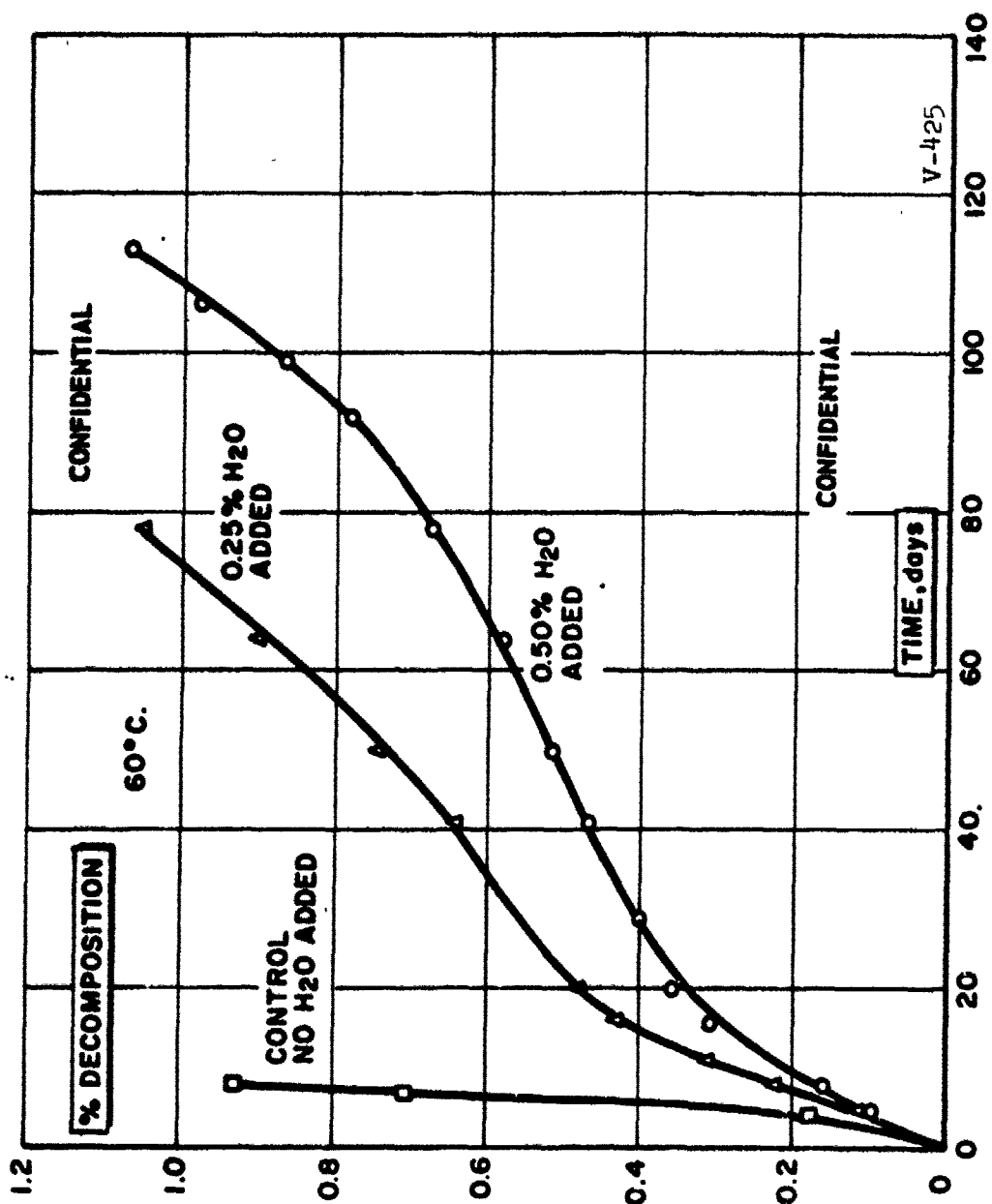
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(C) Fig. 8 - Decomposition of Specimen High Stability Aluminum Hydride-1451 Samples in Double Base Propellant at 60°C.

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(C) Fig. 1 - Effect on 60°C. Decomposition of Adding Small Amounts of Water to Double Base Aluminum Hydride-1451 Propellant

C. STABILITY IMPROVEMENT STUDIES (U)

(C) Although large improvements in the thermal stability of aluminum hydride have been accomplished with the magnesium-doping and DPA treatment techniques, additional improvements may eventually be required for long term propellant storage. This program is not intended as a study of methods of improving hydride stability, but as a direct result of studying the stability of various samples some "fallout" information has led to a few experiments which have produced valuable data on stabilization techniques. In addition, a large number of samples have been made available to this program by work under another contract, AF O4(611)-11606, and the stability of these samples will be reported in this section.

