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MSAR 63-17

Report No. 2
Contract No. DA-36-039-SC-90699
DA Project No. 3M-36-21-004-02

Second Quarterly Progress Report
1 October 1962 to 1 January 1963

to

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

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HYDROGEN GENERATION
FOR
HIGH ALTITUDE BALLOONS

February 26, 1963

MSA Research Corporation

Subsidiary of Mine Safety Appliances Company

Callery, Pennsylvania

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HYDROGEN GENERATION FOR HIGH ALTITUDE BALLOONS

Report No. 2
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

Second Quarterly Progress Report
1 October 1962 to 1 January 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

February 26, 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 set up 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.

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 and 150 charges of NaAlH_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, October 1, 1962 - January 1, 1963, additional small scale laboratory tests were made to obtain engineering data required for sizing the container used in packaging the NaBH_4 and NaAlH_4 , and in controlling the reaction rate for smooth thirty-minute hydrogen evolution. Two new generator designs were fabricated. Large scale tests on single charges were satisfactorily completed with the NaBH_4 and NaAlH_4 in the Model VI generator and a single 760 g charge of NaAlH_4 (138 g paraffin wax) was reacted in the AN/TMQ-3 type generator.

Additional experiments were conducted on small scale to determine the most satisfactory approach to a series of six large scale (45 cu ft) charges described in U. S. Signal Corps Requirement SC-5743A which must be completed within 30 minutes for all type balloons.

Shelf life tests were continued, and early results indicate no reduction in H_2 yields of stored samples, but two NaBH_4 samples stored at elevated temperatures require twice the time for reaction that samples stored at room temperature and freshly prepared control samples required.

REPORTS AND CONFERENCES

Within this report period October 1, 1962 - January 1, 1963, the following reports were issued and conferences were held.

Reports:

1. MSAR 62-139 Letter Report No. 3
"Hydrogen Generation for High Altitude Balloons"
W. J. Carter and M. J. McGoff,
1 November 1962
2. MSAR 62-156 Letter Report No. 4
"Hydrogen Generation for High Altitude Balloons"
W. J. Carter and M. J. McGoff
1 December 1962

Conferences:

1. On 16 October 1962, at Callery, Pa., R. Welt of the U. S. Electronics Research and Development Laboratory and L. Goldberg of the U. S. Army Electronics Material Agency, conferred with MSAR personnel on progress of the project and problems anticipated in production of end items.
2. On 21 November 1962, representatives of MSAR and American Potash Corporation discussed the feasibility of using LiBH_4 as a hydrogen source. After consultation with their research staff, LiBH_4 was found to be not economically feasible as a hydrogen source.

EXPERIMENTAL

A series of small scale (25, 50 and 130 gm) tests were made to investigate the following parameters:

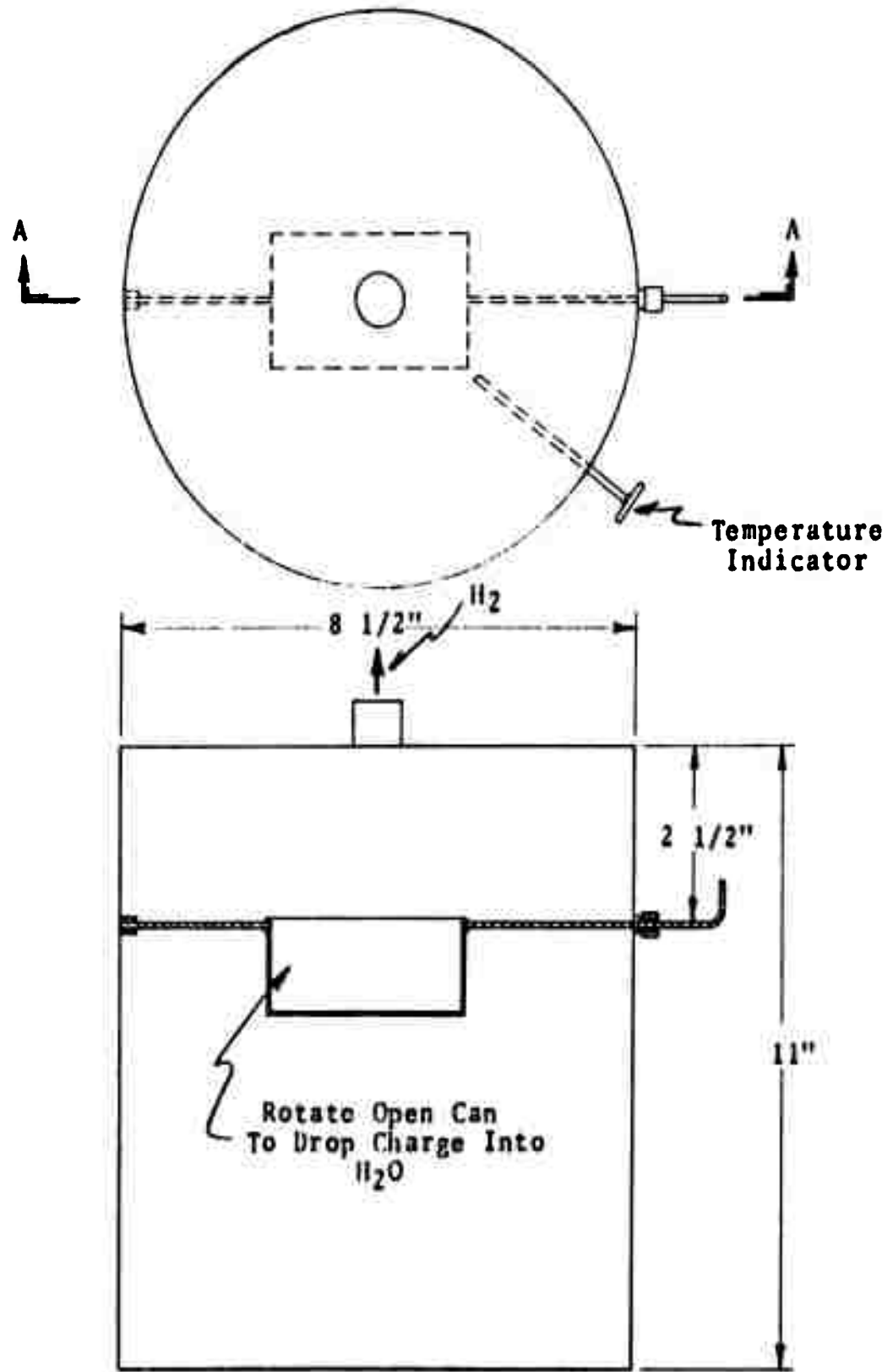
1. Surface area effects, using 27, 42.5, 45 and 70 mm diameter canisters.
2. Packing density effects, using 3000, 6000 and 10,000 psig values to prepare the cups.
3. Concentration effects, using 8 to 50 g NaBH₄/liters of H₂O in the reaction.
4. Shelf life effects, using samples stored at room temperature and 50°C

The results of data obtained from small scale tests combined with data obtained on larger scale tests will be used in sizing the chemical charge and generator for full scale tests. Other specific areas of engineering requiring consideration for sizing the generator for multiple charge operation have been water requirements, and space necessary for eliminating foam entrainment.

1. Laboratory Scale Tests with NaBH₄ and NaAlH₄

NaBH₄ - Early in this program it was found that NaBH₄ reactions in water could be effectively catalyzed for H₂ evolution yields over 90% when CoCl₂·6H₂O was used. The quantity of CoCl₂·6H₂O used in the pellets prepared initially was 8% CoCl₂. The catalyst was mixed with the NaBH₄ prior to pressing the pellet. Since the reactions were relatively fast, reacting completely in 5 to 10 min in many cases, a method of moderating the reaction and obtaining better control to provide a uniform rate of H₂ evolution was sought.

