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MSAR 63-63

Report No. 3
Contract No. DA-36-039-SC-90699
DA Project No. 3M-36-21-004-02

Third Quarterly Progress Report
1 January 1963 to 1 April 1963

to

U. S. Army Electronics Research and Development Laboratory
Fort Monmouth, New Jersey

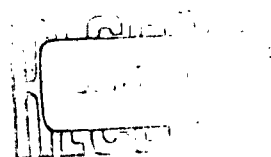
HYDROGEN GENERATION
FOR
HIGH ALTITUDE BALLOONS

31 May 1963

MSA Research Corporation

Subsidiary of Mine Safety Appliances Company

Callery, Pennsylvania



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HYDROGEN GENERATION FOR HIGH ALTITUDE BALLOONS

**Report No. 3
Contract No. DA 36-039-SC-90699
Signal Corps Technical Requirement No. SCL-5743A
(26 September 1961)
DA Project No. 3M-36-21-004-02**

MSAR XA-720318

**Third Quarterly Progress Report
1 January 1963 to 1 April 1963**

The objective of this contract is to study various hydrogen producing chemicals, evaluate their performance and the development of a design for a hydrogen generator suitable for inflation of high altitude balloons.

**W. J. Carter
R. A. Spencer
M. J. McGoff**

31 May 1963

**MSA RESEARCH CORPORATION
Callery, Pennsylvania**

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PURPOSE

The purpose of the work under Contract DA-36-039-SC-90699 is to study various hydrogen generating systems, to design a hydrogen generator for high altitude balloons and to produce a test supply of charges for the U. S. Army. The U. S. Army has need of an economical and portable supply of hydrogen to inflate weather balloons in the field. Heretofore they have used a Hydrogen Generator Set AN/TMQ-3 and Calcium Hydride Charges ML-304/TM and ML-305/TM to inflate 800 gram neoprene ML-518 balloons. Design specifications outlined in accordance with the Signal Corps Technical Requirement SCL-5743-A require:

- (1) A chemical charge of NaBH_4 or NaAlH_4 which will produce 45 SCF of H_2 in 30 minutes and have a minimum shelf life of 10 years.
- (2) An expendable and economical container compatible with the 32 gallons or a maximum of 50 gallons of water, if desired.
- (3) The generator should have a capacity of 1 to 6 charges, and be equal to or smaller than the present AN/TMQ-3 generator.
- (4) The weight of the generator should be equal to or less than the present model, operate at low pressure, and be of a simple, rugged but safe design.
- (5) Hydrogen generation must be started within 10 minutes after setup and a balloon must be inflated within 30 minutes.
- (6) The new design should be capable of being cleaned and repacked or available for reuse within 15 minutes and should also produce 99% hydrogen evolved at temperatures below 140°F .

An outline of the test program is as follows:

1. Make a survey of the literature to determine the state of the art of generating hydrogen and the most promising and economical method of producing H_2 for high altitude balloons.

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2. Conduct laboratory scale tests to determine the best method of accelerating the reaction; water requirements; and the best anti-foam agents, where required.
3. Make a preliminary design of a large scale generator to evaluate 45 cu ft charges to obtain engineering data.
4. Perform shelf life tests on the most promising chemical charges.
5. Make an evaluation of the large scale charges in the preliminary design generator to evaluate performance.
6. Make a final design of generator and prepare 50 NaBH_4 and 50 NaAlH_4 charges for submission to the U. S. Army Electronics Research and Development Laboratory.
7. Following approval of this model, fabricate two development model hydrogen generators and package 150 charges of NaBH_4 .
8. Prepare operation manuals and chemical specifications of the packaged charges.
9. Provide technical personnel to the U. S. Army Electronics Research and Development Laboratory during tests of the generator at Fort Monmouth.
10. Make monthly, quarterly and final reports in accordance with contract requirements as outlined.

ABSTRACT

During this report period, 1 January 1963 - 1 April 1963, ninety (90) small and large scale tests (up to 270 cu ft H₂ STP) were run. Data obtained from these tests enabled the design and packaging of fifty (50) NaBH₄, fifty (50) NaAlH₄ charges and the design and fabrication of a test model Hydrogen Generator (Model XI). The Generator and 100 charges were shipped to the U. S. Army Electronics Research and Development Laboratory.

MSAR personnel demonstrated the test model Hydrogen Generator on 29 March 1963. Two runs were conducted, each reacting three NaBH₄ charges and evolving the required 135 cu ft H₂.

Parameters investigated during this period included the forming pressure and physical packaging technique of large and small quantities of NaBH₄; and the mixing of various amounts and combinations of catalysts. These efforts were primarily directed toward obtaining a uniform hydrogen generation rate from NaBH₄. The best package developed was a 550 gm NaBH₄ charge containing 40 gm LiH, 11 gm CoCl₂ pressed in a 3 in. x 8 7/8 in. canister.

A uniform H₂ generation rate has been achieved with NaAlH₄.

Shelf life tests indicated NaBH₄ samples stored at 50°C gave lower yields than fresh control samples and required a longer time for H₂ evolution. The NaAlH₄ runs showed no significant differences from similar fresh controls.

REPORTS AND CONFERENCES

Within this report period, 1 January 1963 - 1 April 1963, the following reports were issued and conferences held.

Reports

1. MSAR 63-17 Second Quarterly Progress Report
"Hydrogen Generation for High Altitude Balloons"
W. J. Carter, R. A. Spencer and M. J. McGoff
26 February 1963
2. MSAR 63-27 Letter Report No. 5
"Hydrogen Generation for High Altitude Balloons"
W. J. Carter and M. J. McGoff
4 February 1963
3. MSAR 63-45 Letter Report No. 6
"Hydrogen Generation for High Altitude Balloons"
W. J. Carter and M. J. McGoff
4 March 1963
4. MSAR 63-50 Trip Report
U. S. Army Electronics Research and Development
Laboratory
M. J. McGoff
25 March 1963

Conferences

1. On 18 January 1963, at Callery, Pa., R. Welt of the U. S. Electronics Research and Development Laboratory conferred with MSAR personnel on progress of the project and problems anticipated in production of end items.
2. On 29 March 1963, at Fort Monmouth, New Jersey, a conference was held and the test model Hydrogen Generator was demonstrated.

EXPERIMENTAL

During this report period, 1 January 1963 - 1 April 1963, ninety (90) tests were made with NaBH_4 and NaAlH_4 charges. These were conducted in order to develop a charge canister, a generator and a packaging procedure that would evolve the desired amount of H_2 in the required length of time (30 minutes).

The test methods and areas of investigation were as follows:

1. Continued investigations of shelf life samples.
2. A series of runs using six (6) consecutive NaAlH_4 charges in the same water.
3. Sizing the canisters.
4. Tests to corroborate the interpolation of small to large scale test.
5. Sizing the generator.
6. Steps taken to improve the reaction rate of the NaBH_4 charge.
 - a. The use of wax or mineral oil combined with NaBH_4 in the lower portion of the charge.
 - b. A baffle plate with twenty 1/16 in. dia. holes and inserted near the bottom of the charge canister.
 - c. The application of different packaging pressure from the bottom to the top of the charge canister.
 - d. The variation of amounts and percent of CoCl_2 mixed in the different stages of the charge.
 - e. The addition of LiH to the top layer to speed up H_2 evolution and, give a more predictable reaction time.

- f. The use of more than one catalyst (AlSO_4 and B_2O_3 with CoCl_2) to start the reaction.

A charge generates 45 cu ft H_2 measured at standard conditions of 0°C and 760 mm Hg and will be indicated as such in this report unless otherwise stated.

1. Laboratory Tests With NaBH_4 and NaAlH_4

Small scale tests with NaBH_4 and NaAlH_4 are tabulated in Table 1. Large scale test results with NaBH_4 and NaAlH_4 are listed in Table 2. Large scale tests are defined as those that generated 45-270 cu ft (STP) H_2 . Major emphasis during this report period was placed on large scale testing to evolve a suitable hydrogen charge and generator design for the Army which could generate up to 270 cu ft H_2 in a 30 minute period.

Small Scale Tests With NaBH_4

The small scale tests were run to investigate techniques to control H_2 generation before advancing to full scale test charges. Data obtained from these runs which appeared promising was then used as information in packaging charges for large scale testing. Specifically, the concentration of the catalyst CoCl_2 and forming pressures were surveyed in the small scale tests. It was found that small scale test data was limited in the applicability to large scale tests because of the NaBH_4 behavior.

Small Scale Tests With NaAlH_4

Small scale test data with NaAlH_4 was found to be applicable to large scale tests. Extrapolation of small scale data was used to mock-up the large scale tests. Data for small scale tests with NaAlH_4 is given in Table 1.

The primary problem with NaAlH_4 has been the moderation of the reaction with water. Paraffin wax has been found to accomplish the moderation of the reaction but the wax must be melted in the mixing operation. To eliminate the melting of wax, mineral oil, a liquid at room temperature, was investigated as a possible moderating agent. A small scale run prepared with mineral oil did not effectively moderate the reaction as well as the paraffin wax.

A small scale test of six successive charges of NaAlH_4 was run and data plotted in Fig. 1. The test was run in the laboratory test apparatus, in which the water temperature, H_2 temperature, and H_2 flow rate were recorded. As indicated by the

TABLE 1 - SMALL SCALE EXPERIMENTS WITH NaBH_4 AND NaAlH_4

Run No.	Hydride Preparation Composition and Weight (g)	Cup Diameter (mm)	Forming Pressure (psi)	Water			H ₂ Yield		Elapsed Time (min)	Remarks
				Volume (liters)	Temp. Start	End	liters at STP	Theor. (%)		
154	NaBH_4 -118; CoCl_2 -10	70 ⁴	3,900	52 1/2	--	--	7.43	80.0	16	
155	NaAlH_4 -52; Wax-10	27 ²	11,350	1	20	81	2.29	92.0	12	
156	NaAlH_4 -25; Wax-5	45 ¹	3,200	2	31	58	1.31	89.0	3.7	Six (6) consecutive charges added to some water, Water cooled before second and sixth cups were added.
		45		-	33	55	1.31	89.0	3.7	
		45		-	54	71	1.31	89.0	6.0	
		45		-	65	79	1.31	89.0	3.5	
		45		-	76	86	1.36	91.6	4.5	
		45		-	32	56	1.31	89.0	3.8	
157	NaAlH_4 -25; Wax-5	27	3,400	1	20	64	1.33	90.0	6.5	
158		27	6,800	1	19	58	1.34	90.0	8.5	
159		27	11,350	1	20	66	1.30	89.0	7.0	
160	NaAlH_4 -48; Wax-9.5	27	3,400	2	19	52	2.09	92.5	10.0	
161	NaAlH_4 -54; Wax-10.8	27	3,400	4	20	39	2.36	93.0	9.8	
162		27	6,800	2	18	58	2.37	93.5	12.8	
163	NaAlH_4 -52; Wax-10.4	27	13,600	4	16	36	2.24	92.0	12.8	
164	NaAlH_4 -50; Wax-10.0	42.5 ³	2,140	2	22	63	2.68	91.5	4.1	
165	NaBH_4 -50; CoCl_2 -4	42.5	1,780	3	16	45	3.74	90.0	11.7	
166		42.5	1,780	2	20	54	3.85	92.0	8.0	
167	NaBH_4 -25; CoCl_2 -4	45	3,200	1	18	35	0.80	38.0	16.0	Used sea water, low yield
168		45		1	16	49	1.85	88.5	12.0	Control tap water
169		45		1	19	39	1.25	59.9	35.0	Used sea water
195	NaBH_4 -25; NaAlH_4 -2.5	45		1	--	--	--	--	--	No catalyst, only NaAlH_4 reacted
198-205	NaBH_4 -25; CoCl_2	45	—	1	--	--	--	--	--	Small scale samples pack- aged to duplicate large scale test. Result did not duplicate large scale test.
221	NaAlH_4 -25; Mineral Oil-3.8	45	3,200	1	22	63	1.08	72.0	1.8	

Note: 1 45 mm cup; surface area 15.90 sq cm
 2 27 mm cup; surface area 5.72 sq cm
 3 42.5 mm cup; surface area 14.25 sq cm
 4 70 mm cup; surface area 38.5 sq cm