1. Effect of Water on Aluminum Hydride-1451 Stability (C)

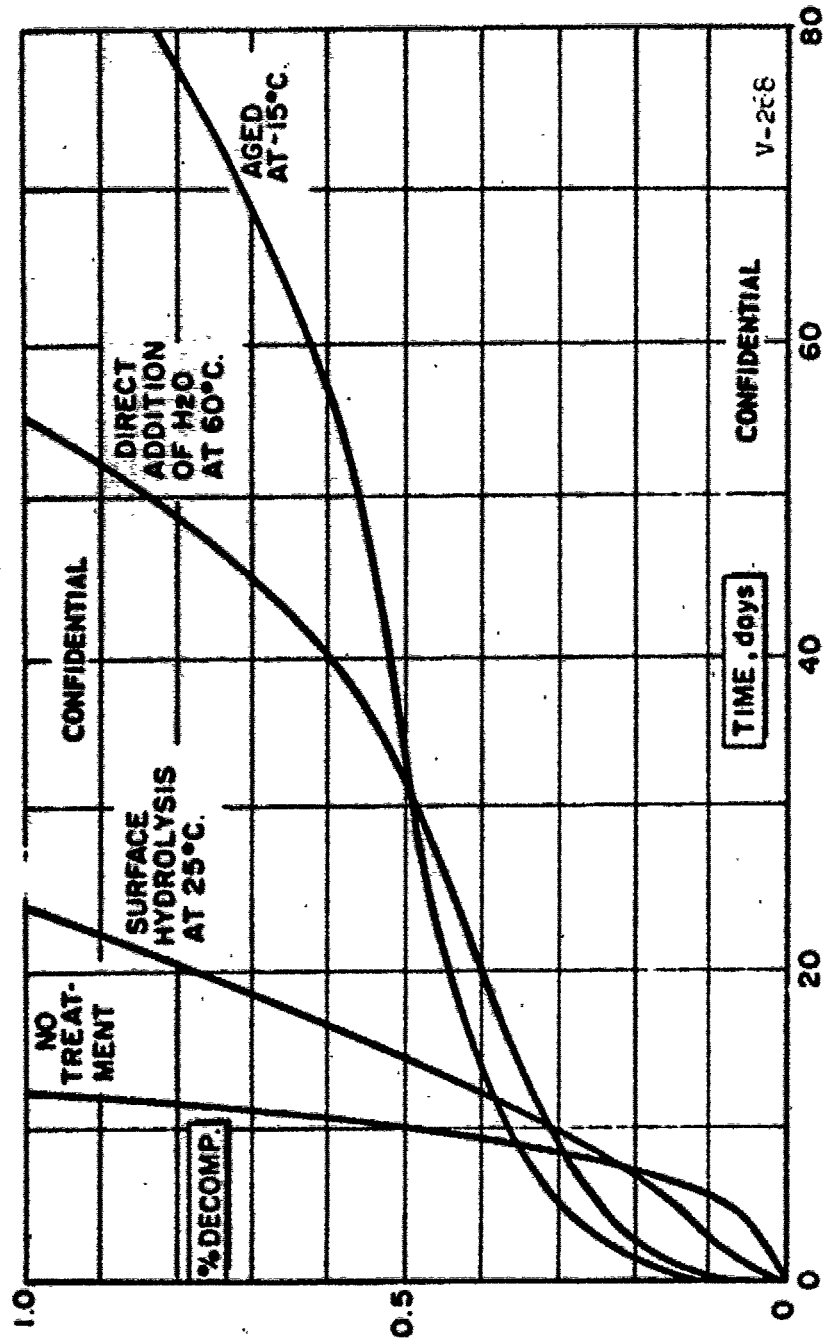
(C) Figure 10 shows the general stability of AlH_3 -1451 after placing water on the surface by different means. The trend is the same in all cases, and the differences between the samples may be caused only by the amount of water present. In general, water present in the environment to which the hydride is subjected will result in gassing during the reaction period, "initial gassing", followed by improved stability of the hydride.

(C) Figure 11 shows the effect of adding 1 wt. % water directly to the sample at the beginning of the modified Taliani stability test at 60°C. In this case, the reaction period is about 5-10 days and the stability after the reaction is about 3 to 4 times better than the untreated hydride. It is important to note that two hydride samples were thoroughly degassed at 5 and 10 days, after the reaction period, with no loss in the improved stability. This indicates that aluminum hydride, treated with 1 wt. % water and heated at 60°C. for 5 days or more, could then be used without subsequent initial gassing and with improved stability.

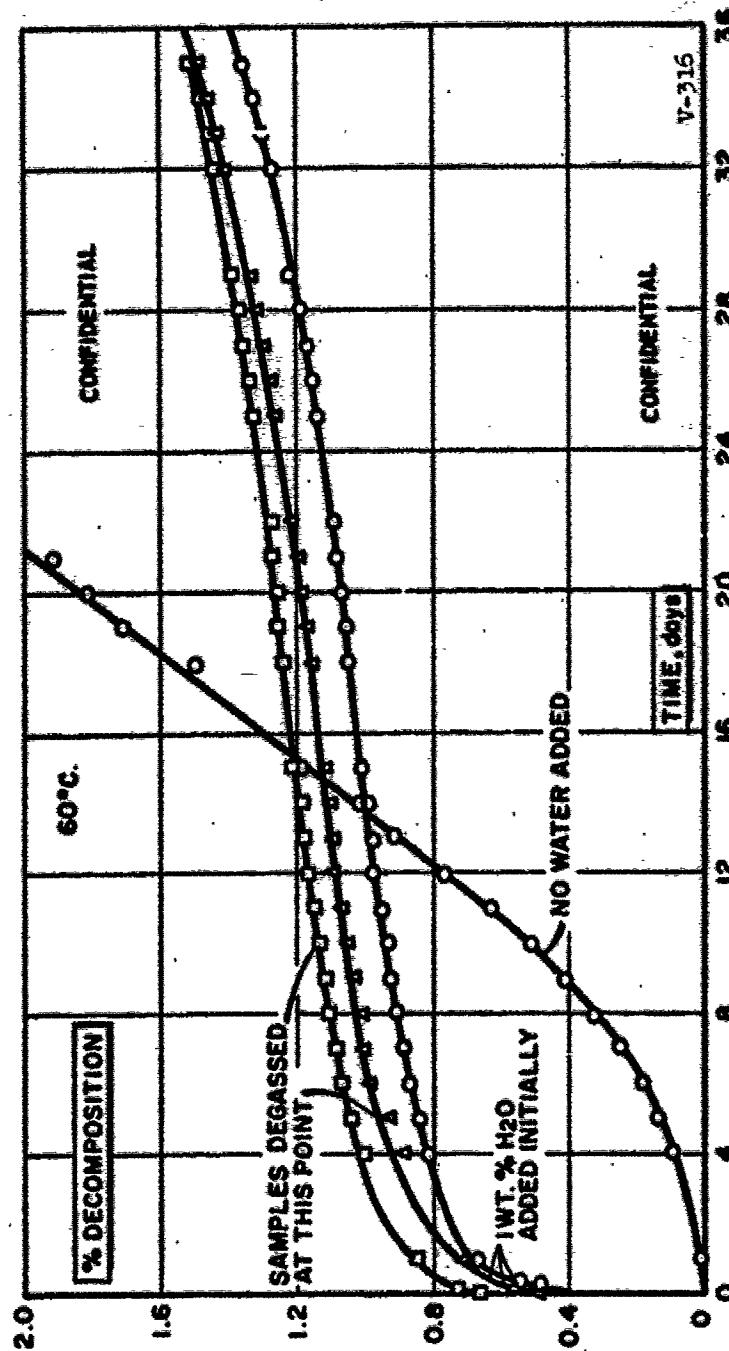
(C) The effect of water on the decomposition of aluminum hydride is just as dramatic when partially decomposed hydride is used. Shown in Figure 12 is the decomposition of a pair of samples of magnesium-doped aluminum hydride. After 1% decomposition had been passed, 1 wt. % water was introduced to the test apparatus containing one of the samples. Immediately a considerable amount of gassing occurred, but stopped after only a few days and practically no decomposition occurred over the following 30 days.