The reactions were controlled by limiting the surface areas of the charge exposed to water. Cups 27 mm ID x 100 mm long, 42.5 mm ID x 22 mm, 45 mm ID x 22 mm, and 70 mm ID x 48 mm were used in these small scale tests. These cups exposed 5.72, 14.20, 15.90 and 35.80 sq cm surface areas respectively. These cups contained 25, 50 and 118 gm of NaBH₄ plus 2 to 4 gm of CoCl₂ pressed into them. Test pressures of 3,000, 6,000 and 10,000 psi were used in preparing the cups. They were dropped into a measured quantity of tap water, in a laboratory test model generator shown in Fig. 1. This unit was similar to the previously used Model IV generator shown in the First Quarterly Progress Report.



Section A-A

FIG. 1 - TEST MODEL GENERATOR

When it was found that foaming was no problem in the 13 in. ID x 32 in. high Model V generator during a large scale test, turpentine as an antifoam agent and aniline as an accelerator were eliminated. It is theorized that if H_2 evolution rates can be maintained uniform, slow enough, and sufficient free space is left in the generator, foaming can be eliminated. The packaging of the material in a cup may also reduce the foam. The tests were run with a cooling coil, two traps, and two meters. Temperature indicators were used to monitor solution and H_2 temperatures throughout the tests. Two Model 175, Rockwell Mfg. Gas meters were used to measure flows. A flow diagram is shown schematically in Fig. 2.

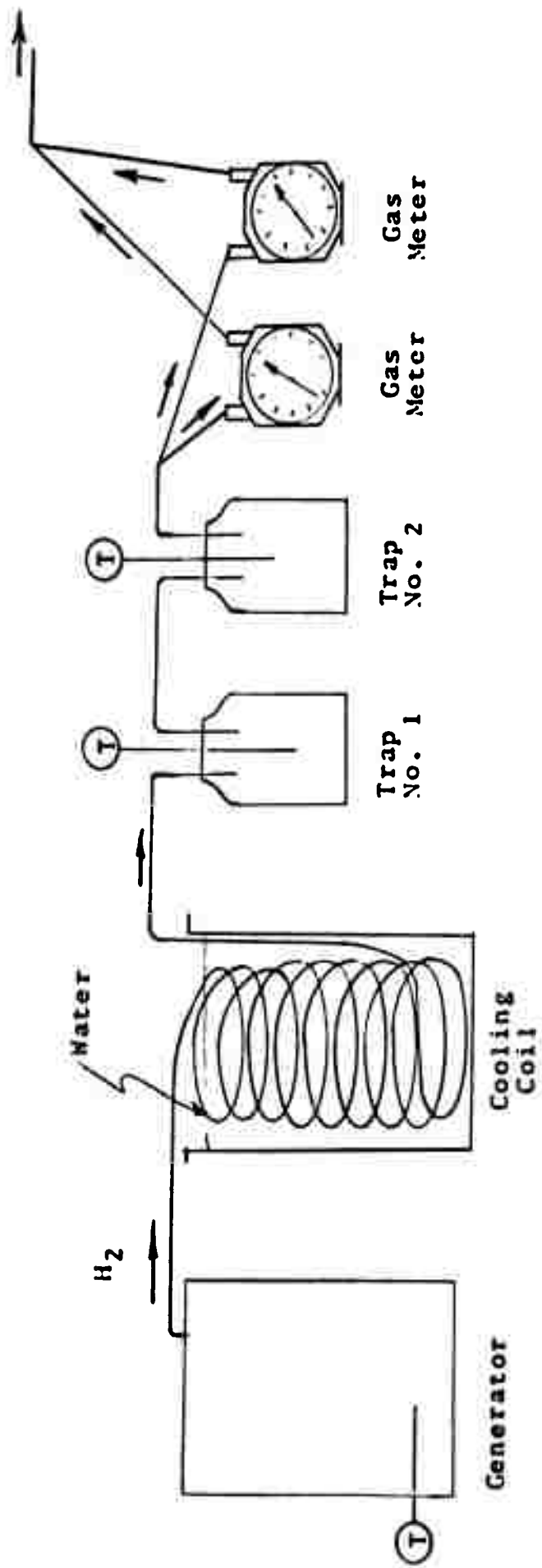
Data obtained from laboratory scale tests with $NaBH_4$ is shown in Table 1. The effects of various surface areas is plotted in Fig. 3. From this small scale experimental data shown in this figure, an estimation of cup size and reaction time for a full scale charge was predicted. Data from 27 mm cups, 42.5 mm diameter cup and 70 mm cups are shown in this figure. The reaction predicted from these tests and an actual test with 5 cups, 70 mm each are also shown in this figure.

Consecutive charges for generating 270 cu ft of H_2 in 30 min was approached on small scale experimentation. The addition of 6 charges in sequence is shown in Runs 75, 81, 82, 126 and 127 of Table 1. Although the initial cup (45 mm) generates H_2 at a uniform rate of 0.2 to 0.3 cu ft/min, (6-8 l/min), the subsequent charges evolve H_2 at an accelerated rate. This is graphically illustrated in Figs 4 and 5. The uniform rate of H_2 evolution in first charge and accelerated rate of a second charge is also shown in Fig. 6 (Run 142).

The effect of concentration in which 8 to 26 gm of $NaBH_4$ per liter of H_2O were used and no temperatures were controlled showed there was no pronounced effect on reaction rates. This is illustrated in Runs 105, 106 and 120 and 121 listed in Table 1 where similar rates were obtained in 1 and 3 liters of H_2O .

The effect of pressure used in preparing the cups indicates the lower 3,000 and 6,000 psi cups may evolve H_2 faster than the 10,000 psi cups but since a more predictable reaction rate is desired all cups will be pressed at 6,000 to 10,000 psi. The higher forming pressure will also provide a more compact charge with the resultant higher unit density.

Although there are exceptions, sufficient data was obtained to indicate that the higher pressure used in preparing the cup, the slower the reaction. Since the density of the packed material was approximately 50 lb/cu ft, an occasional cup was suspected of floating. The latest data and all subsequent charges will be prepared with a lead weight to increase



Legend:



Temperature Indicator

FIG. 2 - SCHEMATIC FLOW DIAGRAM

TABLE 1 - LABORATORY SCALE TESTS OF HYDROGEN GENERATION FROM NaBH_4

NaBH ₄ Weight (g)	Form and Pressure (psig)	Volume H ₂ O (liters)	H ₂ O Temp (°C)		H ₂ Yield (liters) (STP)		Elapsed Time (min)	Run No.	pH	
			Start	End		%			Initial	Final
25	Pellet, - 6,000	0.5	28	42	56.5	95.0	3.8	60		
50	- 10,000	1.9	22	57	113.2	95.7	6.5	65		
50.4	- 10,000	1.9	26	54	113.0	95.6	7.3	66		
50.4	6,000	1.9	23	56	115.8	98.0	9.5	67		
50	2 x 45 mm cup (1) 6,000	1.9	24	57	113.5	95.5	5.0	68		
50	27 mm cup (2) - 10,000	1.9	24	58	116.4	98.6	22.3	71		
50	27 mm cup (2) - 10,000	1.9	23	57	118.5	100.0	20.8	72		
25	45 mm cup (1) - 6,000	0.8	26	63	56.5	95.6	6.5	73		
50	42.5 mm cup (3) - 10,000	0.9	20	56	111.0	94.8	10.2	74		
25	45 mm cup (1) - 6,000	0.95	24	56	58.6	98.8	28.5	75a	pH ₀ 7.6	pH 10.7
25	45 mm cup - 6,000	Successive charges added to same H ₂ O.	71	77	41.4	70.0	2.8	b	10.7	11.2
			70	79	46.7	78.8	3.5	c	11.2	11.2
			70	82	29.6	50.0	3.0	d	11.2	11.5
			66	82	55.2	93.0	4.0	e	11.5	11.5
			81	88	49.8	84.0	4.2	f	11.5	11.5
25	45 mm cup - 6,000	0.95	27	30	53.8	91.0	14.5	79		
		0.95	30	32	52.0	88.7	22.0	80		
		0.95	20	56	54.5	92.0	5.0	81a	8.0	10.6
25(4)		Successive	35	70	57.3	96.6	15.5	b	10.6	11.1
25(5)		charges added to same H ₂ O.	24	69	56.8	96.0	11.2	c	11.1	11.4
25(5)	45 mm cup - 6,000		25	65	57.9	97.5	16.2	d	11.4	11.6
25(5)			--	--	--	--	--	e	(Pellet dissolved, No H ₂ evolution)	
25	45 mm cup - 6,000	0.95	21	53	56.4	95.0	18.0	82a	10.7	11.0
25(5)		Successive	35	68	58.5	98.7	10.0	b	11.0	11.5
25(5)		charges	35	70	57.5	97.0	9.0	c	11.5	11.6
25(5)		added to	22	56	57.5	97.0	37.7	d	11.6	11.6
25(5)		same H ₂ O.	26	73	57.0	96.2	7.5	e	11.6	11.6
25(5)	45 mm cup - 6,000	-	24	64	58.9	99.2	15.0	82f	11.6	11.6
25(5)			23	56	57.8	97.5	34.0	82g	11.6	11.6
25		0.105	25	60	54.0	97.0	9.5	83		
		10,000	14	55	55.5	93.6	11.0	105		
		10,000	16	55	55.5	93.6	12.0	106		
		6,000	16	55	55.8	94.2	10.0	107		
		6,000	17	55	53.5	90.0	10.1	108		
		6,000	16	50	53.5	90.0	20.1	109		
		6,000	17	52	53.5	90.0	18.0	110		
25	27 mm cup - 3,000	1.0	13	52	55.2	93.3	8.0	111		
25	27 mm cup - 10,000	1.0	18	55	55.8	94.2	6.5	112		
25	45 mm cup - 6,000	1.0	14	54	55.0	93.0	5.0	113		
25	45 mm cup - 10,000	1.0	18	55	55.2	93.3	45.0	114		
50	70 mm cup (4) - 3,000	1.0	18	79	114.0	96.0	6.0	115		
50	6,000	1.0	18	79	113.2	95.6	5.5	116		
25	27 mm cup - 10,000	3.0	22	34	52.4	88.5	12.5	120		
25	45 mm cup - 10,000	3.0	23	34	53.0	89.5	9.0	121		
50	70 mm cup (4) - 10,000	6.0	22	30	108.0	91.4	25.0	122		
25	45 mm cup 10,000	1.0	23	57	56.6	95.6	7.1	126a	7.6	10.8
		Successive	27	65	58.8	99.5	6.0	b	10.8	11.3
		charges	29	63	55.0	95.4	5.0	c	11.3	11.3
		added to	32	65	56.8	96.2	6.5	d	11.3	11.6
		same H ₂ O,	30	62	56.6	95.6	13.0	e	11.6	11.8
		cooled	30	65	56.5	95.4	10.0	f	11.8	--
	Pellet, NoCoCl ₂	between	23	68	56.8	96.6	4.0	g	--	--
		addition.								
	45 mm cup - 10,000	3.0	22	34	53.7	90.5	9.5	127a	7.6	10.3
		Successive	32	43	54.4	92.0	4.2	b	10.3	10.6
		charges	30	42	52.7	89.0	4.0	c	10.6	10.7
		added to	30	42	54.5	92.0	4.3	d	10.7	10.8
		same H ₂ O	33	44	56.0	89.5	4.2	e	10.8	11.0
		cooled	32	44	54.5	92.0	4.0	f	11.0	11.1
25(5)	Pellet, NoCoCl ₂	between	30	43	55.3	93.4	5.0	127g	11.1	11.1
		each addition.								
118	70 mm cup - 10,000	15.1	20	31	273.0	89.0	19.2	141		
118	70 mm cup - 10,000	11.3	19	34	274.0	87.5	23.0	142a		
18	70 mm cup - 10,000	Add to Same H ₂ O	34	50	281.0	91.6	5.3	142b	--	--
22	Pellet - 6,000	1.0	24	--	--	--	11.0	135	--	--

Note: (1) 1,590 sq mm exposed surface for each 45 mm cup.
(2) 572 sq mm exposed surface for each 27 mm cup.
(3) 1,420 sq mm exposed surface for each 42.5 mm cup.
(4) 3,850 sq mm exposed surface for each 70 mm cup.
(5) All samples contain 8% $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, except these.