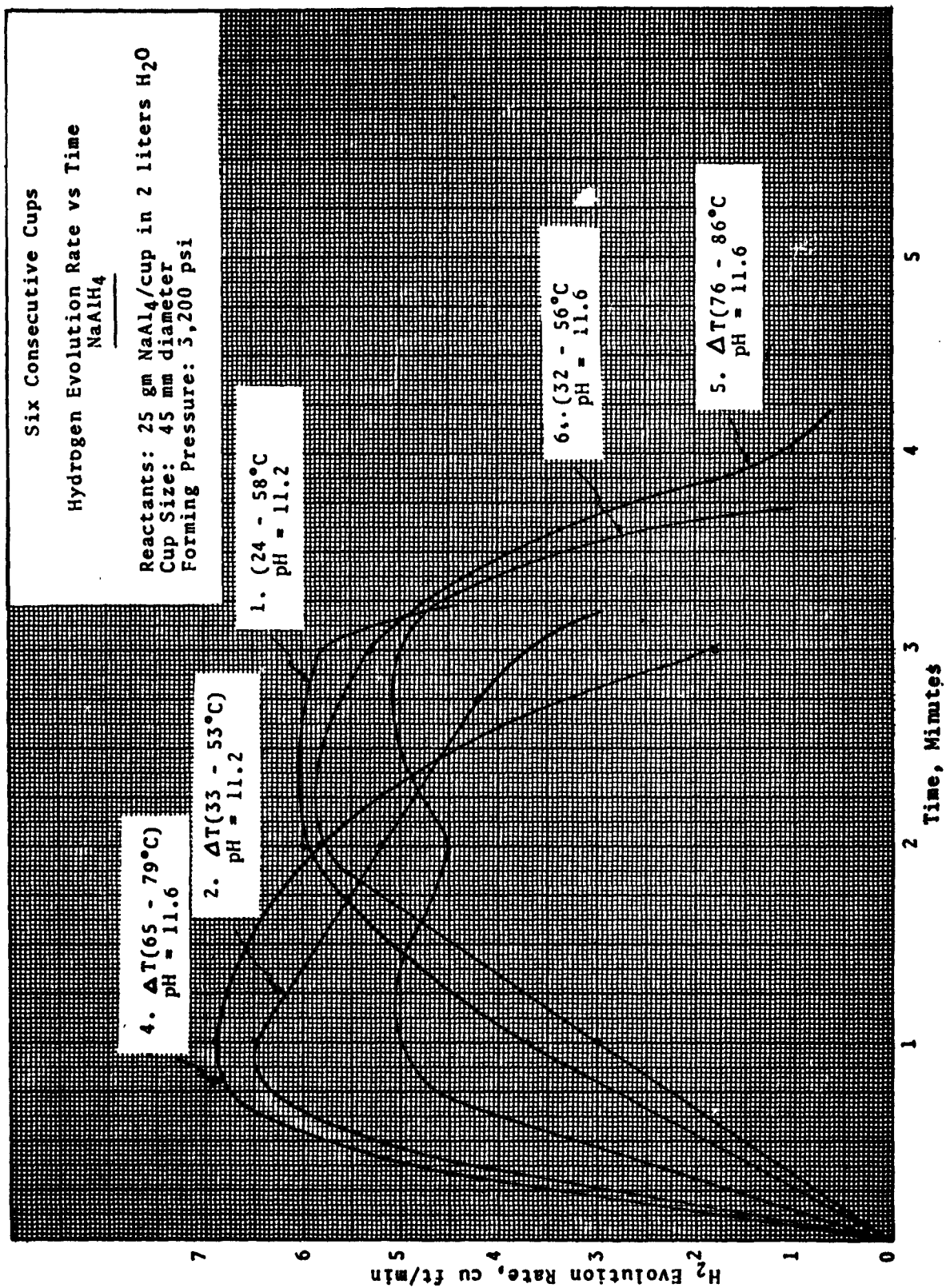
TABLE 2 - LARGE SCALE HYDROGEN GENERATION FROM NaBH_4 AND NaAlH_4

Run No.	Hydride Preparation				Water			H ₂ Yield		Elapsed Time (min)	Remarks
	Composition & Weight (gm)	Number of Canisters	Cup Diam. (in)	Forming Pressure psi	Volume (Gal)	Temp. - (°C)		cu ft at STP	Theor. (%)		
170	600 NaBH_4 52 CoCl_2	one	(18)	3,400	6	15	51	43.4	87.0	8	
171	600 52			3,400	6	16	53	46.2	92.5	8	
172	600 48			4,000	6	25	28	46.0	92.0	46	
173	600 144	three			18	21	40	-	-	11.5	(1)
174	600 48	one			6	22	-	-	-	21.0	(2)
175	24					20	55	-	-	25.9	(3)
176	16					21	56	-	-	-	
177	16					21	55	45.5	91.0	-	(4)
178	48					21	-	-	-	72	
179	14					13	48	46.3	92.5	58	
180	12					20	54	46.0	92.0		
181	200 16					21	55	47.0	94.0	37	
182	400 0										
182	200 16	Two									
183	400 0				12.7	20	52	95.0	91.3	46	(5)
183	200 16	three									
184	400 0	one			17.0	22	59	137.0	91.5	39.5	(5)
185	200 16										
186	400 0				6	21	54	45.6	91.2	48.0	(6)
185	250 20										
186	350 0				6	22	55	45.8	91.6	36.9	
186	200 16										
187	400 0				6	21	54	45.2	90.5	59	(7)
187	300 24										
188	300 0				6	21	55	45.1	90.2	27	
188	200 16										
189	200 0										
189	300 40 Wax				6	21	53	44.5	89.0	50	(8)
190	300 24 CoCl_2				6	21	38	25.7	45.0	16	(9)
190	300 45 Wax										
191	300 24 CoCl_2				4	21	67	45.9	91.6	33	
191	150 0										
192	150 22.5 Wax				5	20	60	45.4	90.6	19.3	
192	300 24 CoCl_2										
193	200 0										
193	100 15 Wax				6	21	54	44.4	87.2	32	
193	300 24 CoCl_2										
193	100 0										
194	200 Below a baffle plate				6	21	56	45.6	91.2	19.5	
194	100 16 CoCl_2			Top 2,500 "							
196	300 0			Center	6	21	55	44.6	89.4	35.5	
196	250 20 CoCl_2			Bottom 5,000							
196	150 0				5.3	20	58	43.1	86.5	39.2	
197	700 Below a baffle plate										
197	250 20 CoCl_2	Six		Top 2,000							
218	350 0			Bottom 5,000	32	19	58	272	90.0	22.6	(10)
218	250 20 CoCl_2	One									
219	150 0			Top 2,000							
219	200 20 mineral oil			Bottom 5,450	6	20	51	46.2	92.4	38	
219	250 20 CoCl_2			Top 2,500							
220	350 0			Bottom 5,450	6	10	47	45.6	91.2	44	
220	200 8 CoCl_2			Top 3,000							
222	400 0			Bottom 5,450	6	20	55	45.8	91.6	59	
222	250 20 CoCl_2	Six		Top 4,000							
223	350 0			Bottom 4,000	6	20	56	274	91.5	29.9	(11)
223	200 50 LiAlH_4	One		Top 4,000							
	350 11 CoCl_2			Bottom 5,450	6	21	59	48.4	96.0	30.5	

TABLE 2 - LARGE SCALE HYDROGEN GENERATION FROM NaBH_4 AND NaAlH_4 (cont)

224	825 NaAlH_4	140 Wax	↓	5,450	8	22	71	45.5	94.0	25	
225	200 NaBH_4	50 LiH		Top 4,000	6	21.5	60.5	45.7	95.0	26.3	
		11 CoCl_2									
	350	0		Bottom 5,450	6	21	60	45.7	95.0	26.5	
226	200	40 LiH	Six	Top 4,000							
		11 CoCl_2									
	350	0		Bottom 5,450	36	17	56	280	93.4	31	(12)
227	825 NaAlH_4	140 Wax	Three	5,450	27	21	63	135	90.0	28	
231	540 NaBH_4	50 LiH	310 gm NaBH_4 on bottom								
		8 Wax	heated to 100°C 2 hrs								
		10 CoCl_2	pressed to 15,000 psig.	6	21	60	46.6 ft ³			24	
			Balance of charge								
			pressed to 10,000 psig								
232	575 NaBH_4	25 LiH	25 gm LiH placed between								
		16 CoCl_2	175 gm NaBH_4 and 200 gm	6	21	58	48.0 ft ³			32	(13)
			NaBH_4 pressed to								
			14,000 psig. Top 200 gm								
233	550 NaBH_4	40 B_2O_3	NaBH_4 pressed to 6,000 psig								
		11 CoCl_2	350 gm NaBH_4 on bottom	6	21	53				35.7	(14)
			14,000 psig. Balance on								
234	550	40 LiH	top pressed to 7,000 psig	6	21	46				60	(15)
		11 CoCl_2	(See Run No. 228)								
235	550 NaBH_4	40 LiH	(See Run No. 228)	6	21	58				36	(16)
		11 CoCl_2									
236	550	40 LiH	(See Run No. 228)	6	21	44				60	(17)
		11 CoCl_2									

- (1) Reaction became violent after 15 min.
 (2) Repeat of Run 173. Foamed out of 10 gal. container inside a 30 gal. drum.
 (3) Meter failed during run.
 (4) Baffle plate on top of canister.
 (5) Canister in vertical position.
 (6) Canister in horizontal position.
 (7) Same H_2O as Run 185.
 (8) Wax slowed reaction down.
 (9) Stopped at wax.
 (10) Foam.
 (11) 1075 ml Foam and condensate.
 (12) 263 cc foam and condensate.
 (13) LiH reacted as expected at 26 min. H_2 evolution rate increased from 1 ft³/min to 3.8 ft³/min.
 (14) B_2O_3 boric anhydride gave smooth H_2 evolution. No acceleration of reaction near end.
 (15) Run outside in ~0°C atmosphere open drum to observe foam — ~5 in.
 (16) Repeat of above except drum was insulated. Foam — 5 in.
 (17) Used 240 ml gum turpentine, 80 ml aniline. Reduced foam to less than 1 in. Note: slow reaction time, also obnoxious vapor.
 (18) 4 3/4 in. diameter cup used in Runs 170 and 171, all other runs used a standard 3 in. diameter canister.

FIG. 1 - SIX CONSECUTIVE NaAlH₄ CHARGES

curves, NaAlH_4 charges can be run in succession using the same reaction water (pH 11.7) with very little difference in H_2 rate reaction. Some of these charges were run at higher initial starting temperatures. The higher temperatures ($24^\circ - 76^\circ\text{C}$) also had little effect on the H_2 reaction rate.

Large Scale Tests With NaBH_4

Table 2 lists data obtained on large scale tests with NaBH_4 .

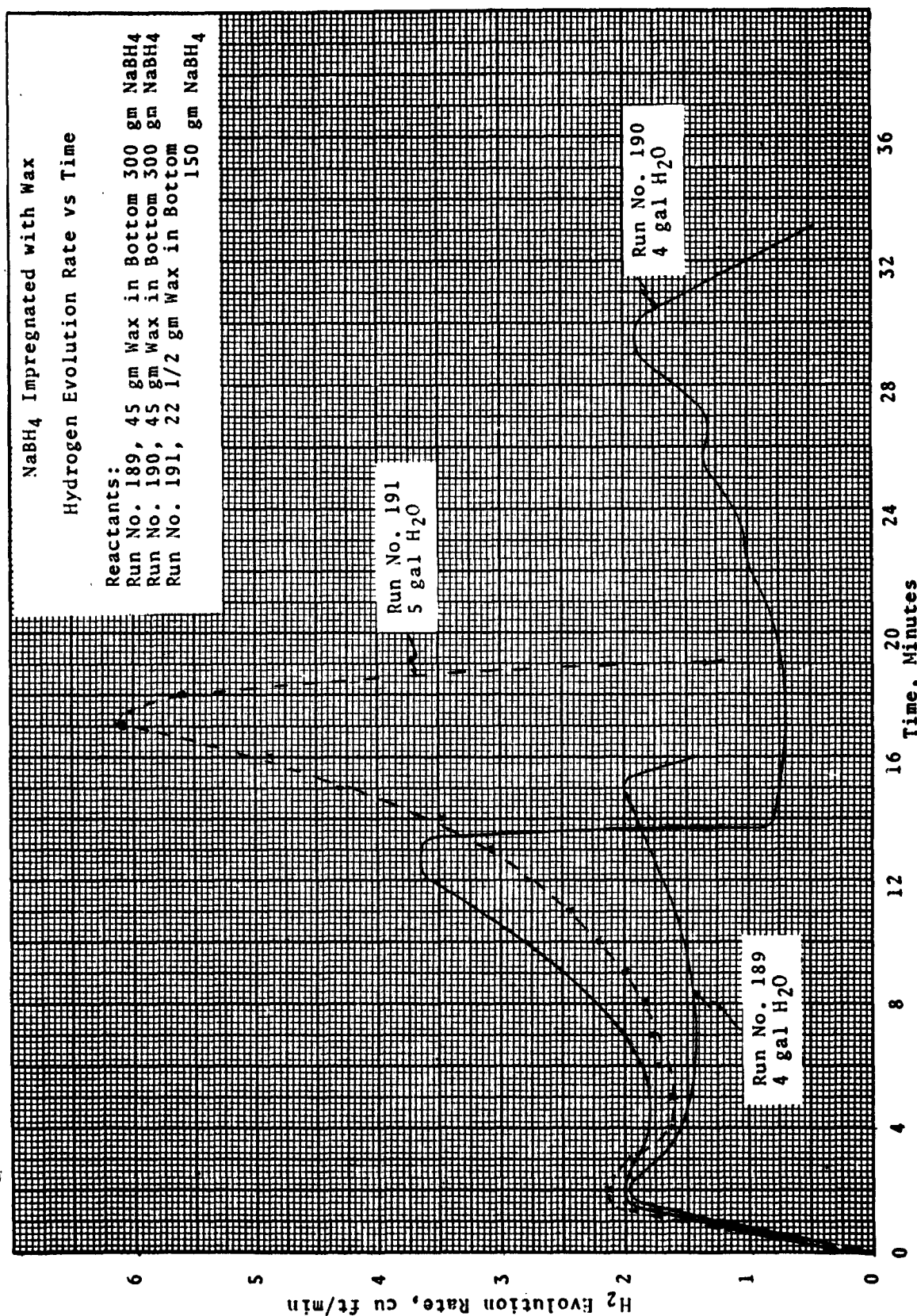
Various techniques were examined in an effort to promote uniform hydrogen generation with NaBH_4 . One of the major problems in generating H_2 with NaBH_4 is the formation of foam. Foam is a function of instantaneous H_2 generation rate. If generation rate is too rapid, excessive foaming results thus requiring a large free volume to contain the reaction. Therefore, a major solution to development of a suitable hydrogen generation using NaBH_4 lies in the techniques of packaging NaBH_4 to give a uniform H_2 evolution rate. The following techniques have been explored in large scale tests to develop a uniform H_2 generation rate with a minimum formation of foam.