2. Treatment of Aluminum Hydride with Liquids Containing Water (Data from Work Under Contract Nr. AF O4(611)-11606) (C)

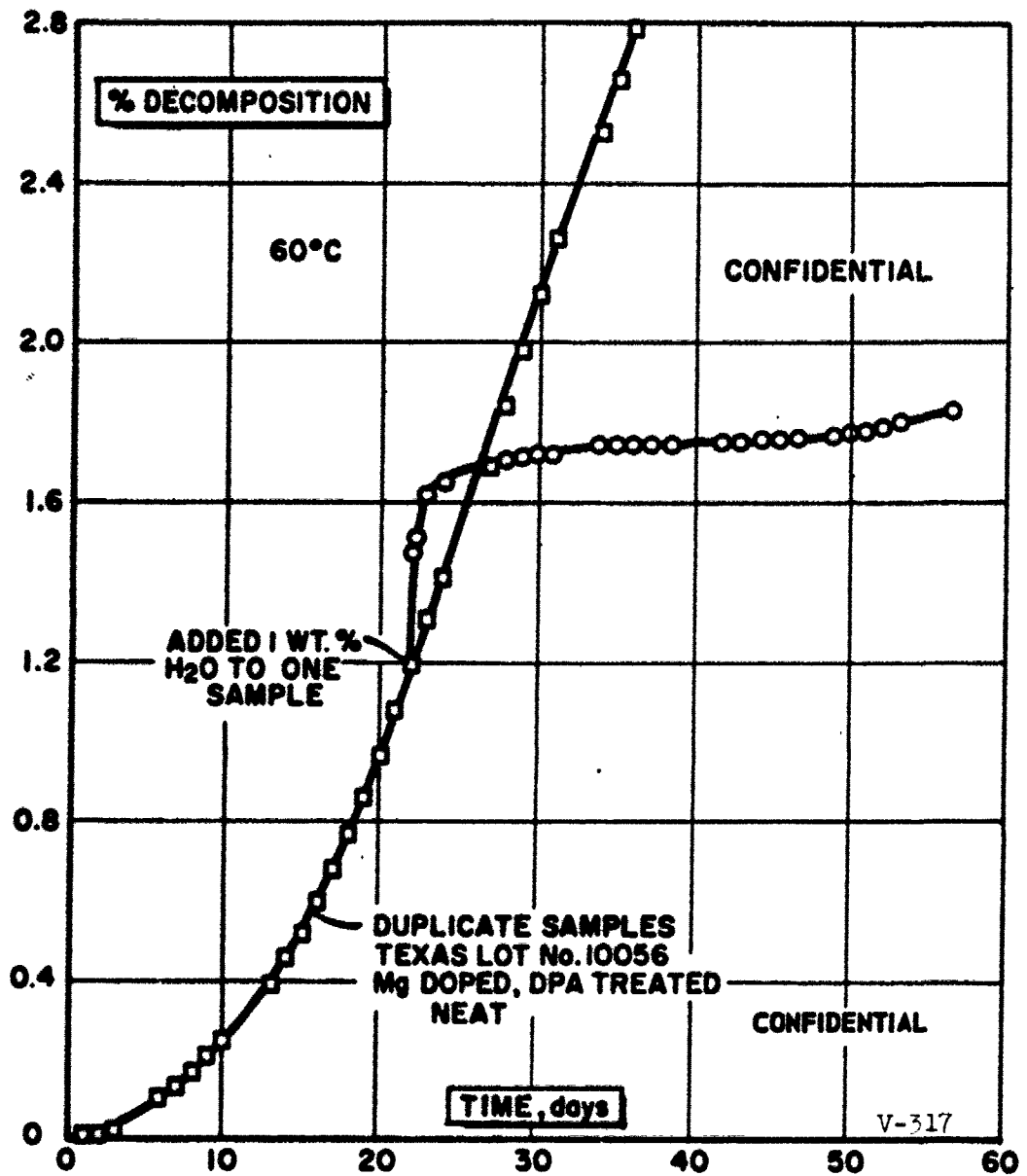
(C) The effect of small amounts of water in liquids on the decomposition of aluminum hydride formulated in the liquids is dramatic. Of particular interest is the stability of heterogeneous aluminum hydride/hydrazine gelled propellant. The results of studies of aluminum hydride in hydrazine are shown in Figure 13. Examination of the hydride by a metallographic



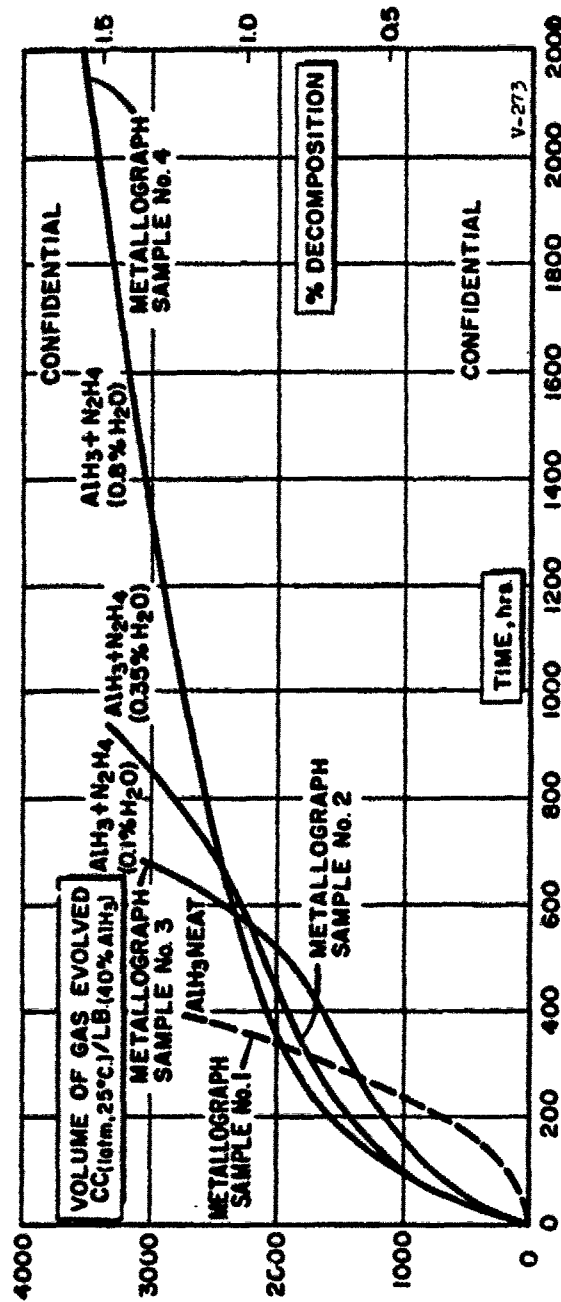
(C) Fig. 10 - Effect of Water on the Decomposition of Aluminum Hydride-1451 at 0°C.



(C) Fig. 11 - Effect of Addition of 1 Wt. % Water to Aluminum Hydride-1451 at 60°C.