Reaction of NaBH_4 vs Time

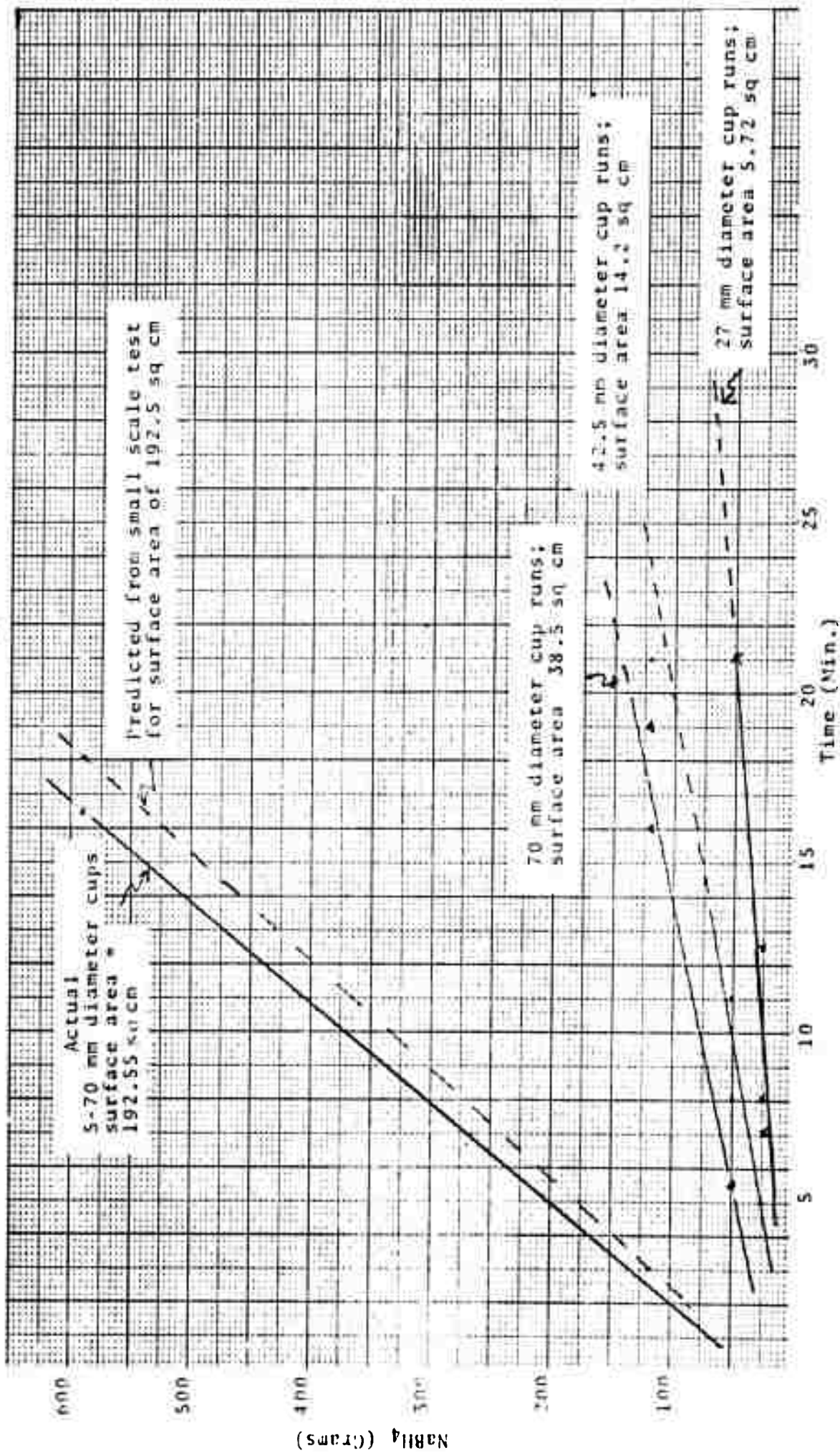


FIG. 3 - EFFECT OF SURFACE AREA ON REACTION TIME

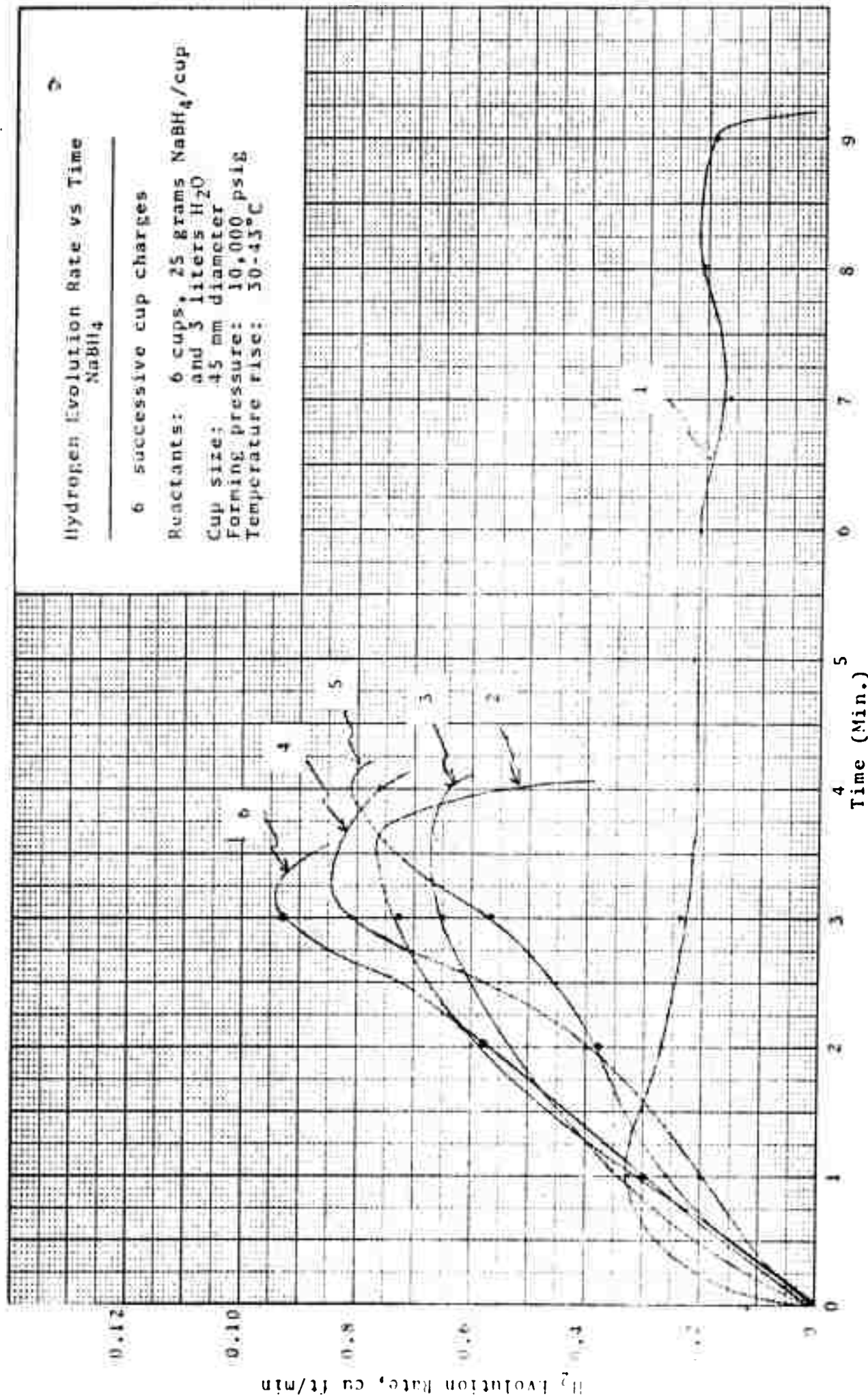


FIG. 4 - SIX CONSECUTIVE NaBH₄ CHARGES

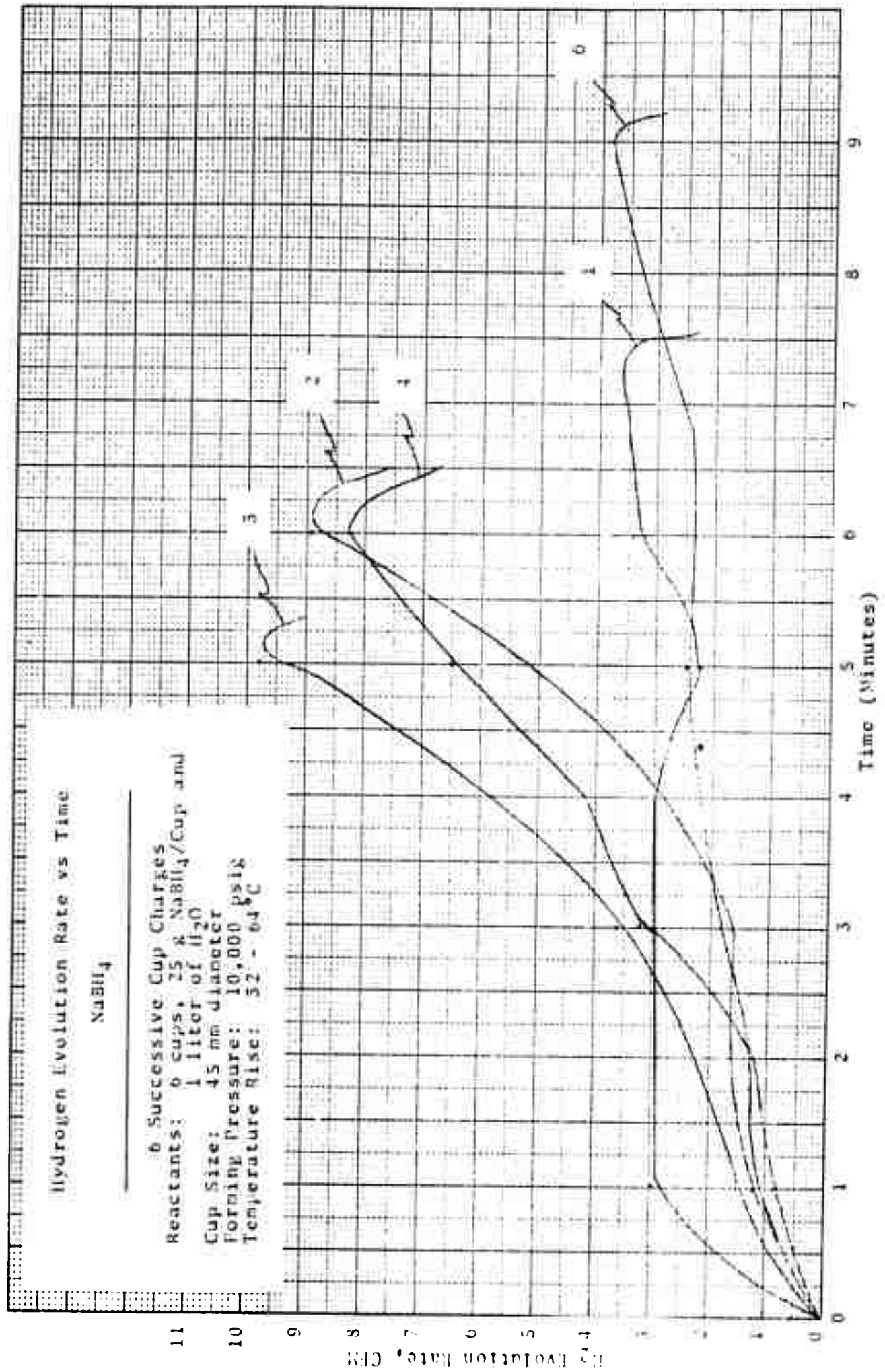


FIG. 5 - SIX CONSECUTIVE NaBH₄ CHARGES

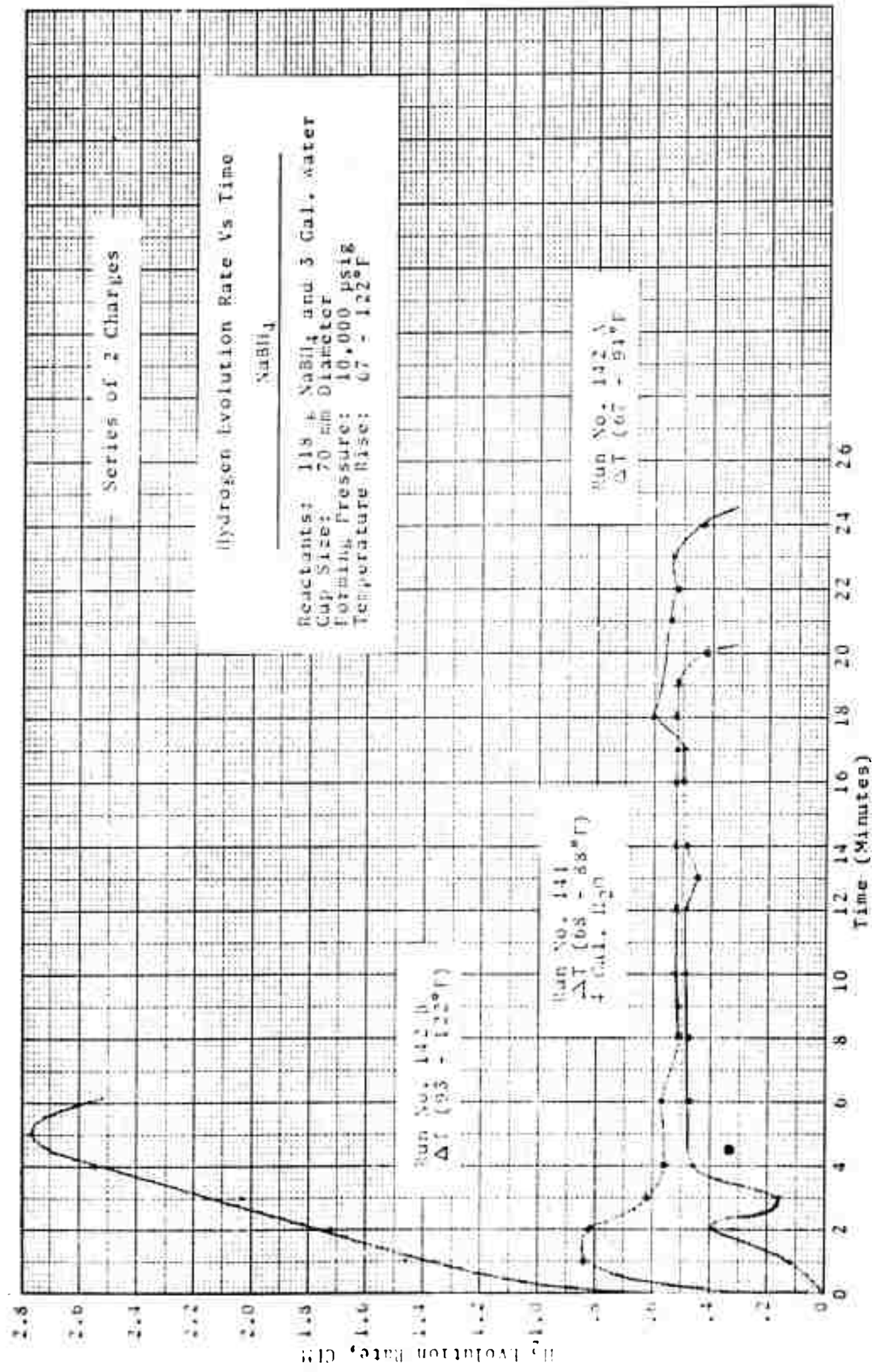


FIG. 6 - CONSECUTIVE NaBH₄ CHARGES

the density, so the material will sink. One run observed in a glass container indicated the cup could float. This condition could have occurred in some tests where exceptionally long reaction rates were encountered.

Other small scale tests with NaBH_4 were:

1. The addition of water dropwise to a bed of the NaBH_4 in a granular form. This method, although satisfactory from a single observation, would involve more elaborate equipment than is presently envisioned for the full scale generation of H_2 from NaBH_4 .
2. The CoCl_2 was added to the water and the charge then added to the water. The reaction was so slow, 0.04 cu ft/min, it was interrupted after 11 min and less than 20% complete (Run No 135).
3. An attempt was made to control the reaction by raising and lowering a basket containing a cup of NaBH_4 into the water (Run No 80). A fairly uniform rate of H_2 evolution was obtained and control of this reaction appeared possible. However, this method would require greater manipulation and more complex equipment than desired at this stage of development.