These approaches have been:

- a. The use of wax and mineral oil
- b. Baffle plate
- c. Application of different packaging pressure
- d. Varying the amount of CoCl_2
- e. Seeding NaBH_4 charge with LiH
- f. Combination of other catalysts with CoCl_2

a. The Use of Wax and Mineral Oil

A number of charges were run in which mineral oil or wax was incorporated into the charge. This was suggested as a means of forming a partial barrier to slow down the accelerating reaction which takes place in the last half of the charge. As can be seen in Fig. 2, Run No. 189, the wax completely stopped the reaction with 50% of the H_2 evolved. A subsequent charge Run No. 190 was run in which less water was used in order to increase the temperature of the water so that the wax would melt. On

FIG. 2 - NaBH₄ IMPREGNATED WITH WAX

reacting this charge, the reaction rate was slowed up at the wax front (after 14 minutes) and then proceeded to accelerate again. Temperatures were 26°F higher when the wax barrier was encountered. A third charge was (Run No. 191) run in which the wax was pressed in the bottom quarter of the charge. On reaction of this charge, the wax had no visible effect on the H_2 evolution rate. Mineral oil was used in Run No. 218; 200 gm was mixed and pressed with the bottom 1/3 of the charge. On reaction of this charge, the decelerating effect was too long; i.e., after 30 minutes. In view of these results, no further work was done to incorporate the wax or mineral oil into the $NaBH_4$ charge.

b. Baffle Plate

A baffle plate with twenty 1/16 in. dia. holes was placed 1/3 of the way up from the bottom of the charge canister. It was anticipated that the decreased area would limit the amount of water present at the reacting chemical front. On reacting the charge, there was not visible difference between this and a standard canister, so effort along this line was discontinued.

c. Application of Different Packaging Pressures

A number of experiments were run in which the forming pressure of the charge was varied from 1,000 to 6,000 psi. Data from these runs indicated that the higher forming pressures tend to give longer reaction time. The data obtained looked promising enough that further work using higher pressures or hot pressing the material would be worthy of consideration.

d. Varying the Amount of Cobalt Chloride

A new approach was required to slow down the prohibitively fast reaction caused by a $NaBH_4$ charge packaged with 8% $CoCl_2$ and reacted in 6 gal of water. Fig. 3 shows two characteristic type curves: One curve is a $NaBH_4$ charge having 8% $CoCl_2$ (a total of 48 g) mixed with the charge and the other is a $NaBH_4$ charge having 8% $CoCl_2$ (a total of 16 g) mixed with the top 200 gm $NaBH_4$. Experiments were set up to study the effects of varying the per cent of $CoCl_2$ mixed with the top portion of the charge. The data obtained indicated that even for identical charges, time required for reaction was very unpredictable, with as much as 25 minutes variation in reaction time.

e. Seeding $NaBH_4$ Charge with LiH

In order to develop a more predictable charge, it was theorized that a highly active hydride material could be seeded with the $NaBH_4$ to disperse the $CoCl_2$ more quickly in the reaction water.

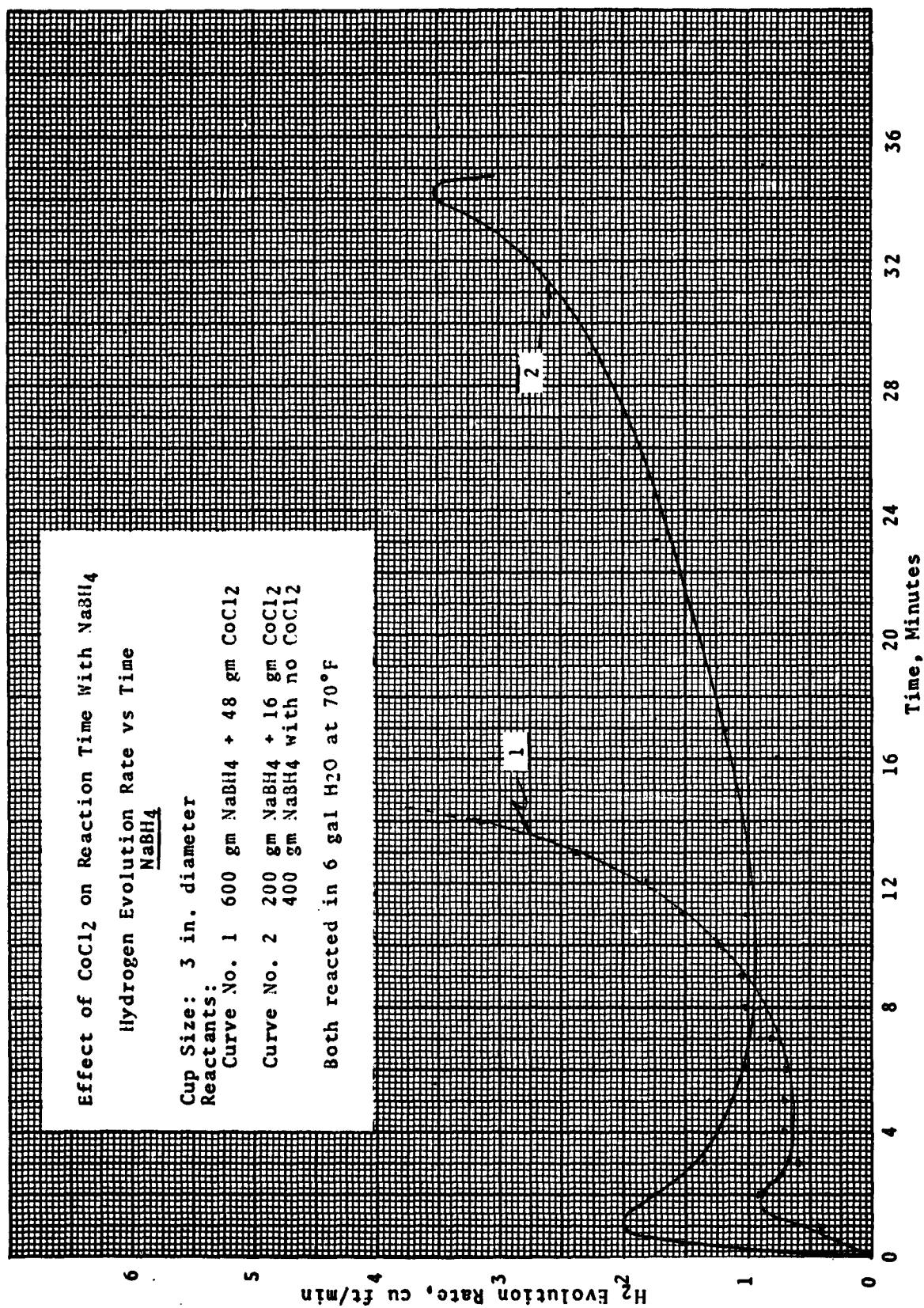


FIG. 3 - EFFECT OF CoCl_2 ON REACTION TIME WITH NaBH_4

The top portion of the NaBH_4 charge was seeded with LiH which was selected because of its high available yield of H_2 per unit weight. Fig. 4 shows two of these charges seeded with 50 gm of LiH and reacted in a sealed H_2 generator using 6 gal of water at temperatures of 70°F . The total time of reaction of this type charge has been predictable within ± 3 minutes.

f. Combination of Other Catalysts With CoCl_2

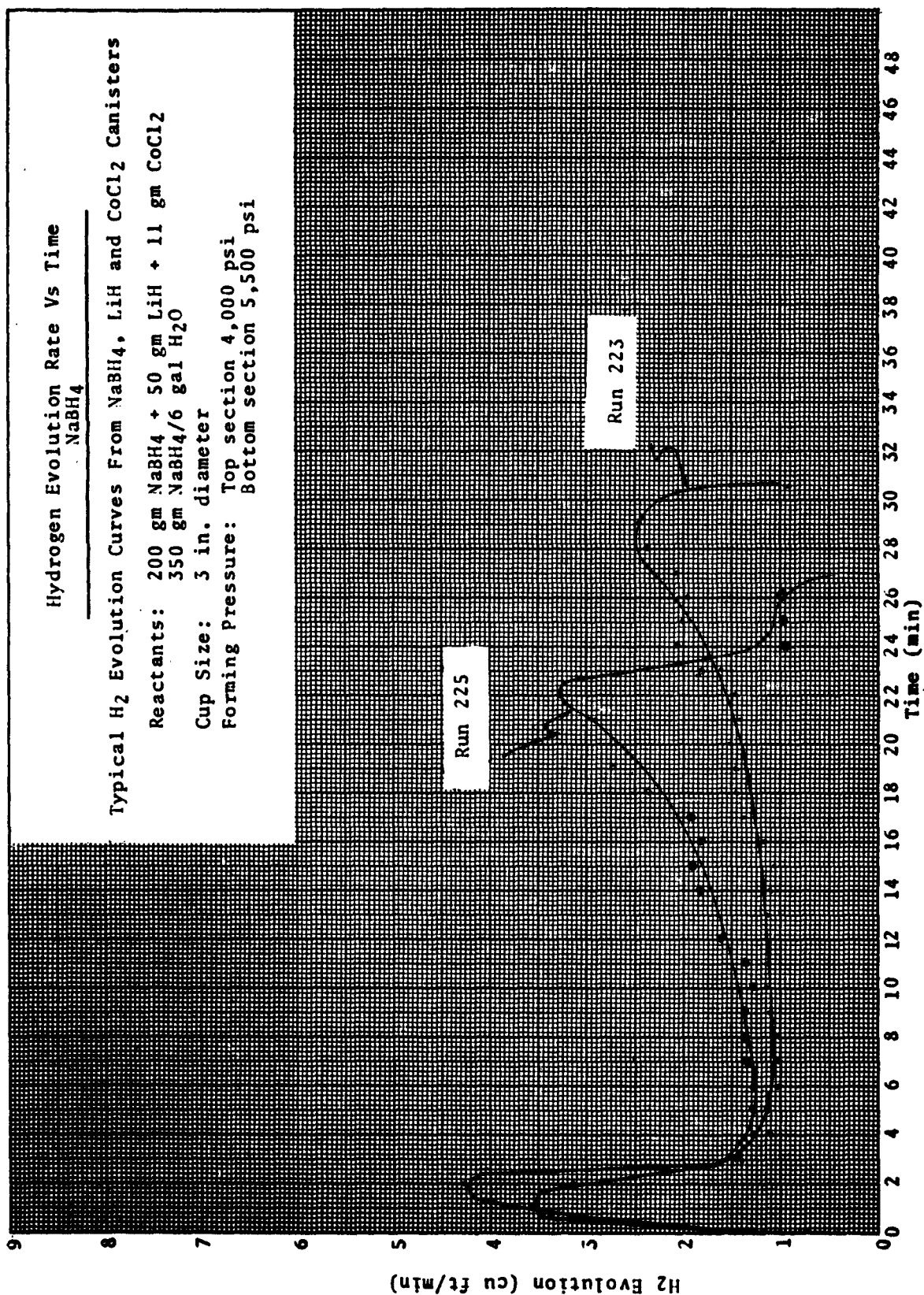
One test was conducted using boric oxide and CoCl_2 mixed and compressed with the NaBH_4 charge. As shown on Fig. 5, the behavior of the charge with boric oxide has a somewhat more uniform H_2 generation reaction without greatly altering the total reaction time or reducing the yield. In view of the results, it is felt that further work on mixing of different types of catalysts would provide more uniform H_2 evolution rates.

One test was run using 4% aluminum sulfate (8 gm) and 4% cobalt chloride (8 gm) mixed with the top 200 gm of NaBH_4 . On reacting the charge, the H_2 evolution rate curve was very similar to that of a straight CoCl_2 catalyst charge. This similar reaction rate was attributed to the low per cent of AlSO_4 initially packaged with the charge.