(C) Fig. 12 - Effect of Addition of 1 Wt. % Water to Partially Decomposed Aluminum Hydride-1451



(C) FIG. 13 - Effect of Hydrazine at Various Water Contents on the Decomposition of Aluminum Hydride-1451 at 60°C.

technique at the sampling points indicated the presence of metal in the neat sample but none in the sample formulated in as-received hydrazine (0.8% H₂O). It was further learned that hydride recovered after long periods in the as-received hydrazine exhibited much improved stability, both neat and when reformulated in hydrazine. (A more detailed discussion of this study can be found in Quarterly reports of work performed under Contract Nr. AF 04(611)-11606).

(C) Work was then directed at studying other liquids, water concentrations, and temperatures to increase the stability of the hydride and to make the treatment more feasible. The samples produced by the screening program conducted were evaluated for neat stability as well as stability in hydrazine. The neat stability data are of considerable interest to this program and are included in Table XV. Essentially, the program resulted in the choice of n-butylamine containing approximately 2.0% water as the treatment liquid, and conditions of 60°C. for about 17 days, although longer treatment may further enhance the stability of the hydride. This treatment was then scaled up to treat 3/4 lb. batches of aluminum hydride. The resultant 60°C. stability of the hydride is shown in Figure 14 for the laboratory sample and for the scale-up batches. A sample of the first scale-up batch has been included in the surveillance program to determine its decomposition rate at 25° and 40°C. No measurable decomposition has occurred after 2-1/2 months at 25°C. The available results at 40°C. are plotted in Figure 15 and are compared to an untreated sample with stability similar to the material used in the scaleup.

D. CORRELATION OF STABILITY DATA (U)

(C) The decomposition data obtained from the various aluminum hydride samples in this surveillance program are very useful in correlating the observed decomposition rates at ambient (25°C.) temperature to accelerated stability tests at 60°C., and correlating the stability of the samples to other characteristics such as impurities, doping agents, surface treatment effects. The most significant correlations developed from data obtained in this program are discussed in this section.

1. Correlation of 25° with 60°C. Stability Data (U)

(C) The temperature during the storage of solid rocket propellant motors containing aluminum hydride will determine the rate of decomposition of the hydride. It is anticipated that this temperature will be close to 25°C. It is therefore necessary to know with some certainty the long term (up to 5 years) decomposition rate of the hydride at 25°C. Obviously, this cannot be measured immediately at the time the rocket motors are formulated. An accelerated stability test must be used to predict the long term 25°C. decomposition rate.

Table IV
Stability and Analysis of Toxic Substances in Human Blood

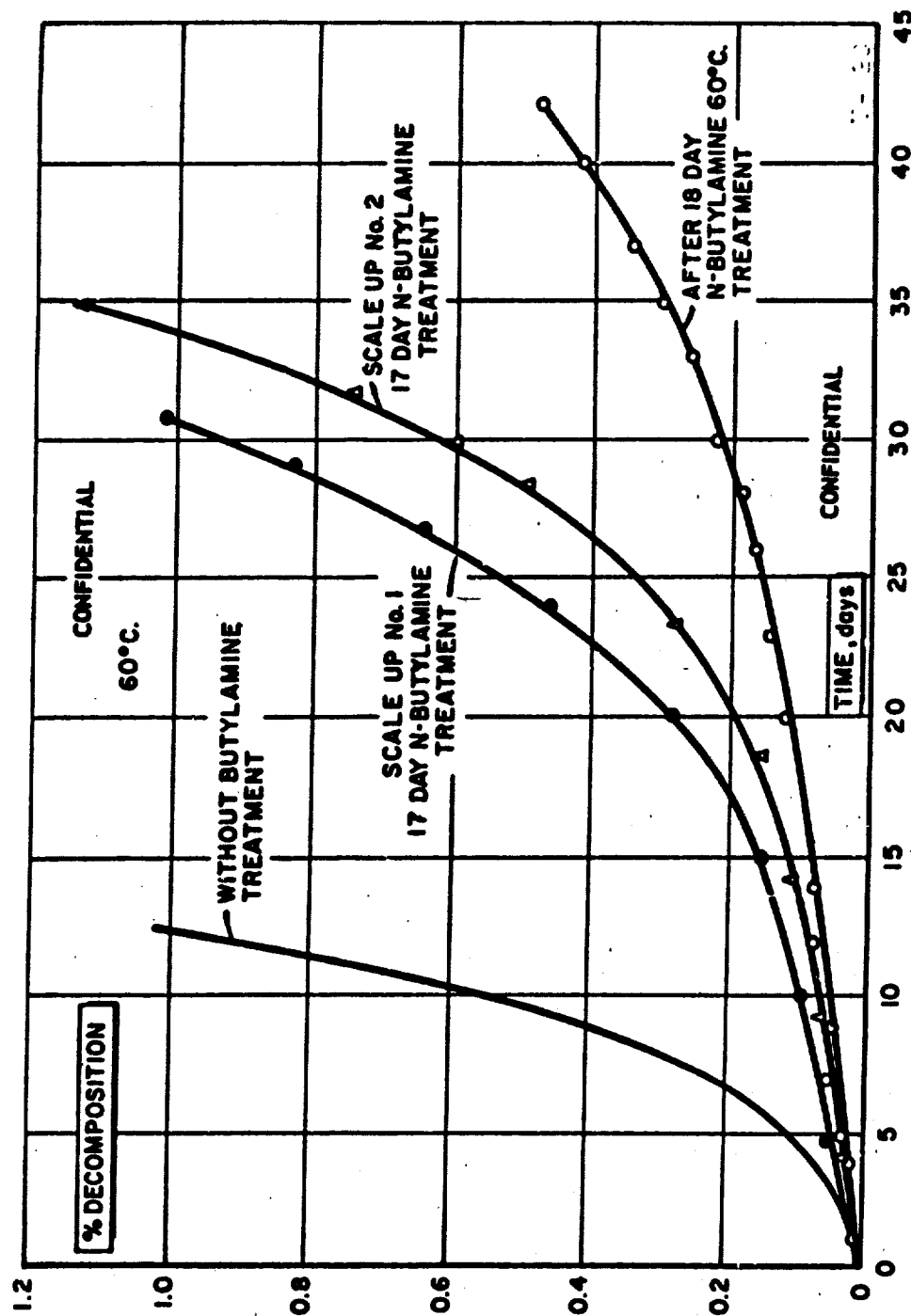
Sample Name	Hydrate	Exposure	Toxicant				Analysis				Remarks
			Conc.	Time	Temp.	Pressure	Method	Reagent	Time	Temp.	
1-13-1	Oxide	---	---	---	---	---	---	---	---	---	Control
1-13-2	Oxide	H ₂ O	0.1	12	---	---	---	---	---	---	---
1-13-3	Oxide	H ₂ O	0.1	12	---	---	---	---	---	---	---
1-13-4	Oxide	H ₂ O	0.1	6	10	---	---	---	---	---	---
1-13-5	Oxide	H ₂ O	0.1	6	10	---	---	---	---	---	---
1-13-6	Oxide	H ₂ O	0.1	12	10	---	---	---	---	---	---
1-13-7	Oxide	H ₂ O	0.1	6	10-70	---	---	---	---	---	Sample out.
1-13-8	Oxide	H ₂ O	0.1	6	10	---	---	---	---	---	2/1 10/10
39-1-1	Oxide	H ₂ O	0.1	12	10	---	---	---	---	---	2/1 10/10
1-12-1	Oxide	H ₂ O	0.1	12	10	---	---	---	---	---	---
1-13-2	Oxide	H ₂ O	0.1	6	10	---	---	---	---	---	---
39-1-1	Oxide	n-Butylamine	1.11	6	60	---	---	---	---	---	Stirred
39-1-2	Oxide	n-Butylamine	1.11	6	60	---	---	---	---	---	---
39-1-3	Oxide	n-Butylamine	2.05	18	60	---	---	---	---	---	---
1-13-4	Oxide	n-Butylamine	2.05	6	10	---	---	---	---	---	---
1-13-5	Oxide	n-Butylamine	2.05	6	60	---	---	---	---	---	Sample out.
39-1-1	Oxide	n-Butylamine	2.05	6	60	---	---	---	---	---	0.05% H ₂ O work
39-1-2	Oxide	iso-Butylamine	1.11	6	60	---	---	---	---	---	---
39-1-3	Oxide	iso-Butylamine	1	6	60	---	---	---	---	---	---
39-1-4	Oxide	iso-Butylamine	1.01	6	60	---	---	---	---	---	---
39-1-5	Oxide	iso-Butylamine	1	6	60	---	---	---	---	---	Not determined
1-13-6	Oxide	Benzene	0.5	6	60	---	---	---	---	---	---
1-13-7	Oxide	Benzene	0.5	6	60	---	---	---	---	---	---
1-13-8	Oxide	Benzene	0.5	12	10	---	---	---	---	---	---
1-13-9	Oxide	Benzene	1.2	6	10	---	---	---	---	---	---
1-13-10	Oxide	Benzene	1.2	12	10	---	---	---	---	---	---
1-13-11	Oxide	n-Propylamine	0.61	6	60	---	---	---	---	---	---
1-13-12	Oxide	FLAC	0.61	6	60	---	---	---	---	---	---
1-13-13	Oxide	FLAC	0.61	6	10	---	---	---	---	---	---
1-13-14	Oxide	FLAC	1.01	6	10	---	---	---	---	---	---
1-13-15	Oxide	THF	0.1	6	60	---	---	---	---	---	---
39-1-1	Blood	---	---	---	---	---	---	---	---	---	Control
39-1-2	Blood (1)	n-Butylamine	1.01	12	60	---	---	---	---	---	Stirred (3/4 lb.)
39-1-3	Blood	n-Butylamine	---	9	60	---	---	---	---	---	Stirred (1/4 lb.)
39-1-4	Blood	n-Butylamine	---	12	10	---	---	---	---	---	Stirred (1/4 lb.)
39-1-5	Blood	n-Butylamine	---	---	---	---	---	---	---	---	---
39-1-6	Blood	n-Butylamine	---	---	---	---	---	---	---	---	---
39-1-7	Blood	n-Butylamine	---	---	---	---	---	---	---	---	---
39-1-8	Blood	n-Butylamine	---	---	---	---	---	---	---	---	---
39-1-9	Blood	n-Butylamine	---	---	---	---	---	---	---	---	---
39-1-10	Blood	n-Butylamine	---	---	---	---	---	---	---	---	---