NaAlH_4 - An experimental program on a laboratory scale was initiated to determine the effects of surface area, concentration and forming pressures used in preparing cups on H_2 generation, as was done with NaBH_4 . The effect of multiple charges will be investigated early in the next report period, using a Model VI generator and the AN/TM 3 generator.

The most significant experience encountered was a violent reaction which occurred when a 50 gm cup of NaAlH_4 containing no wax was prepared. This was an attempt to eliminate wax from the pressed charge. The material probably crumbled and sparks from the dust, which have been observed previously on 1 gm pellet samples, ignited the H_2 as it evolved. Since maximum safety precautions had been taken there was no injury or property damage encountered. However, this experience pointed out the need for extreme caution in handling, packaging and generating H_2 from this material. Wax is definitely required to prepare NaAlH_4 cups and provide safe H_2 generation.

Immediately following this experience and proving the absence of wax caused the violent reaction, a 25 gm cup and a 50 gm cup containing 20% parafin wax were successfully reacted under similar conditions. Data obtained from these and other small scale tests with NaAlH_4 are shown in Runs 136, 137 and 138, Table 2.

The effect of pressure used in preparing the cups on reaction time is clearly shown in Fig. 7, (3,000 psi); Fig. 8, (6,000 psi); and Fig. 9, (10,000 psi). The average rates of evolution are 21, 21 and 13 liters/min respectively. Since the slower reaction rate is desired, and a well compacted cup is required to eliminate crumbling, higher pressure will be used in preparing all subsequent NaAlH_4 charges, as described for NaBH_4 .