The behavior of the hydrolysis reaction of a solid NaBH_4 charge was examined in other runs. One experiment was set up to check whether a NaBH_4 charge packaged in a 3 in. dia. canister reacted as a moving front or is the NaBH_4 dissolved out into the water after a short period of time. The test canister was packaged first pressing 175 gm NaBH_4 in the bottom followed by a 25 gm section of LiH , then 200 gm NaBH_4 and on top 200 gm NaBH_4 and 16 gm CoCl_2 mixture. Thus, the section of LiH was located $2/3$ from the top of the charge. As shown on Fig. 6, a vertical line crossing the H_2 rate curve is the theoretical point at which 66% of the available H_2 has been evolved. The H_2 evolution rate curve indicates that the LiH is reacting at this point and that the NaBH_4 dissolves as a moving front through the charge.

One of the disadvantages in the use of NaBH_4 for the generation of hydrogen is the tendency of the reacting solution to form a varied volume of foam. This formation of foam is peculiar to the borohydride among the different materials tested for use in H_2 generation; however, it may be tied in with the fact that of these materials, only the borohydride dissolves in water prior to reacting.

In other experiments, a NaBH_4 charge was reacted in an open container to observe foam formation. The foam appeared shortly after the start of the reaction. Small bubbles completely covered the water surface and were dispersed after reaching a

FIG. 4 - TYPICAL H₂ GENERATION WITH NaBH₄ CHARGE CANISTER

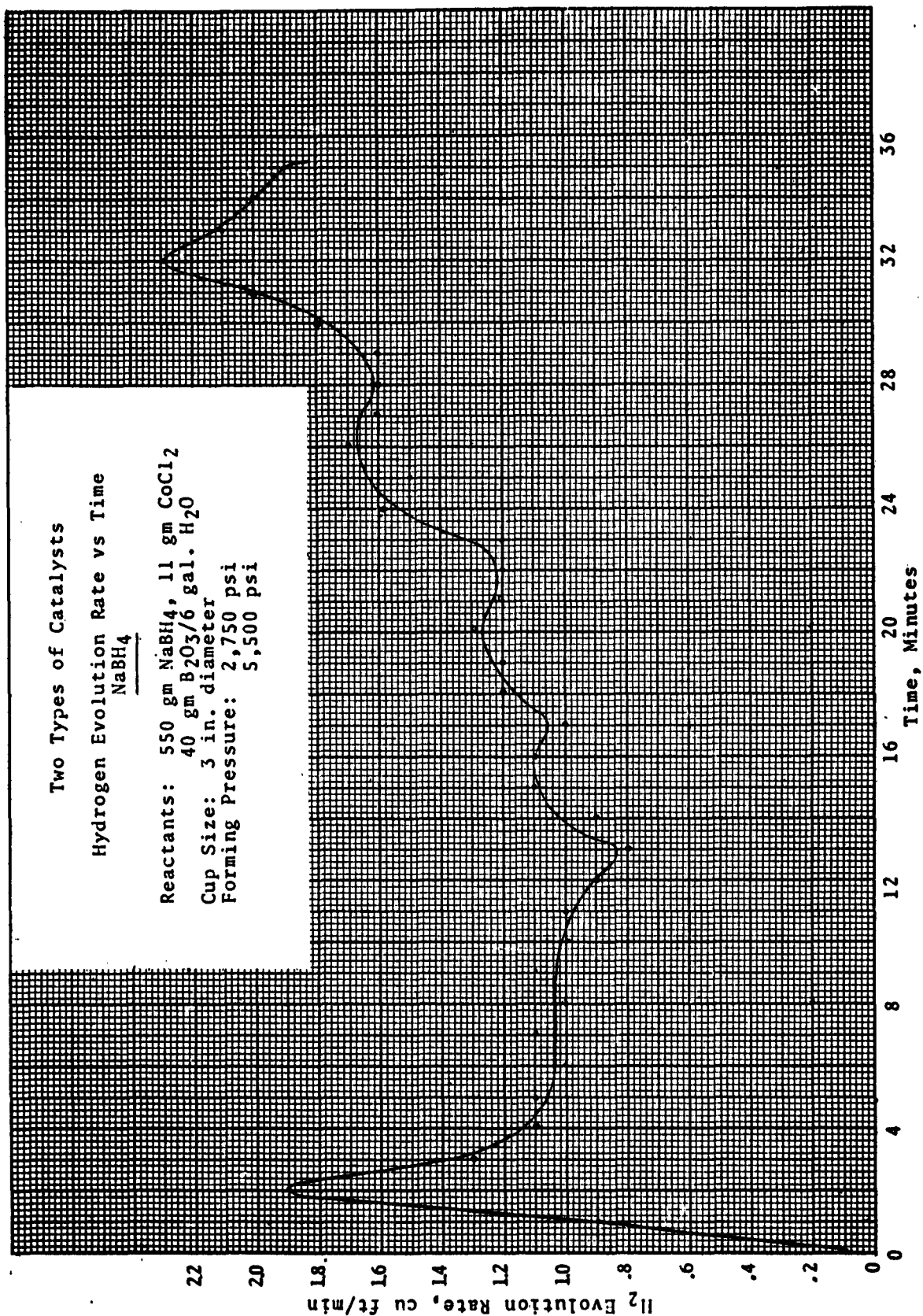


FIG. 5 - GENERATION OF H_2 WITH NaBH_4 BLENDED WITH
 CoCl_2 and B_2O_3 CATALYSTS

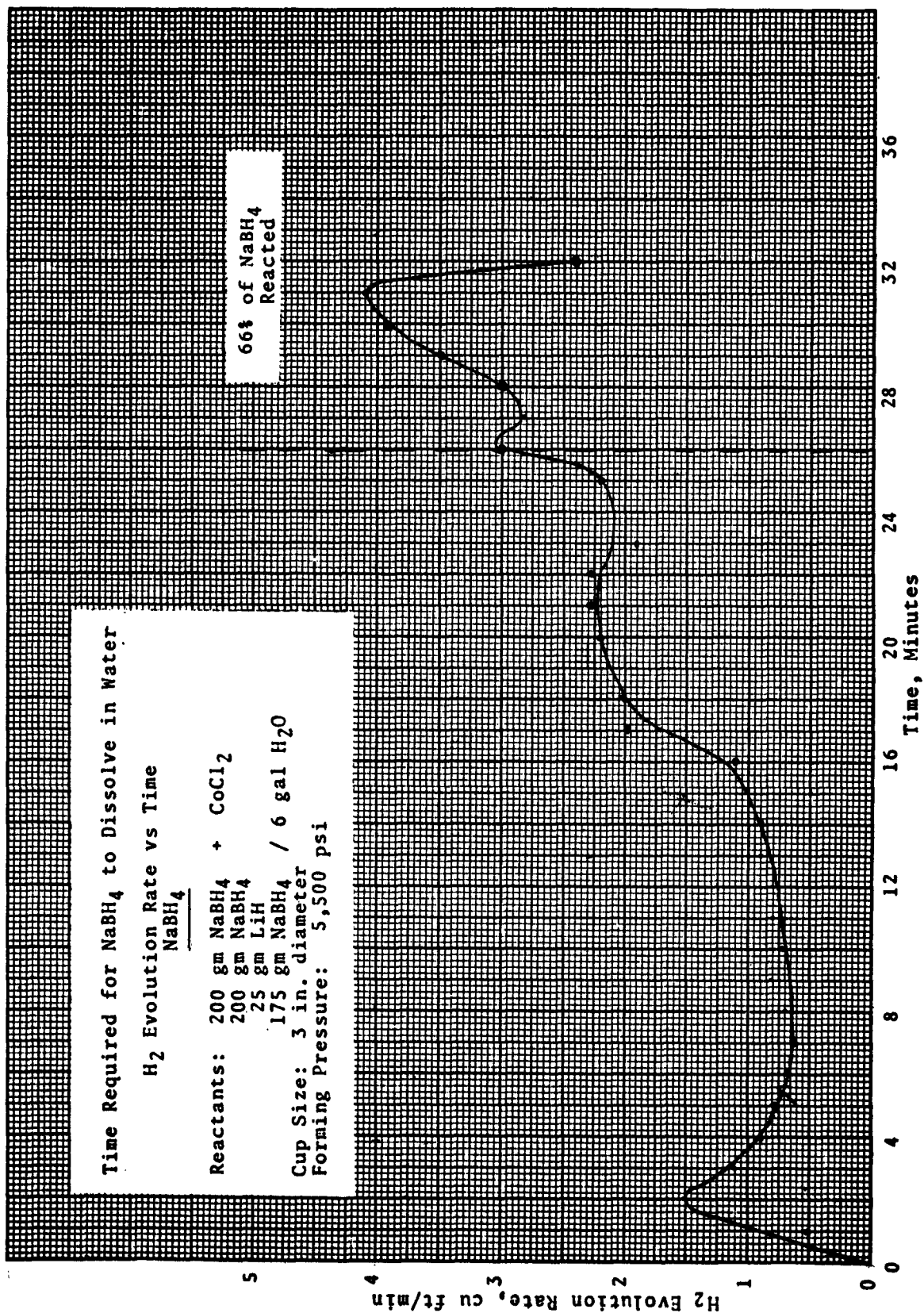


FIG. 6 - TIME REQUIRED FOR NaBH_4 TO DISSOLVE IN WATER

diameter of approximately 1/2 in. These bubbles look and acted similar to those found in soapsuds. In several of the open foam experiments, black particles (believed to be cobalt boride) about the size of a pin head were observed floating and being agitated on the foam. As the reaction progressed, the bubbles continually increased in diameter before dispersing.

Three runs were analyzed to determine the maximum amount of foam produced at any time during; one (1) charge, three (3) charges, and six (6) charges. This data is plotted in Fig. 7 and indicates that the foam has a tendency to increase as the amount of the charge material is increased. Fig. 8 shows a comparison between volume of foam and rate of H_2 evolved and that a corollary relationship exists.

No tests were run to maintain a constant water temperature in the generator because it was felt that a field generator held at a constant temperature would not be practical. Therefore, the mixtures were allowed to heat up as the reaction proceeded.

Initial water temperature has an effect on reaction time with $NaBH_4$. The observed effect is most prominent in the range below $20^\circ C$. This is illustrated in Fig. 9 where two identical charges were packaged, one was reacted in 6 gal of water with an initial temperature of $10^\circ C$ and the other in 6 gal of water with an initial temperature of $21^\circ C$. About 8 minutes longer reaction time was noted in the run with $10^\circ C$ water.

On 28 February 1963, fifty (50) $NaBH_4$ charges were supplied to the U. S. Army Electronics Research and Development Laboratory. The generation of six (6) $NaBH_4$ charges (the same formulation of those supplied) reacted with 36 gal of water is shown in Fig. 10. This generation curve is typical of the $NaBH_4$ charges whether it be one charge with six (6) gal of water or three (3) charges with eighteen (18) gal of water at initial water temperature of $20^\circ C$. There is an initial high generation rate of H_2 , a slow down, and then an accelerating effect as the reaction proceeds to completion.

Large Scale $NaAlH_4$ Tests

Small scale data on $NaAlH_4$ runs were successfully applied to mock-up large scale tests with $NaAlH_4$. The major problem encountered with $NaAlH_4$ has been in moderating the reaction with water. This has been solved by blending the $NaAlH_4$ with paraffin wax and controlling the surface exposed to reaction with water. Two large scale runs were performed with $NaAlH_4$ and these tests illustrated that a uniform H_2 generation in the specified 30 minutes is obtained. Fig. 11 shows the characteristic generation rate of $NaAlH_4$ for a single 45 cu ft H_2 charge. The