*Analysis by neutron activation; all other analyses by standard chemical methods.

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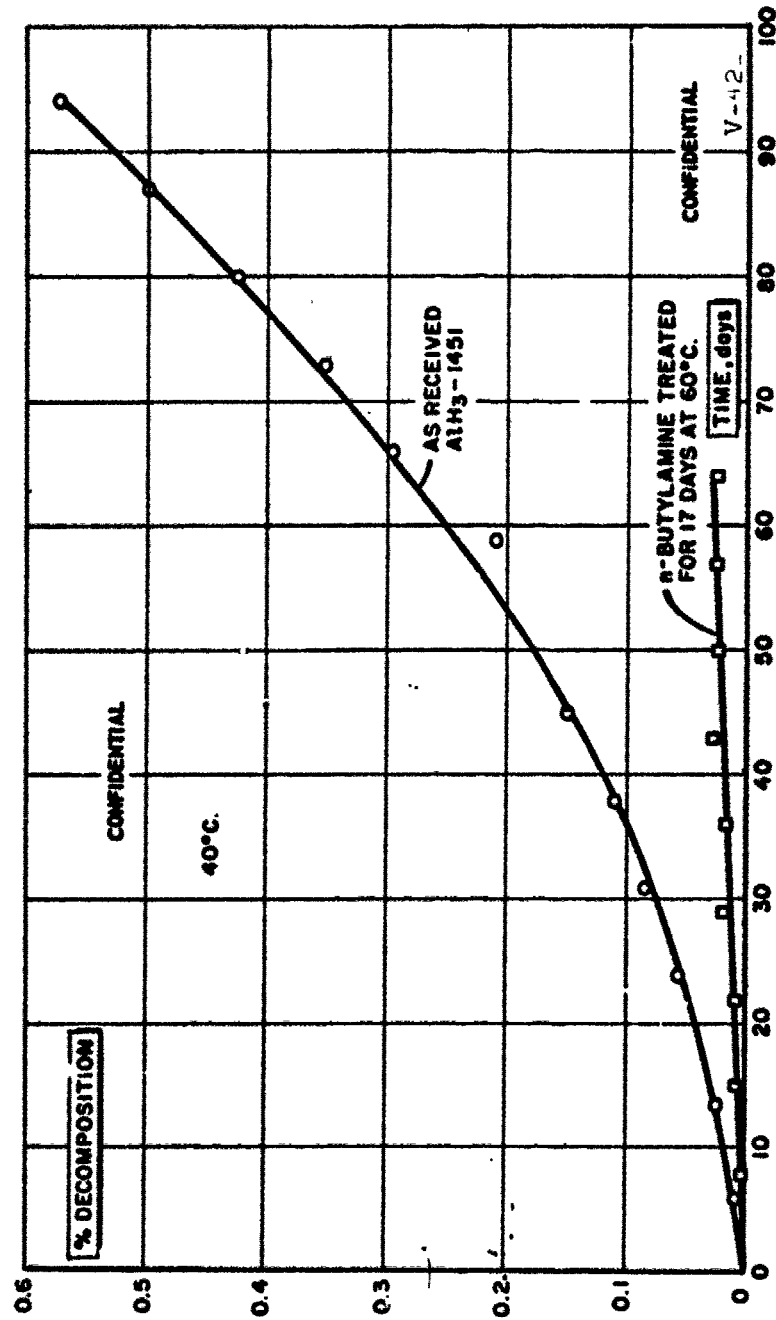
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(c) FIG. 14 - Effect of n-butylamine treatment on the stability of Alkylamine Oxide-1953