The effect of concentration (gm NaAlH_4 /liters H_2O) on reaction rate is indicated in Runs 92, 95 and 97, where 1000 ml H_2O was used, and in Runs 93, 96 and 98, where 2000 ml of H_2O was used. There was no clearly decisive evidence of concentration effects from this data.

Surface area effects are shown in Fig. 10. Reactions with surface areas can be retarded from 0.68 cu ft/min (19.00 l/min) with 15.90 cm^2 areas to 0.16 cu ft/min (4.5 l/min) with 5.72 cm^2 area. From this data full scale charges have been scaled up to give satisfactory H_2 evolutions.

2. Large Scale NaBH_4 and NaAlH_4 Tests with Preliminary Model

Hydrogen Generators

NaBH_4 - These tests were run using Preliminary Hydrogen Generator Models V, VI, and VII. The Model V (shown in First Quarterly Progress Report) was designed with a divided drum turntable for adding pellet type charges. This unit was used for testing several full scale NaBH_4 charges and performed satisfactorily. These are Runs 55, 57 and 58 listed in Table 3. In Run No. 55, the Model V Hydrogen Generator was used in which the pellets were divided into two portions and added in two steps. This reaction was successfully used to inflate an 800 gm neoprene balloon.

Following this test, design and effort was put forth in small scale tests for the purpose of collecting data which could be applied to reacting six 45 cu ft charges in a 30 min period. The first step was to design a generator which could be used for one charge or any successive addition of charges. The first unit designed for this purpose was the Model VI shown in

TABLE 2 - LABORATORY SCALE EXPERIMENTS WITH NaAlH₄

Weight (gms)	Form and Pressure (psig)	Vol H ₂ O (liters)	H ₂ O Temp. °C		H ₂ Yield Liters, STP	Yield %	Elapsed Time (Min.)	Run No.
			Initial	Final				
25	27 mm Cup, 10,000	0.5	25	60	39.0	92	13	84
42.5	45	→	25	50	37.8	90	6	85
27	27	→	28	50	38.5	91.6	5.33	86
27	27	→	19	82	40.5	96.5	9.33	87
42.5	45	→	20	84	41.9	99.6	10	88
42.5	45	→	18	84	43.3	103	9.66	89
45	45	→	17	83	41.6	99	5.3	90
		→	16	86	42.6	101	6.5	91
		→	24	68	38.5	91.6	4	92
		→	25	48	34.8	83	3.3	93
		→	18	83	42	100	5	94
		→	20	66	37.2	88.5	2.83	95
		→	25	49	37.5	89	3	96
		→	22	66	37.1	88.5	3	97
		→	23	47	37.2	88.5	27.5	98
		→	16	88	42.1	100	3.3	99
	3,000	→	19	86	42.3	100.5	3.5	100
	3,000	→	18	85	41.1	87.8	6	101
	3,000	→	17	86	43.4	103	3	102
	6,000	→	19	86	43.7	104	3.66	103
		→	17	87	43.7	104	3.66	104
		→	23	68	40.1	96	5	129
		→	26	70	41.3	98.3	2	130
		→	25	67	40.1	95.5	3	131
		→	24	68	40.0	95	2.75	132
	10,000	→	25	74	41.8	100	2.5	133
	10,000	→	28	72	42.25	100	3	134
50	10,000	→	--	--	--	--	--	136
25	6,000	→	28	33	39.8	94	2	137
50	10,000	→	21	28	80.0	94.5	1.5	138