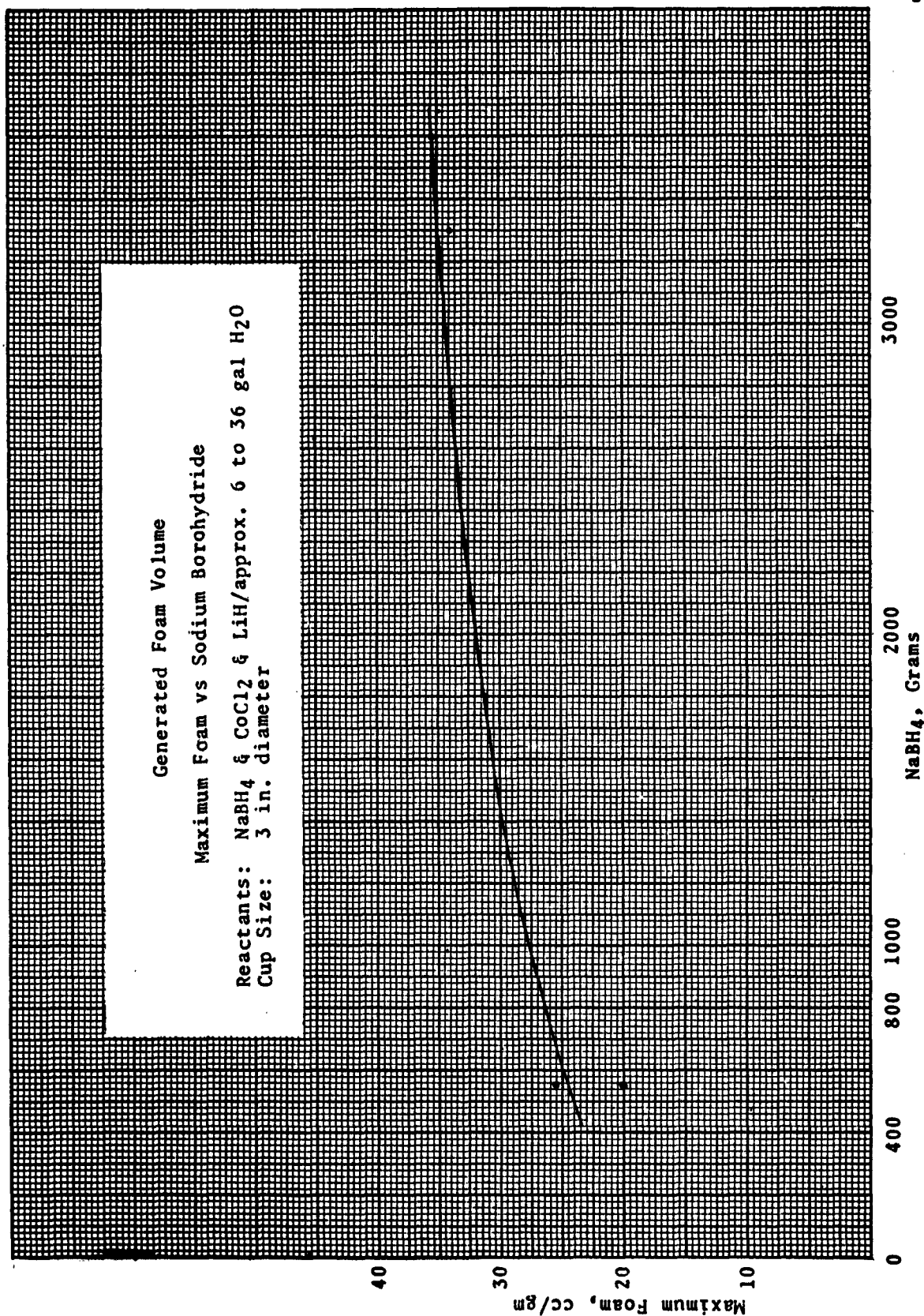


FIG. 7 - GENERATED FOAM VOLUME

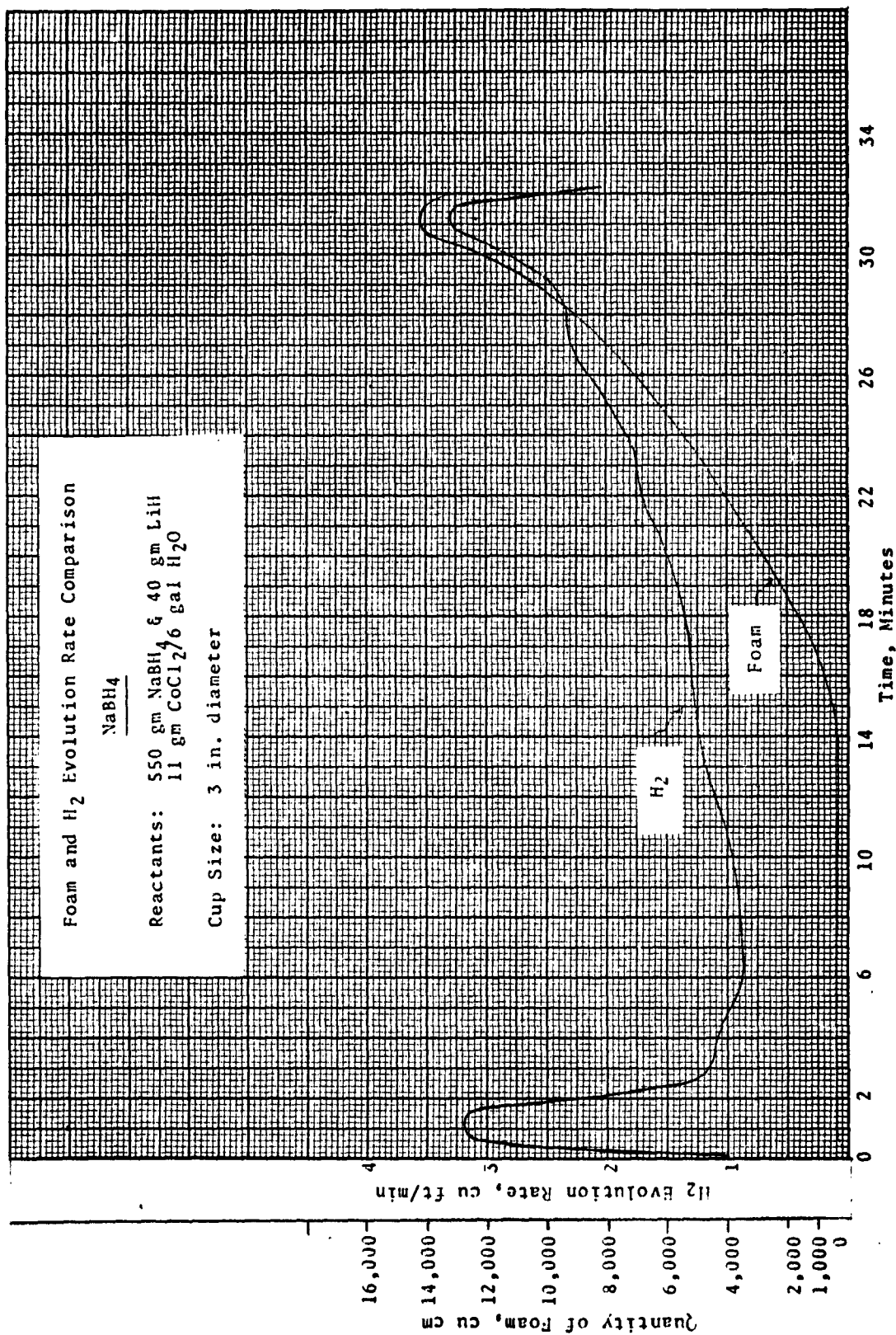


FIG. 8 - FOAM AND H₂ EVOLUTION RATE COMPARISON

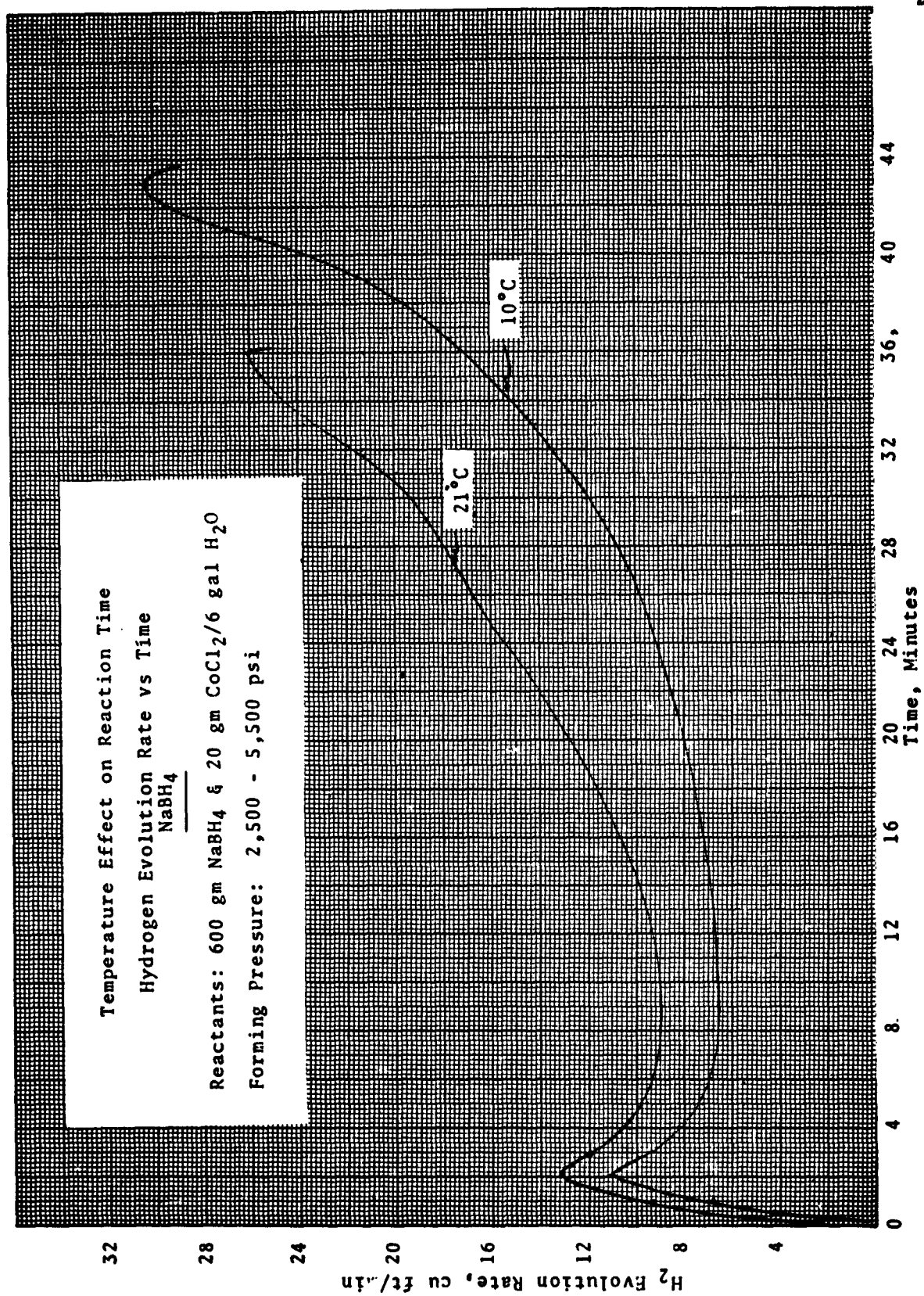


FIG. 9 - TEMPERATURE EFFECT ON REACTION TIME

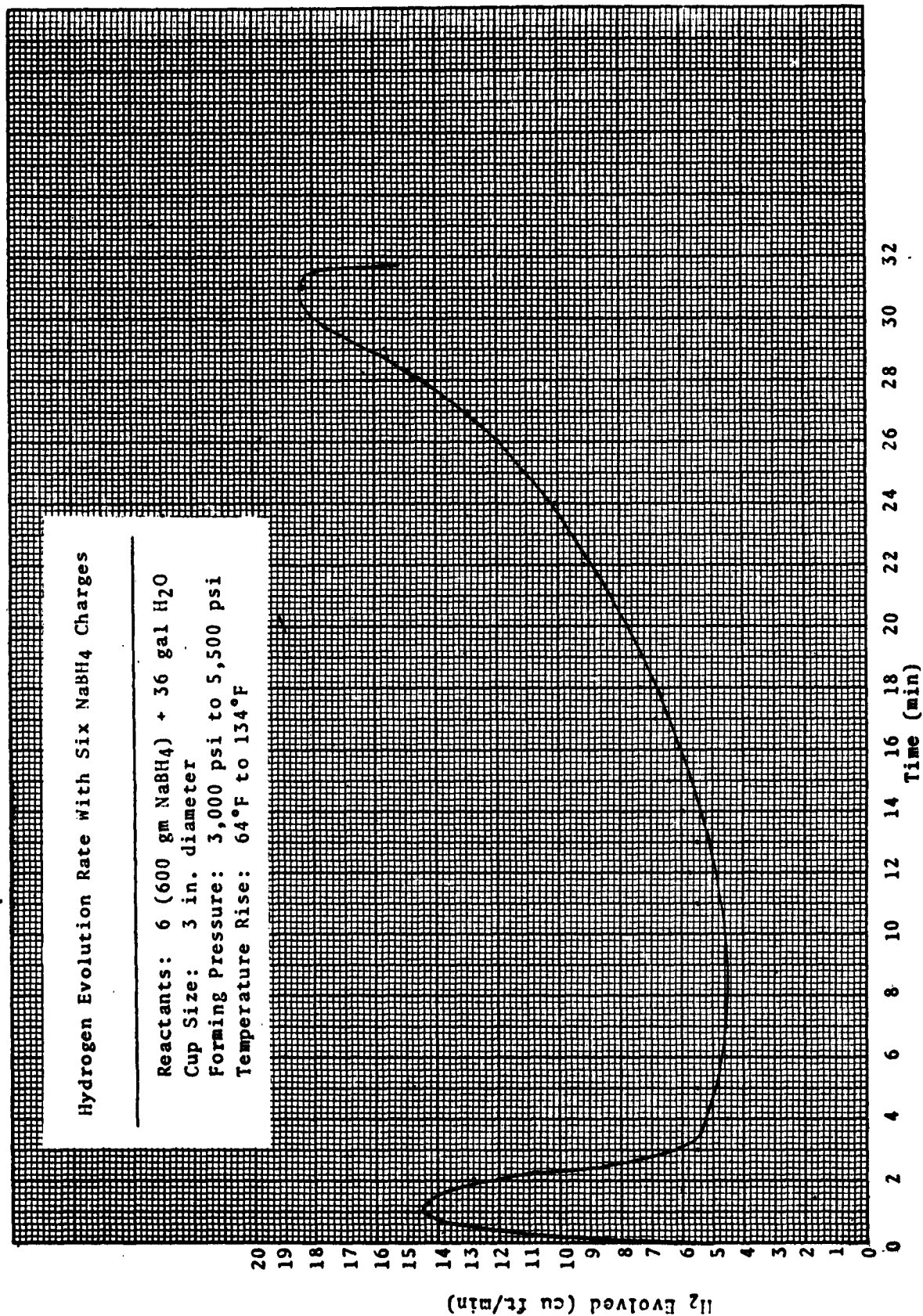
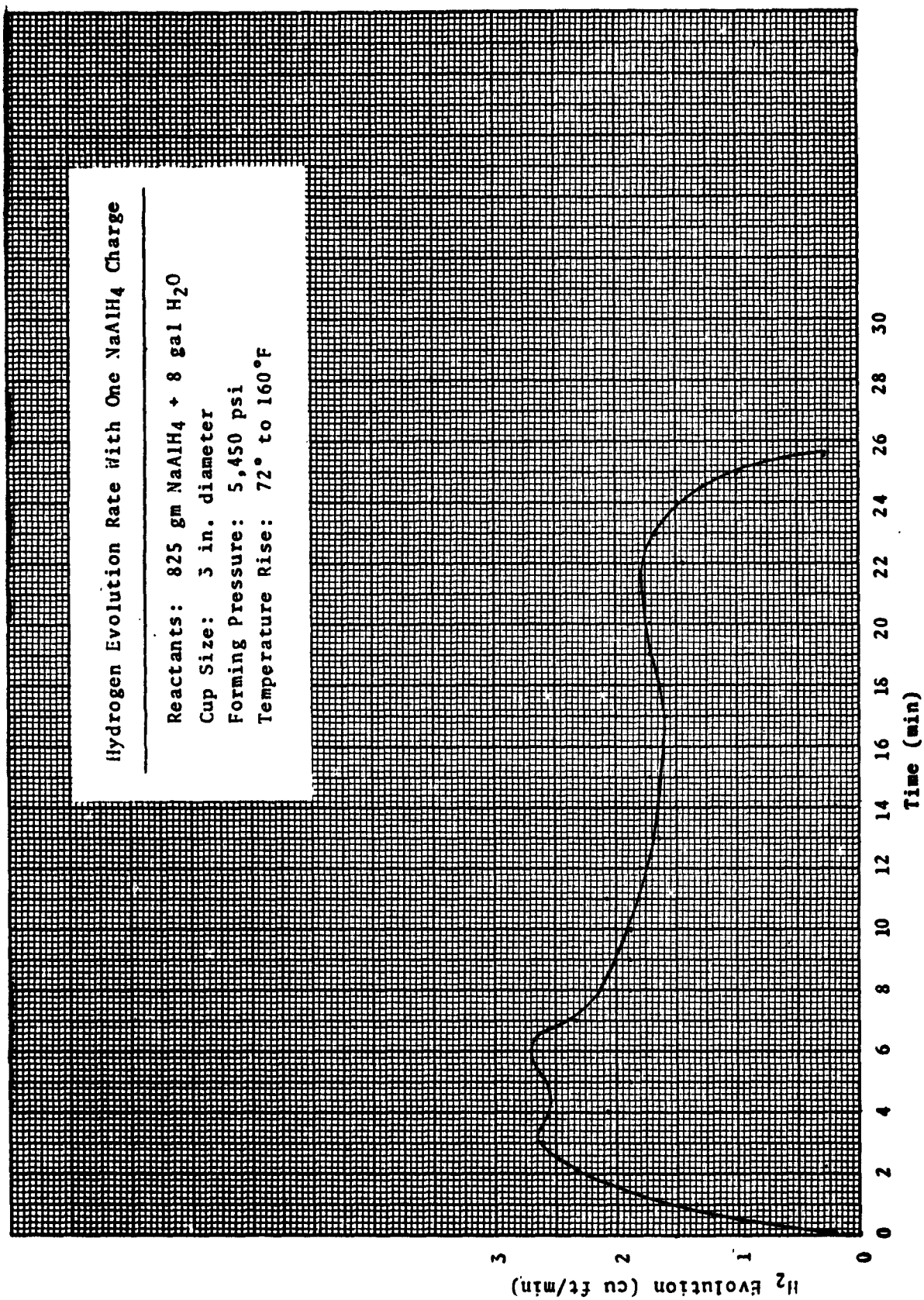


FIG. 10 - HYDROGEN EVOLUTION RATE WITH SIX NaBH_4 CHARGES

FIG. 11 - HYDROGEN EVOLUTION RATE WITH ONE NaAlH_4 CHARGE

second large scale NaAlH_4 run was made using three (3) 45 cu ft (STP) H_2 charges (or a total of 135 cu ft H_2 at STP) in the AN/TMQ-3 Generator. The NaAlH_4 charges supplied to U. S. Army Electronics Research and Development Laboratory on 28 February 1963 can be used in the experimental generator (Model XI) supplied on that date or in the AN/TMQ-3 Generator.

On 28 February 1963, six (6) NaAlH_4 charges were shipped to the USAELRDL. A total of fifty (50) charges were scheduled, but because of delay in receipt of NaAlH_4 the remaining forty-four (44) charges were not packaged until 29 March 1963.

2. Shelf Life Tests

The shelf life tests were continued on both the NaBH_4 and NaAlH_4 . The data obtained is tabulated in Table 3.

Samples of NaBH_4 and NaAlH_4 have been packaged in pellet and cup form for shelf life evaluation. Additional full scale NaBH_4 charges; i.e., 45 cu ft H_2 rated charges, have been packaged and are being evaluated for shelf life.

NaBH_4 - Samples of 50 gm NaBH_4 , stored for three (3) months at 20°C were evaluated and found to yield equivalent amounts of H_2 but required slightly longer times for reaction.

Cups containing 25 gm NaBH_4 at 20°C for three (3) months gave slightly lower yields (3%), in slightly longer periods. However, samples stored at 50°C in an oil bath gave significantly lower yields, 30% less and required three times as long for reaction. From these results it appears that storage at elevated temperature may adversely affect NaBH_4 .

However, when samples packaged in canisters in the manner supplied to the U. S. Electronics Research and Development Laboratory were examined for this effect, it was found that by reducing the amount of water used in the reaction, yield was unaffected and reaction time could be reduced. The charge containing 550 gm NaBH_4 , 40 gm LiH , and 11 gm CoCl_2 when stored in oil at 100°C for 46 hours gave an equivalent yield as the fresh control sample but required almost twice the reaction time. When an identical sample was stored at 100°C for 113 hours and reacted in 4 gal of water instead of 6 gal, an equivalent yield was obtained in about 60% the reaction time of the control; i.e., 19 1/2 minutes instead of 32 minutes.

From these results, it appears the effect of prolonged storage at elevated temperatures may require less water or some agitation of the packaged material to enable the NaBH_4 , which may be caked, to dissolve faster in the water.

TABLE 3 - SHELF LIFE TESTS WITH NaBH_4 AND NaAlH_4

Run No.	Hydride Preparation Composition and Weight (g)	Storage		Form	Forming Pressure (psig)	Water			Elapsed Time (min)	Yield		Remarks
		Time (months)	Temp. ($^{\circ}\text{C}$)			Volume (liters)	Temp. Start	Temp. End		liters at STP	Theor. (%)	
147	NaAlH_4 -73	0	--	Pellet	--	2.9	22	59	3.0	4.25	99.1	Control Run
148		0	--	Pellet	--	2.9	24	54	3.0	4.66	--	Control Run
149		3	20	Pellet	--	2.9	24	66	2.0	3.25	76.0	
150		3	20	Pellet	--	2.9	22	60	2.5	4.03	94.0	
212	NaAlH_4 -25	0	--	Cup	6000 ⁴	1.0	23	65	2.1	1.26	83.5	Control Run
213		0	--	Cup	6000	1.0	23	64	2.5	1.32	89.5	Control Run
214		3	20	Cup	6000	1.0	20	60	3.1	1.32	89.5	
215		3	20	Cup	6000	1.0	23	62	3.5	1.37	92.5	
216		3	50	Cup	6000	1.0	15	63	4.2	1.32	89.5	
217		3	50	Cup	6000	1.0	23	63	3.1	1.30	88.0	
151	NaBH_4 -50; CoCl_2 -4	3	20	Pellet	10,000	1.9	25	59	7.7	3.59	85.0	
152		3	20	Pellet	10,000	1.9	22	58	9.5	3.71	88.0	
153		0	--	Pellet	10,000	1.9	20	54	7.3	3.50	83.0	Control Run
206	NaBH_4 -50; CoCl_2 -2.2	0	--	Cup	6000	1.0	21	56	6.9	1.87	89.0	Control Run
207		0	--	Cup	6000	1.0	21	55	9.0	1.85	89.0	Control Run
208		3	20	Cup	6000	1.0	21	55	9.0	1.81	86.0	