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(c) Fig. 15 - Effect of n-Butylamine Treatment on the 40°C. Stability of Aluminum Hydride-1451

(C) Figure 16 shows the relationship of 25°C. stability to 60°C. stability in terms of time to reach 0.05% decomposition at 25°C. vs. time to reach 1% decomposition at 60°C. This curve is drawn for five established data points and one extrapolated point as indicated. The results strongly indicate much more than a one to one relationship of 25°C. stability to 60°C. stability; i.e., a twofold stability improvement in 60°C. stability corresponds to at least a fivefold improvement in 25°C. stability. This indicates that small additional improvements in 60°C. stability will result in aluminum hydride with excellent 25°C. stability.

2. Correlation of Stability to Lithium Content (C)

(C) It has been observed that the stability of aluminum hydride samples is somewhat related to the lithium content of the samples, particularly the lithium content in excess of that believed to be lithium chloride. Figure 17 shows the 60°C. stability (days to reach 1% decomposition at 60°C.) plotted vs. the excess lithium content of the surveillance samples. The data plotted represent the 12 most recent samples received from the Texas Pilot Plant, and all samples are magnesium-doped; some are DPA treated. Specific information on the samples has been presented in Tables V and IX.

(C) Figure 17 shows that there is an inverse relationship between lithium content and stability, i.e., samples high in lithium are less stable, and samples low in lithium are more stable. This strongly indicates that lithium may be taking part in the process of decomposition. It must be remembered, however, that other variables, particularly crystal size and perfection have a definite effect on the decomposition process which occurs predominantly on the surface of the hydride crystals.

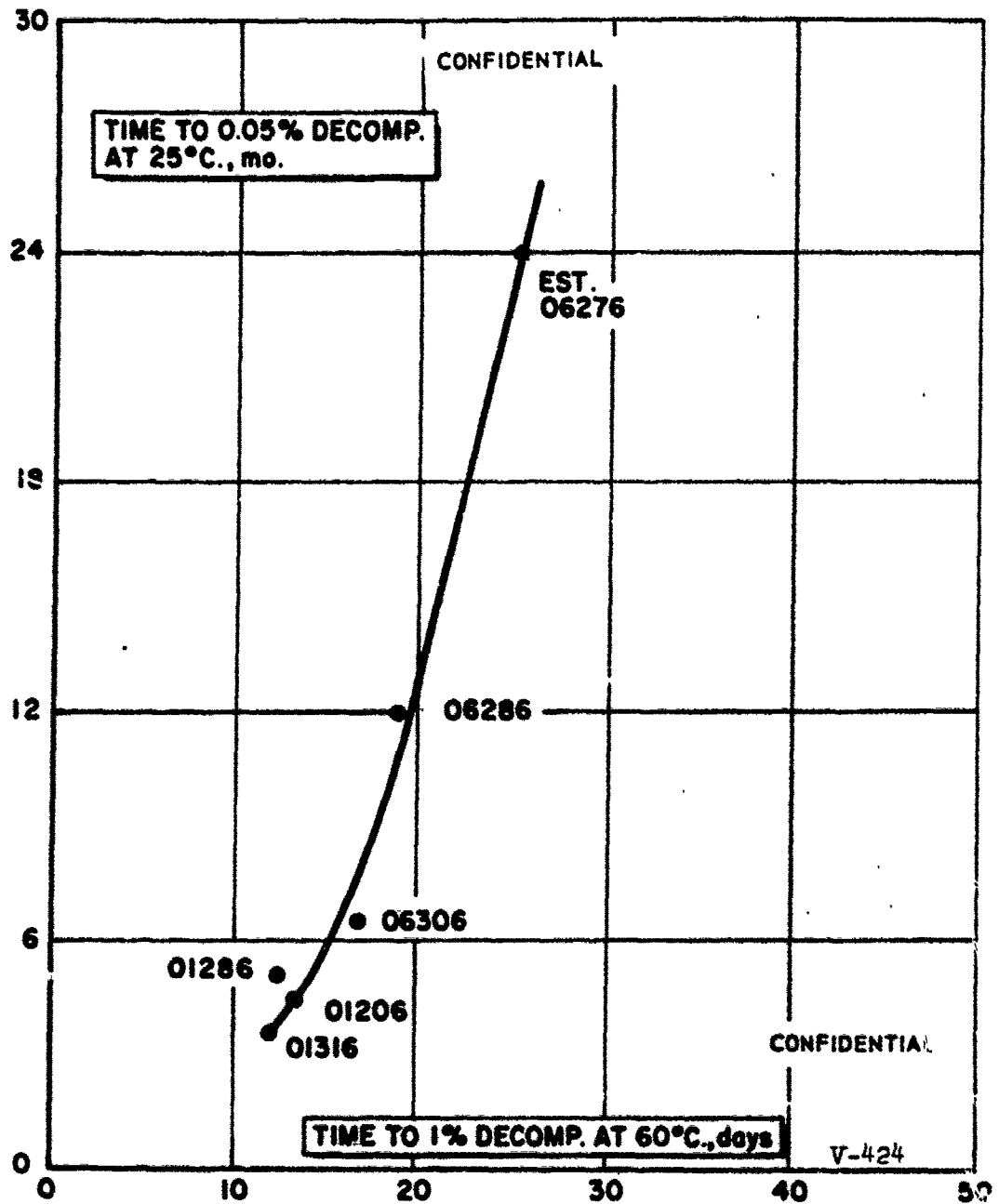
(C) Further consideration of the effect of lithium on stability is being given on this program as well as others (1,2). The presence of the lithium is caused by the need to use excess lithium aluminum hydride (LAH) in the desolvation of aluminum hydride etherate to the non-solvated 1451 crystalline phase. The mechanism by which the excess LAH enables the desolvation is now known, but attempts to desolvate aluminum hydride etherate without the LAH present have not succeeded.