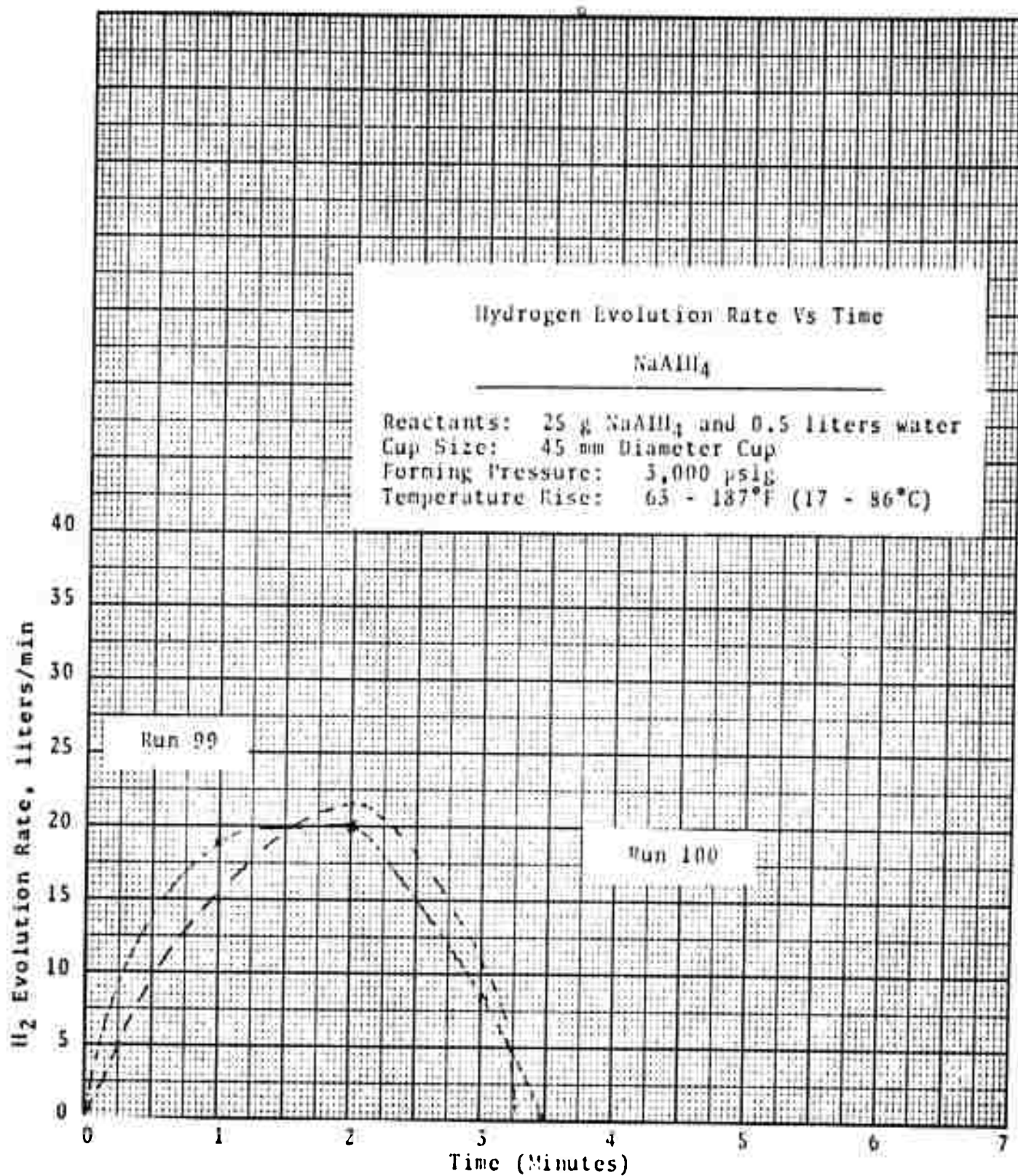


Fig. 7 - H_2 EVOLUTION FROM NaAlH_4 CUPS
PRESSED AT 3,000 psi

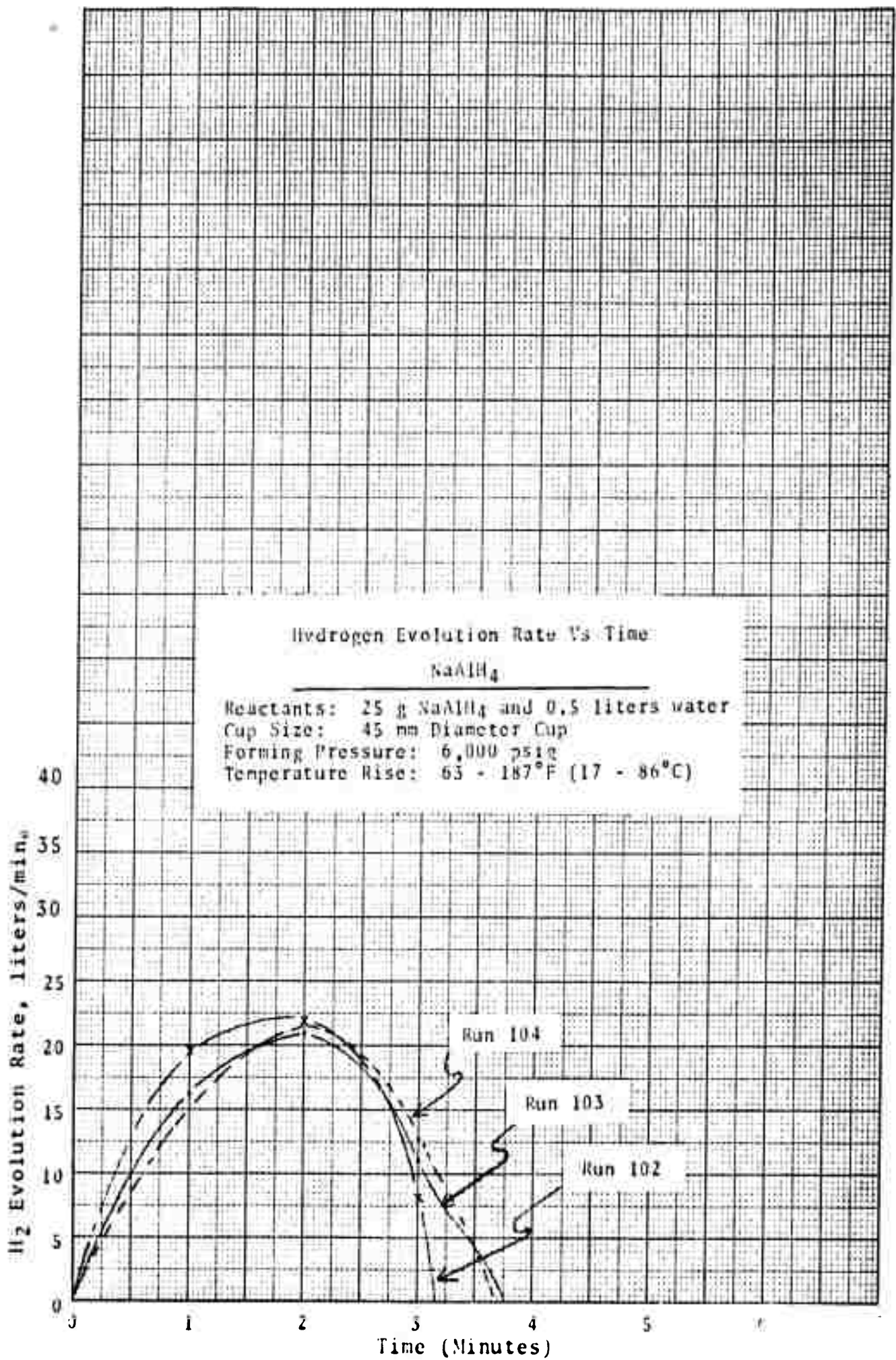


FIG. 8 - HYDROGEN EVOLUTION FROM NaAlH₄ CUPS
PRESSED AT 6,000 psi

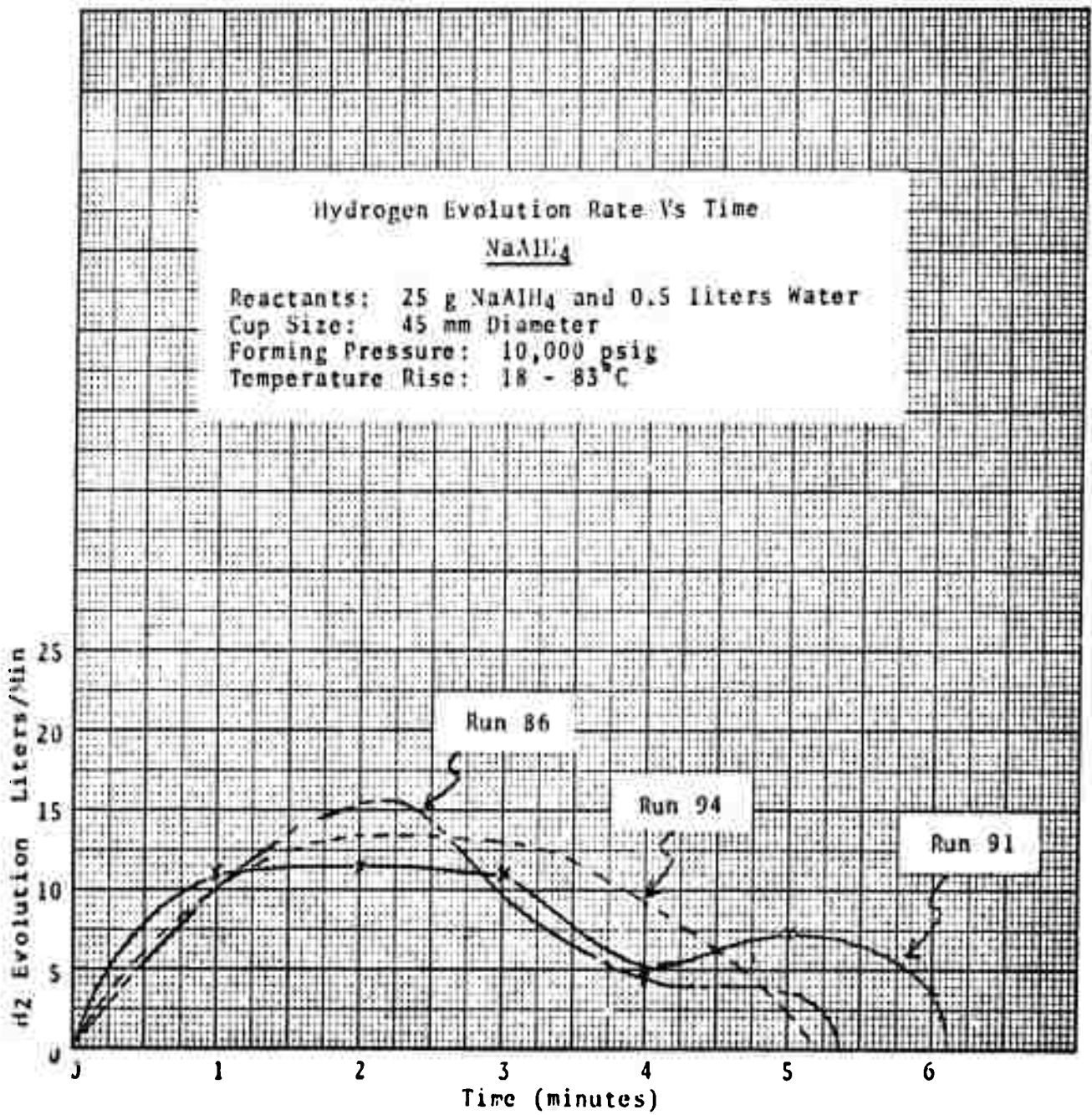


FIG. 9 - HYDROGEN EVOLUTION FROM NaAlH₄, CUPS PRESSED AT 10,000 psi

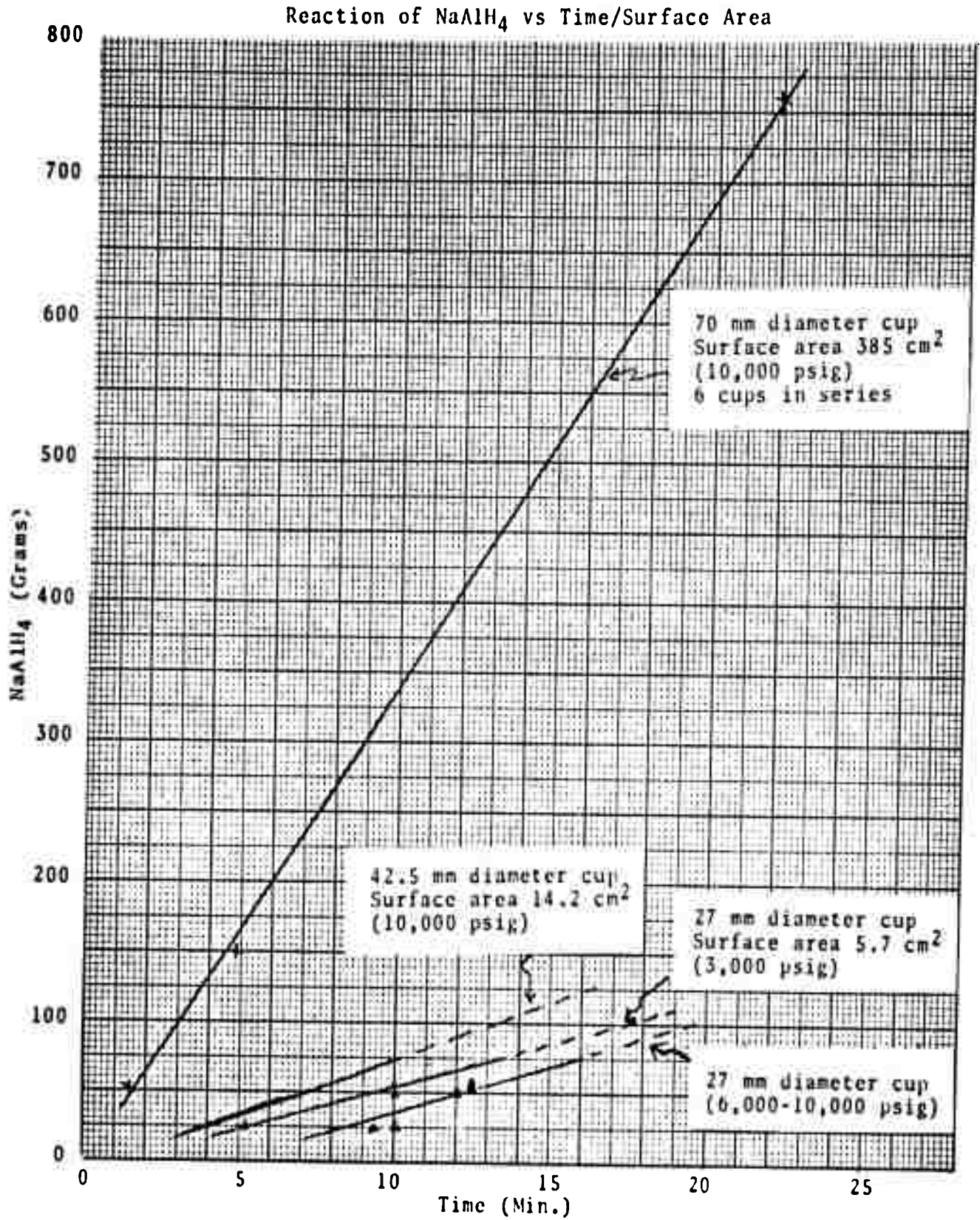


FIG. 10 - SODIUM ALUMINUM HYDRIDE REACTION TIME VS SURFACE AREA

TABLE 3 - LARGE SCALE HYDROGEN GENERATIONS FROM NaBH_4 AND NaAlH_4

Run No.	Weight of Charge (gms)		Form and Pressure of Charge (psig)	Volume H_2O (liters)	Water Temp. °C		Elapsed Time (min)	Remarks
	NaBH_4	$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$			Initial	Final		
55	NaBH_4	508 (1) 80 gm $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	Pellets 6,000 psig	19	26	31	11.0	Inflated 800 gm ML Type 518 balloon H_2 not metered.
57		508 (1) 80 gm $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	Pellets 6,000 psig	19	20	36	13.0	$\sim 43.7 \text{ ft}^3$ (STP)
58		508 gm 80 gm $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	Pellets 6,000 psig	19	19	56	15.0	40 ft^3 (STP)
143		590 47 gm $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	5-70 mm cups - 10,000 psig	45.4	19	38	16.5	45.3 ft^3 (STP)
59	NaAlH_4	760 152 gm paraffin wax	Pellets 10,000 psig	19	68	155	5	Reaction too fast for 3 L/min meter An almost violent reaction used AN/TMG-3 generator
144		760 152 gm paraffin wax	6 cups	53	27	-	7	After evolving $\sim 17 \text{ ft}^3$ the lid was blown from the generator.
145		762 138 gm paraffin wax	6 cups	37.8	57	120	15	Used Model VI generator. 39 liters STP
146		762 138 gm paraffin wax	6 cups	75.6	16	100	15	Used AN/TMG-3 generator

1. Water in generator contained 225 gm turpentine as anti-foam agent and 75 ml aniline as an accelerator.

Fig. 11. This closed model generator contained a hinged, spring loaded, trap door which was remotely released to drop the charge in the water. After release of the charge, the spring closed the door and sealed it from the reaction zone enabling the further addition of charges. This unit was tested with a series of small scale charges and performed satisfactorily.