209		3	20	Cup	6000	1.0	22	55	8.5	1.78	85.2	
210		3	50	Cup	6000	1.0	21	53	24.5	1.15	55.0	
211		3	50	Cup	6000	1.0	21	51	22.2	1.23	59.0	
228 ¹	NaBH_4 -550; CoCl_2 -11; LiH -40	46 hr	100	Cup	6000/14,000	22.7 ²	20	53	55.0	46.0 ³	92.0	Accelerated at 100 $^{\circ}\text{C}$ Storage
229		--	--	Cup	6000/14,000	22.7	20	57	32.0	46.0 ³	92.0	Control Run
230		113 hr	100	Cup	6000/14,000	22.7	20	70	19.5	46.0 ³	92.0	

Note: 1 Charges in Runs 228-230 typical of those furnished to USERDL on 28 February 1963.
 2 Note 22.7 liters equals 6 gal.
 3 Cubic feet.
 4 A gage pressure of 6,000 psig gives a forming pressure of 1,900 psi on the material as its pressed in a 45 mm diameter cup.
 A gage pressure of 14,000 psig gives a forming pressure of 4,500 psi on the material as its pressed in a 45 mm diameter cup.

This difficulty with the NaBH_4 charge as packaged in a can requires further investigation.

NaAlH_4 - Three month shelf samples of NaAlH_4 in pellets and pressed cups stored at 20°C and in an oil bath at 50°C continue to show no significant difference from fresh control samples. This would indicate that storing of NaAlH_4 at elevated temperatures has no significant effect on its shelf life. Additional samples of NaAlH_4 packaged in sealed cans will be evaluated for shelf life when sufficient material is on hand.

3. Apparatus Design

The purpose of all experimentation has been to evolve a system which utilizes NaBH_4 and NaAlH_4 to generate H_2 for inflating high altitude balloons. Both NaBH_4 and NaAlH_4 will generate more H_2 per unit weight than CaH_2 which is currently being used. However, the problems associated with NaBH_4 and NaAlH_4 and the relative material cost must be considered in the selection of a suitable material for future use by the Army.

A method of packaging NaBH_4 and NaAlH_4 has resulted from this work. A description of these designs follows. It has not been possible to reduce the size of the generator from the size of the AN/TMO-3. This is because of the problem of foam associated with the use of NaBH_4 . Foaming has been materially reduced, through packaging techniques, and review of the literature indicates that foam has been reduced over that encountered by previous experimenters.*

In the development of the canisters, direction of design pertained to assembly of standard types of packaging even though the characteristics of H_2 generation with NaBH_4 is adversely affected by conditions such as quantity, temperature and types of water (fresh as compared to saline). The programming of the H_2 generation with NaBH_4 and NaAlH_4 has also been directed to make both packages react within the specified 30 minute time period. Both materials were packaged in 3 in. dia. canisters, this being considered as the optimum size.

Canister Design

The dimensions of the canister were arrived at by projecting the small scale test into a large scale basis. A curve was constructed which indicated that a 5 in. dia. canister

* The Generation of Hydrogen from Sodium Borohydride
Ethyl Corporation, 30 December 1944.

would be required for the reaction of NaBH_4 to evolve 45 cu ft H_2 at STP from a single charge in a time of 25 minutes.

A 4 3/4 in. dia. mold was used to package two (2) test canisters (4 3/4 in. dia. x 3 1/4 in. high). The water used to react the NaBH_4 was reduced from twelve (12) to six (6) gal per each 45 cu ft charge. The water was reduced to stay within the specifications of the contract (less than 50 gal of water for 270 cu ft H_2). The reaction time for both of these test canisters was approximately 8 minute and the H_2 was evolved at an excessive, accelerating rate. The data was taken from these runs and plotted on log log paper as surface area (sq cm) vs grams reacted per minute. The new canister size was predicted from this curve at 3.14 in. dia., but rounded off to 3 in. which was considered the minimum size dia. that could be used for both the NaBH_4 and NaAlH_4 canister. This was due to the restriction of the adapter used to connect the NaAlH_4 charges in the AN/TMQ-3 Generator.