3. Correlation of the Stability of Surface Treated Samples to Oxygen Content (C)

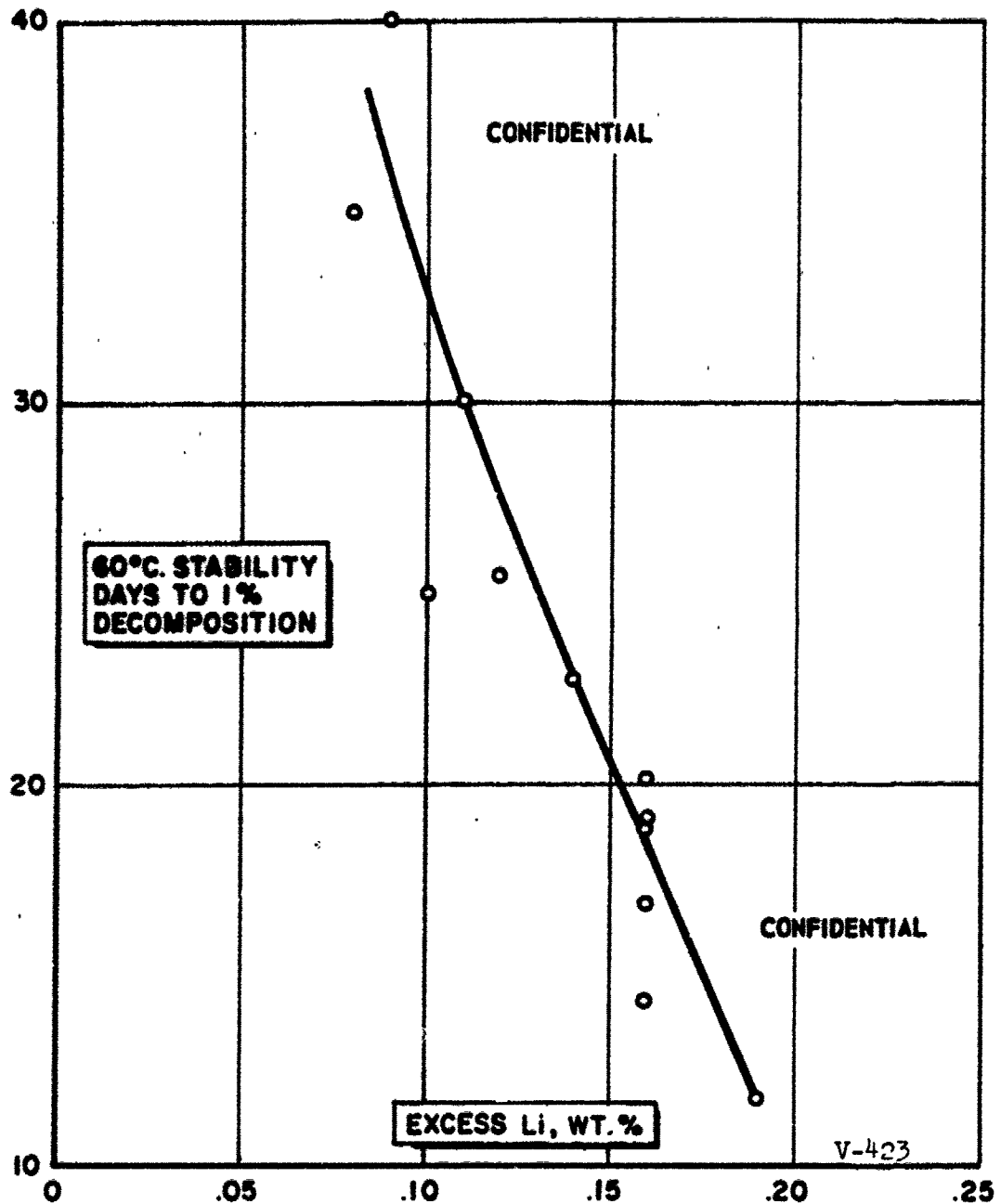
(C) Surface hydrolysis of aluminum hydride has been known for some time to have a definite effect on the stability of the hydride. New procedures, such as the n-butylamine treatment,

¹Contract Nr. F04611-63-C-0021, "Thermal Stability and Kinetic Studies of AlH_3 ," The Dow Chemical Company, Midland, Michigan.

²Contract Nr. F33615-67-C-1619, "LMH-1 Process Development," The Dow Chemical Company, Midland, Michigan.



(U) Fig. 16 - Relationship of Long-Term 25°C. Stability to Accelerated 60°C. Stability Tests



(C) Fig. 17 - Relationship of Stability to Lithium Content for Surveillance Samples

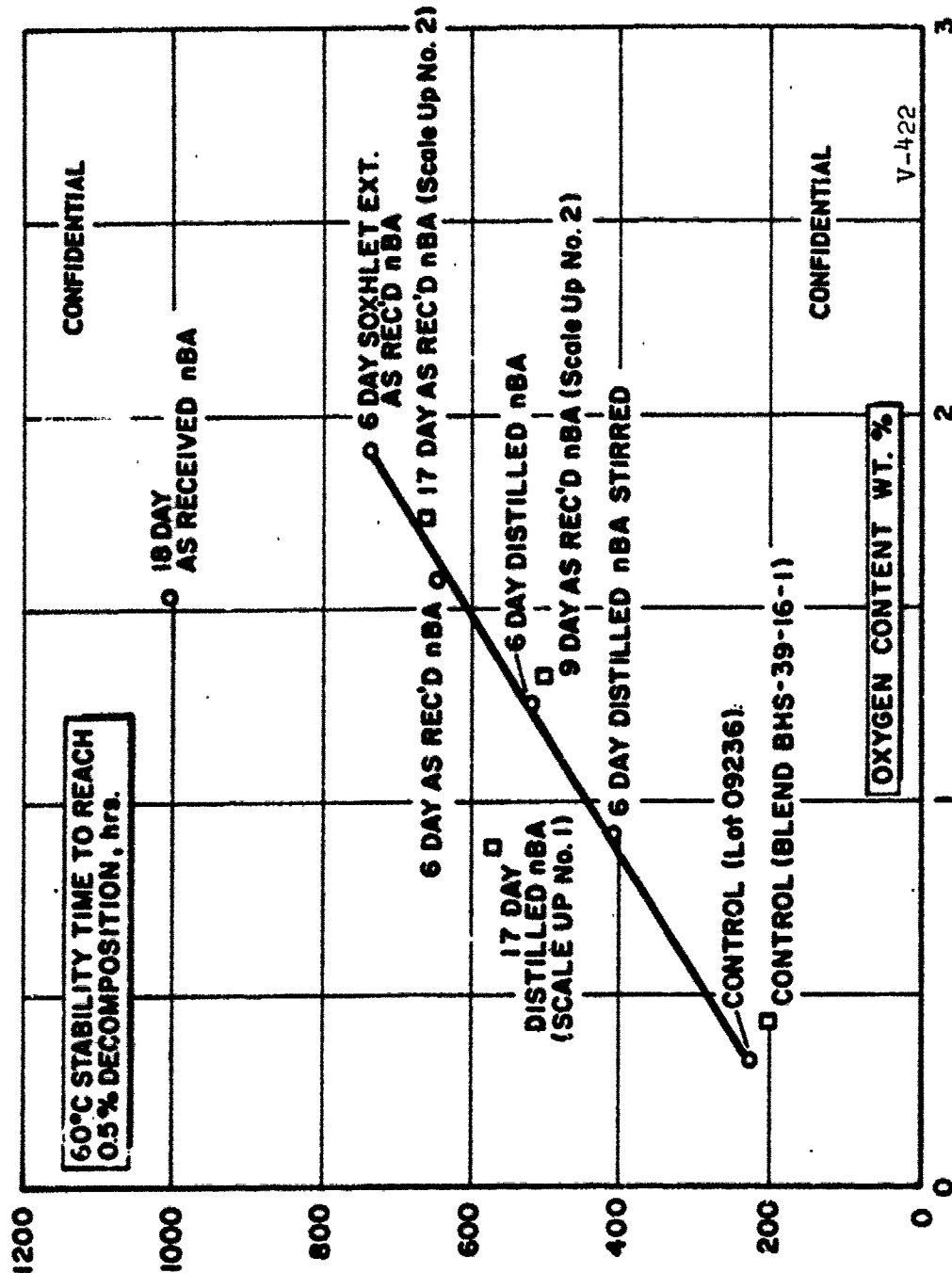
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are believed to be simply a controlled hydrolysis. However, some interesting trends can be seen when the stability of the treated samples is plotted vs. oxygen content.

(C) In this case the data shown in Table XV relate the stability of n-butylamine treated samples (time to reach 0.5% decomposition at 60°C.) to the oxygen content of the samples. The results are shown in Figure 18. Although these results do show a strong correlation of stability to oxygen content, an effect of treatment time is also observed. This indicates that something other than gross surface hydrolysis is occurring. Considering the relationship of stability to lithium discussed previously, the possibility must be considered that during the period of surface treatment some of the lithium compound which may catalyze decomposition is destroyed as it reaches the surface of the hydride crystals. Further investigations to substantiate this hypothesis are planned.

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(C) Fig. 16 - Relationship of Stability of n-Butylamine Treated Samples to Oxygen Content and Treatment Time

SECTION II

(U) CONCLUSIONS AND FUTURE WORKA. CONCLUSIONS (U)

(C) Based on the results obtained during this reporting period, the following conclusions are made:

- (i) Improvement in the 60°C. stability of aluminum hydride by a factor of two is accompanied by a factor of four to five improvement at 25°C. for very low levels of decomposition.
- (ii) A magnesium-doped sample which exhibited a 60°C. stability of 25.5 days to 1% decomposition has decomposed only 0.019% after one year at 25°C. Many samples have now been made with even better 60°C. stability and are undergoing surveillance studies.
- (iii) The 60°C. stability of aluminum hydride appears to be related to the amount of lithium present. The unknown lithium compound present may act as a catalyst for decomposition.
- (iv) The long-term stability of aluminum hydride propellant is characteristically better than for neat hydride and decomposition rates decreased with time in most propellant samples. "Initial gassing" is a problem and may be related to trace amounts of moisture present in the formulations.
- (v) Treatment of aluminum hydride in certain wet solvents, such as n-butylamine, for extended periods of time at elevated temperature results in much improved stability which is related to both the oxygen content of the sample and the treatment time and temperature.

B. FUTURE WORK (U)

(C) The following work is planned for the second year of this three-year program:

- (i) Continue surveillance of samples now on test until they are fully characterized.
- (ii) Add new samples of aluminum hydride-1451 to the surveillance program as they become available through efforts on Contract Nrs. FO4611-68-C-0021 and FO33615-67-C-1619.

- [REDACTED]
- [REDACTED]
- (iv) Continue to feedback stability correlations observed with surveillance samples to efforts under Contracts F04611-68-C-0021 and F33615-67-C-1619.
 - (v) Further substantiate the relationship between 60°C. stability and long term decomposition rates at 25°C. to enable valid extrapolations for samples with improved 60°C. stability.

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13 ABSTRACT Surveillance has continued on various samples of aluminum hydride including: unstabilized macrocrystalline, magnesium-doped, and magnesium doped, DPA (diphenylacetylene) treated AlH_3-1451 . These samples are providing valuable decomposition rate data both neat at -15° , 25° and $40^\circ C.$, and in propellant at 25° , 40° and $60^\circ C.$		
Results of surveillance of neat aluminum hydride continue to indicate greater improvement in $25^\circ C.$ stability than was observed with these samples at $60^\circ C.$ Two samples which reached 1% decomposition in 18.8 and 25.5 days at $60^\circ C.$ have decomposed only 0.05 and 0.019% respectively after 1 year at $25^\circ C.$ Results of surveillance of aluminum hydride propellant are continuing to show decreasing gas generation rates with time. At $25^\circ C.$, double base propellant containing unstabilized macrocrystalline hydride decomposed approximately 0.28% and 0.09% during the first and second year respectively and samples containing stabilized aluminum hydride, decomposed only approximately 0.10% during the first year.		
Small amounts of water in the environment which the aluminum hydride is subjected to have been found to prevent the typical "autocatalytic" shaped decomposition curve. Instead, an "initial gassing" period is observed followed by excellent stability. Treatments utilizing small amounts of hydrolysis to improve stability are being developed.		
The stability of neat aluminum hydride has been found to correlate inversely with the amount of lithium, other than lithium chloride, present as an impurity in the hydride. Excess lithium aluminum hydride is required in production of AlH_3-1451 of the desolvation of aluminum hydride etherate.		

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14. KEY WORDS: Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of index rules and weights is optional.

CONFIDENTIAL

Security Classification

SUPPLEMENTARY

INFORMATION



THE DOW CHEMICAL COMPANY

MIDLAND, MICHIGAN 48640

AD-389 180
April 19, 1968

TO: ALL RECIPIENTS ON CPIA BASIC AND CATEGORIES I AND II LIST

Subject: OMISSION OF ESPIONAGE AND DOWNGRADING STAMP ON
REPORT AFRPL-TR-68-35 (CONFIDENTIAL)

Subject report must be properly stamped with the Espionage Notation, DoD Industrial Security Manual, page 19, paragraph (1), and the Downgrading Stamp, page 164, paragraph 5 - Group 4 Normal.

The title of the report is "Surveillance Studies of IMH-1 (U)," Annual Technical Summary Report (1 January to 31 December 1967), prepared under Contract Nr. FO4611-67-C-0067, by The Dow Chemical Company.

Very truly yours,

Robert S. Karpiuk

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