The results from the testing of the 3 in. dia. cups with NaBH_4 were not satisfactory, because of the continued excessive accelerating affect from the CoCl_2 as the reaction proceeded through the length of the charge. At this point, the amount of CoCl_2 was reduced (discussed previously) to lessen the accelerating affect.

The canisters to hold both NaBH_4 and NaAlH_4 charges were specified and fabricated as shown on Fig. 12 and 13, respectively.

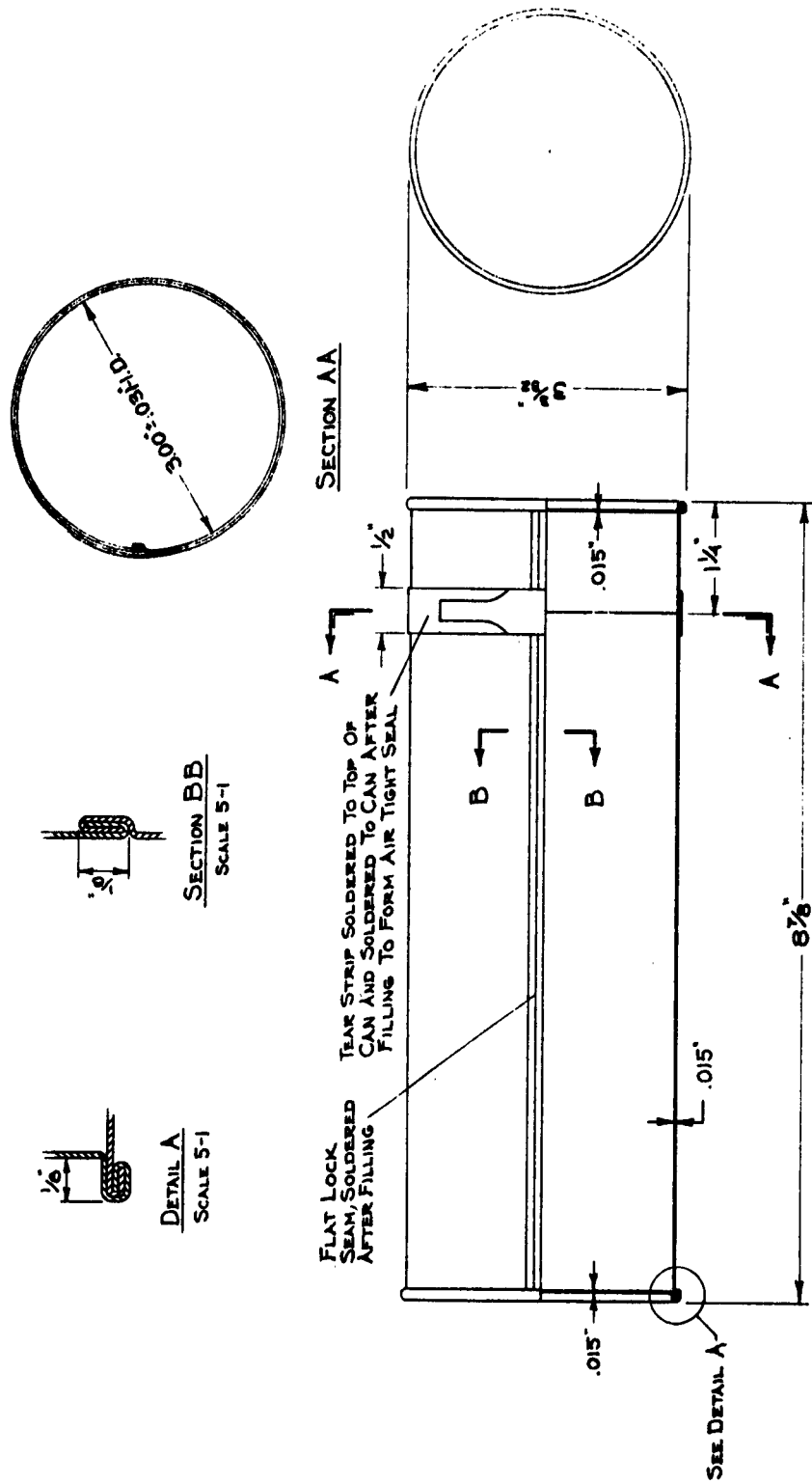
The NaBH_4 charge canister contains 550 gm NaBH_4 , 40 gm LiH and 11 gm CoCl_2 and is pressed in two stages, top at 2,750 psi and the bottom at 5,500 psi. This charge is operable in only the MSAR Model XI Hydrogen Generator (See Fig. 14).

The NaAlH_4 charge canister contains 825 gm NaAlH_4 , 140 gm wax and pressed at 5,500 psi and is operable in both the AN/TMQ-3 and the MSAR Model XI Hydrogen Generator.

Generator Design

The preliminary design and fabrication of the generator was completed on 28 February 1963 and shipped to the U. S. Army Electronics Research and Development Laboratory, Fort Monmouth, New Jersey.

The specifications given in the Army Technical Requirements SCL-5743-A are listed in a section of this report, (See Purpose). The design and shape of the generator also took into consideration: compactness, ease of operation, temperature of evolved hydrogen, ease of assembly and disassembly and the maximum amount of evolved hydrogen.



PD-0015

FIG. 12 - NaBH_4 CANISTER

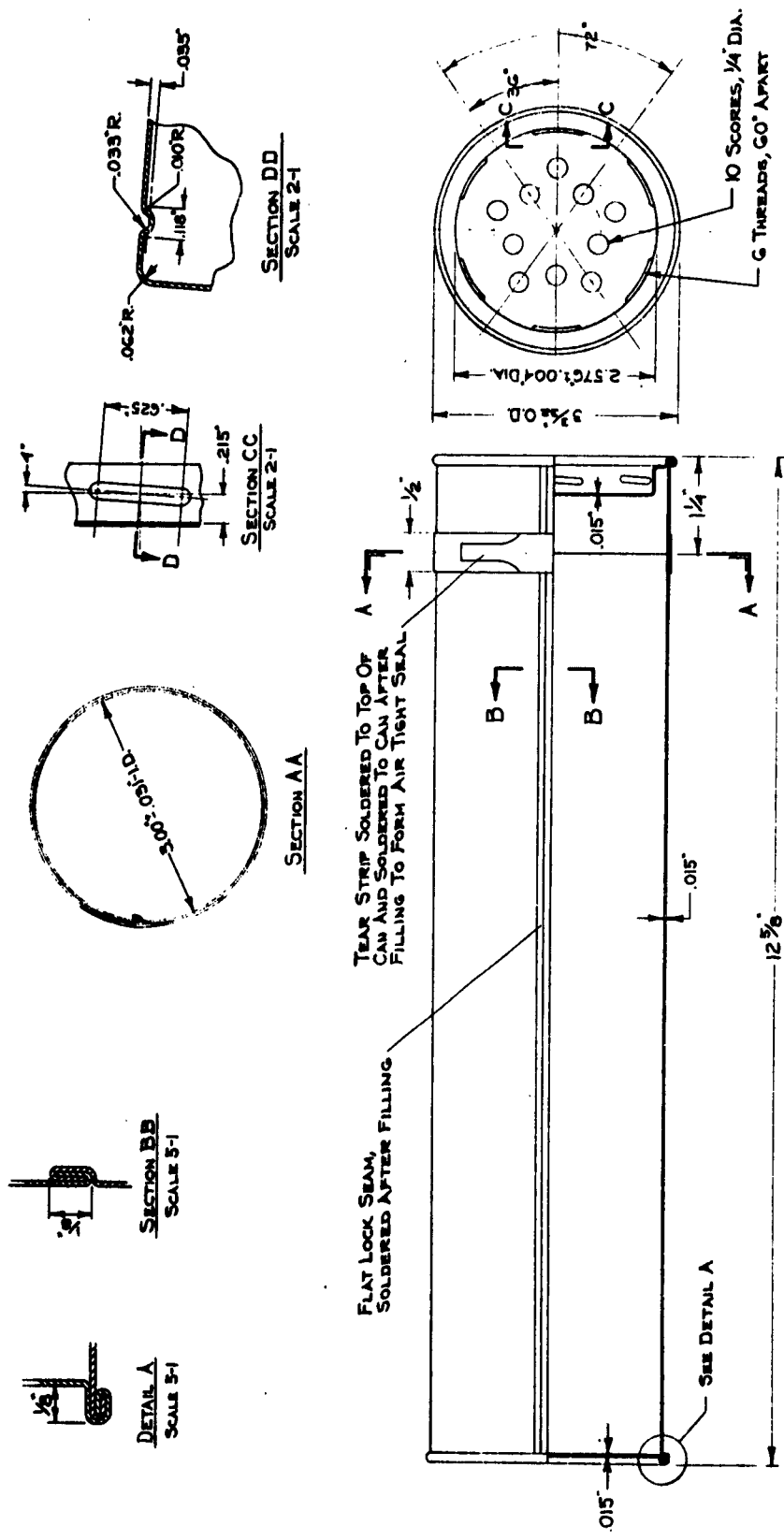


FIG. 13 - NaAlH₄ CANISTER

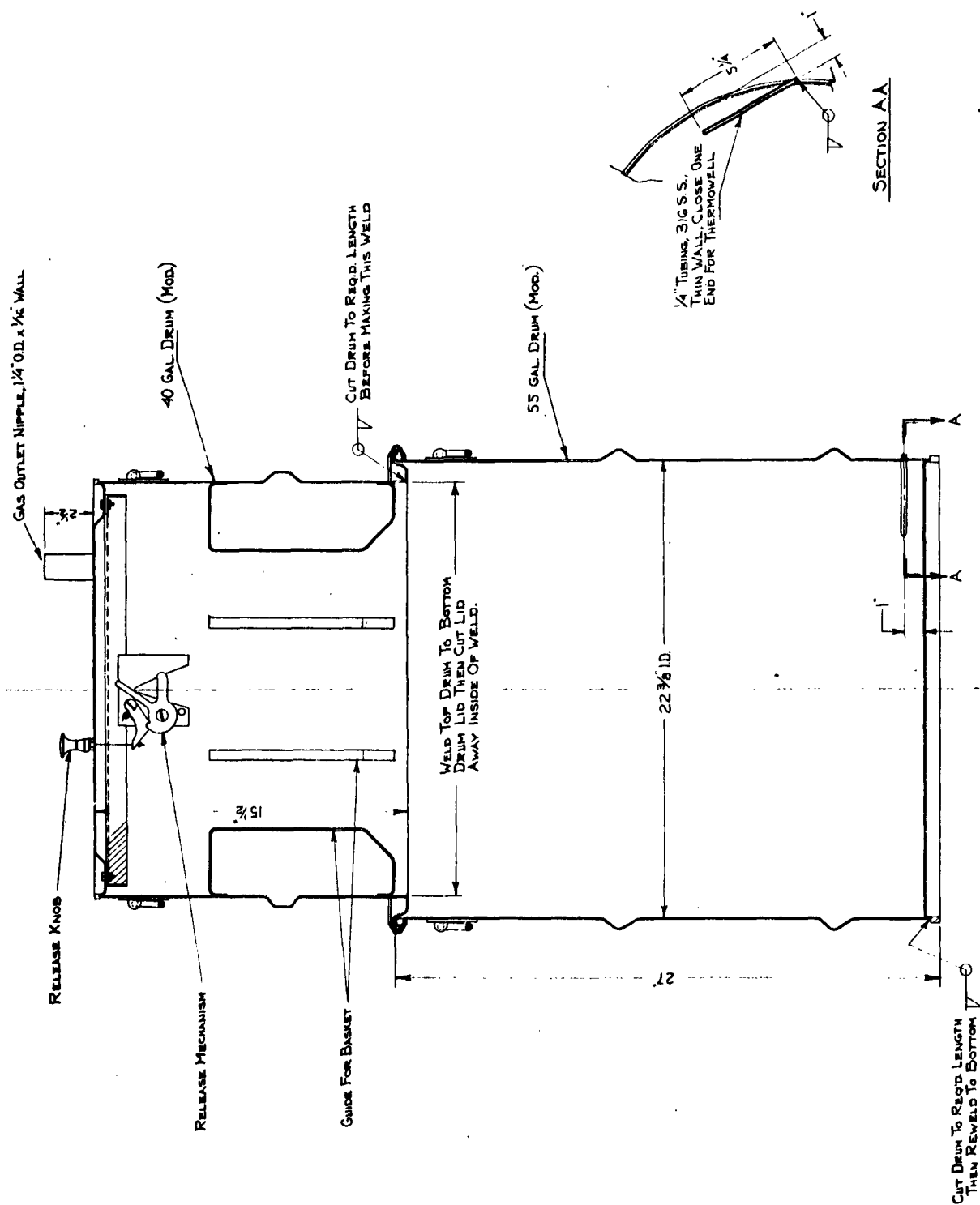


FIG. 14 - MODEL XI HYDROGEN GENERATOR

PD-0032

Six gal. of water for each charge was used to maintain the water temperature below 140°F and a H₂ evolution time of 30 minutes for NaBH₄. The volume required for foam containment was determined and estimated to average about 35 cc/gm NaBH₄. Thus, for a six (6) charge run, it would require a void space of approximately 4.4 cu ft and a total volume of approximately 9.2 cu ft.

From this data, an apparatus was designed which would have a volume of 9.2 cu ft while in operation and a volume of 6.2 cu ft in the stored position. The apparatus is shown in Fig. 14 and shows the generator in an operating position. The generator as seen consists of a 20 3/4 in. ID x 15 in. high unit, which contains a stainless steel basket, Fig. 15, used to hold one (1) or up to six (6) charge canisters. The basket is held in place by brackets and is dropped into the water by a quick release mechanism which is operated by an external knob located on top of the generator. This unit is designed so that it may be placed over any size of open head drum having a 22 3/8 in. ID rolled steel edge. The generator is sealed by placing the manual lever action u-type clamp over the two drum edges and closing. A water level scale is painted on the inside of the water drum at increments of 6 gal, up to 36 gal.

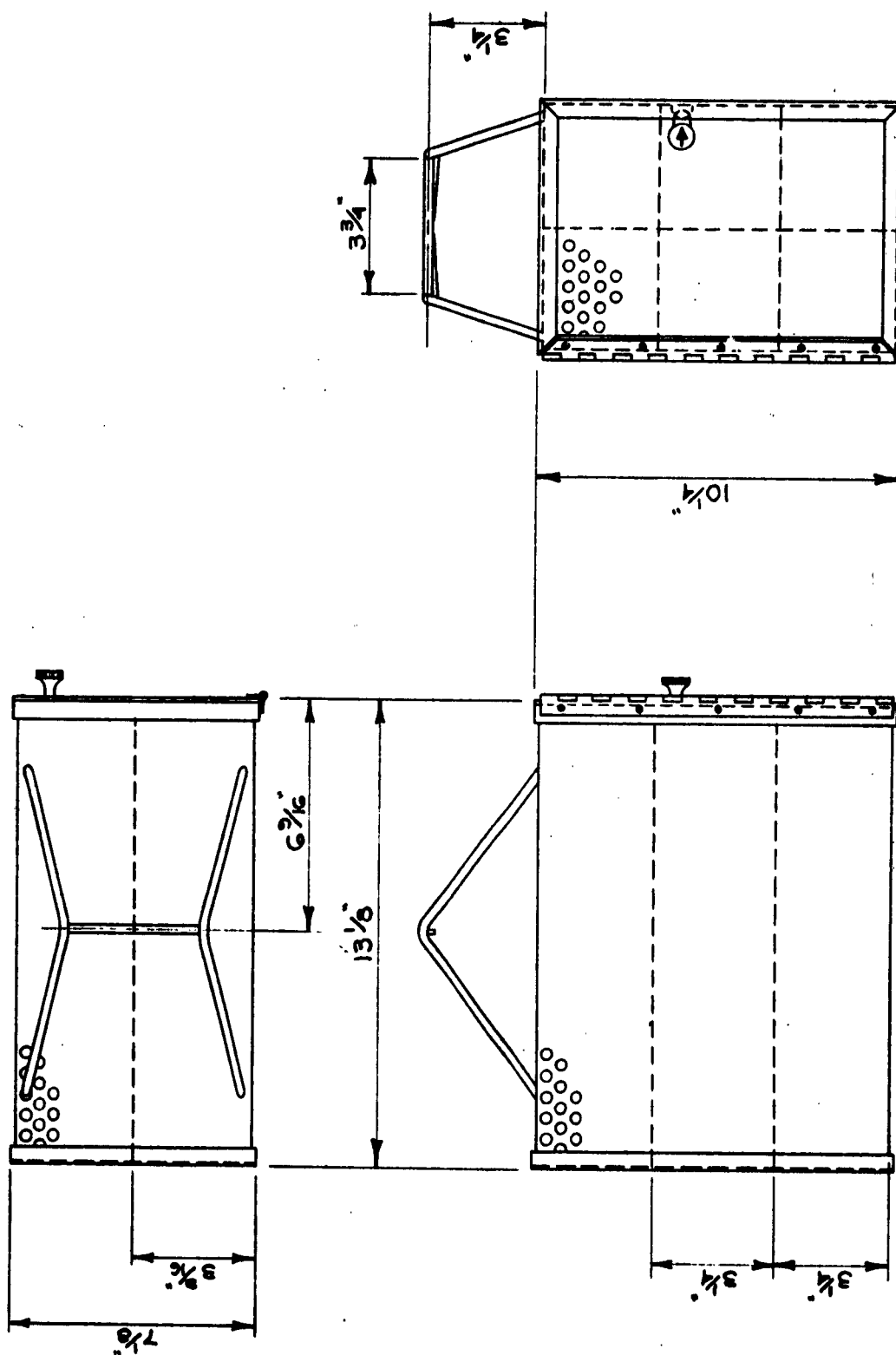


FIG. 15 - CHARGE BASKET

CONCLUSIONS

During this quarter the preliminary design and fabrication of NaBH_4 and NaAlH_4 canisters and a model hydrogen generator were completed. Fifty (50) NaBH_4 and fifty (50) NaAlH_4 charges, each containing sufficient material to generate 45 cu ft (STP) H_2 were completed and shipped to the U. S. Army Electronics Research and Development Laboratory. MSAR personnel demonstrated two NaBH_4 runs using three (3) charges (or 135 cu ft of H_2) in each run to U. S. Electronics Research and Development Laboratory personnel on 20 March 1963.

The major problems encountered in obtaining uniform H_2 generation has been presented by NaBH_4 , with NaAlH_4 being much more predictable. A summation of the work conducted thus far is as follows:

1. Attempts to scale up small laboratory size NaBH_4 experiments failed to produce the desired results. This was particularly true of surface area affects. A 4 3/4 in. cup reacted completely in 8 minutes rather than the predicted 30 minutes.
2. As of this report a uniform H_2 evolution rate has not been obtained; however, seeding the NaBH_4 with LiH and CoCl_2 has produced a more predictable reaction time.
3. Foam has been materially reduced by using a 3 in. dia. canister and a limited amount of CoCl_2 . H_2 evolution may be improved with other combinations of catalysts.
4. Although foam has been reduced from values reported by other investigators, it continues to be a problem in large scale, 270 cu ft H_2 generations. A generator slightly larger than the AN/TMQ-3 will be required for H_2 generation of 270 cu ft.
5. Generation of H_2 from NaAlH_4 is relatively simple in the current AN/TMQ-3 Generator as well as the MSAR Model XI H_2 Generator designed for NaBH_4 reactions.

6. Since a reduced amount of water adversely affects the H_2 generation from $NaBH_4$, sufficient water is required to maintain the reaction temperatures below about $60^\circ C$. This factor also helped dictate the generator size. It had to be of sufficient size to contain the desired amount of water. There must also be sufficient free volume in the generator to prevent foaming over. Anti-foam agents other than turpentine which has proven the most effective may also be investigated since uniform H_2 generation from $NaBH_4$ is highly desirable.

FUTURE WORK

During the next quarter efforts to improve the NaBH_4 charge will be made by using additional catalyst such as B_2O_3 , AlCl_3 and oxallic acid in combination with CoCl_2 and investigation of water soluble plastic films for containing anti-foam agents which are required for the final stages of a NaBH_4 reaction when foam-over occurs will be investigated. Anti-foam agents other than turpentine which has proven the most effective may also be investigated; uniform H_2 generation with NaBH_4 does not make the use of anti-foam agents mandatory.

An economic evaluation of materials and the relation of NaBH_4 , NaAlH_4 and other promising materials will be compared to CaH_2 on a production basis. Large quantities of materials required in the event of a National emergency will also be considered.

Following the evaluation of the test Model XI Generator by the U. S. Electronics Research and Development Laboratory, two development model generators will be fabricated incorporating any possible revisions that would improve the generator design.

One hundred-fifty (150) NaBH_4 charges will also be packaged including any revisions which will improve the charge. These are to be supplied on 31 May 1963.

Shelf life tests will continue and thus add to the information from which specifications required for storage will be determined.

A manual with directions for operating the H_2 generators and descriptions of the charges supplied will be completed.

PERSONNEL ASSIGNED TO CONTRACT

	<u>Hours Charged</u>
Carter, William Julian	213
Kunard, David J.	24
McGoff, Miles J.	68
Spencer, Richard A.	<u>335</u>
TOTAL	640

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NSA Research Corporation, Callery, Pa. HYDROGEN GENERATION FOR HIGH ALTITUDE BALLOONS. By W. J. Carter, R. A. Spencer, M. J. McGoff. Report No. 3. THIRD QUARTERLY PROGRESS REPORT, 1 January to 1 April 1963. P. p. illus., tables, 16 April 1963. (NSAR 63-61) Contract DA-36-039-SC-90699; DA Project No. 3N-36-21-004-02 (Unclassified) Large scale test using 1 to 6 charge canisters (45 cu ft H₂ at STP/Charged Canister) were made to obtain data necessary for sizing the containers and hydrogen generator. A test hydrogen generator NSAR Model No. XI 50 NaBH₄ canister and 50 NaAlH₄ canister were shipped to U. S. Army Electronics Research and Development Laboratory at Fort Monmouth, New Jersey. Shelf life tests are continuing. Samples after three months were evaluated and early results indicate that prolonged storage of NaBH₄ at elevated temperatures (50°C) may have a prolonging effect on the hydrogen evolution. There was no visible difference between the NaAlH₄ samples at 20°C and 50°C.	NSA Research Corporation, Callery, Pa. HYDROGEN GENERATION FOR HIGH ALTITUDE BALLOONS. By W. J. Carter, R. A. Spencer, M. J. McGoff. Report No. 3. THIRD QUARTERLY PROGRESS REPORT, 1 January to 1 April 1963. P. p. illus., tables, 16 April 1963. (NSAR 63-61) Contract DA-36-039-SC-90699; DA Project No. 3N-36-21-004-02 (Unclassified) Large scale test using 1 to 6 charge canisters (45 cu ft H₂ at STP/Charged Canister) were made to obtain data necessary for sizing the containers and hydrogen generator. A test hydrogen generator NSAR Model No. XI 50 NaBH₄ canister and 50 NaAlH₄ canister were shipped to U. S. Army Electronics Research and Development Laboratory at Fort Monmouth, New Jersey. Shelf life tests are continuing. Samples after three months were evaluated and early results indicate that prolonged storage of NaBH₄ at elevated temperatures (50°C) may have a prolonging effect on the hydrogen evolution. There was no visible difference between the NaAlH₄ samples at 20°C and 50°C.	NSA Research Corporation, Callery, Pa. HYDROGEN GENERATION FOR HIGH ALTITUDE BALLOONS. By W. J. Carter, R. A. Spencer, M. J. McGoff. Report No. 3. THIRD QUARTERLY PROGRESS REPORT, 1 January to 1 April 1963. P. p. illus., tables, 16 April 1963. (NSAR 63-61) Contract DA-36-039-SC-90699; DA Project No. 3N-36-21-004-02 (Unclassified) Large scale test using 1 to 6 charge canisters (45 cu ft H₂ at STP/Charged Canister) were made to obtain data necessary for sizing the containers and hydrogen generator. A test hydrogen generator NSAR Model No. XI 50 NaBH₄ canister and 50 NaAlH₄ canister were shipped to U. S. Army Electronics Research and Development Laboratory at Fort Monmouth, New Jersey. Shelf life tests are continuing. Samples after three months were evaluated and early results indicate that prolonged storage of NaBH₄ at elevated temperatures (50°C) may have a prolonging effect on the hydrogen evolution. There was no visible difference between the NaAlH₄ samples at 20°C and 50°C.	NSA Research Corporation, Callery, Pa. HYDROGEN GENERATION FOR HIGH ALTITUDE BALLOONS. By W. J. Carter, R. A. Spencer, M. J. McGoff. Report No. 3. THIRD QUARTERLY PROGRESS REPORT, 1 January to 1 April 1963. P. p. illus., tables, 16 April 1963. (NSAR 63-61) Contract DA-36-039-SC-90699; DA Project No. 3N-36-21-004-02 (Unclassified) Large scale test using 1 to 6 charge canisters (45 cu ft H₂ at STP/Charged Canister) were made to obtain data necessary for sizing the containers and hydrogen generator. A test hydrogen generator NSAR Model No. XI 50 NaBH₄ canister and 50 NaAlH₄ canister were shipped to U. S. Army Electronics Research and Development Laboratory at Fort Monmouth, New Jersey. Shelf life tests are continuing. Samples after three months were evaluated and early results indicate that prolonged storage of NaBH₄ at elevated temperatures (50°C) may have a prolonging effect on the hydrogen evolution. There was no visible difference between the NaAlH₄ samples at 20°C and 50°C.
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