An alternate design was conceived on the same principle but without a spring loaded trap door. This Model VII unit shown in Fig. 12 was of simple construction and contained fewer mechanical parts. It was designed for the addition of single charges. The Model VII Generator was successfully used in Run 143 where a full 590 gm NaBH_4 - 47 gm CoCl_2 charge evolved 45.3 cu ft (STP) H_2 in 16.5 min. A plot of data obtained in this run, showing an approximate 3 cu ft/min H_2 evolution over the 16.5 min run shown in Fig. 13.

NaAlH_4 - The first full scale charge, 760 gm NaAlH_4 , 152 gm paraffin wax in pellet form was reacted in the Model V generator, Run No. 59, Table 3. This reaction, approaching violence, was too fast because of excessive surface area. The H_2 evolution rate exceeded the capacity of the meter and small scale laboratory tests described in a previous section of this report were undertaken to achieve a more moderate and uniform H_2 evolution from the NaAlH_4 . This was accomplished by controlling surface area for reaction with a cup. Subsequent tests with cups containing no wax proved unsatisfactory and demonstrated the need for wax as discussed previously in the small scale tests.

The conventional generator currently used by the U. S. Electronics Research and Development Laboratory for generating H_2 from CaH_2 was used in several small scale tests with NaAlH_4 . When it appeared feasible from the small tests to use this generator on a full 760 gm charge, maximum precautions were taken. The charge was prepared in six 70 mm diameter cups and placed in the canister similar in design to the CaH_2 charge ML-305/TM. The interrupted threads were soldered to the lid of the canister containing the 70 mm cups. When the charge was attached to the AN/TMQ-3 generator the water flowed into hole punched out as is customarily done for CaH_2 evolutions

As the water flowed into the canister it reacted with the NaAlH_4 . After 7 min H_2 evolution a stoppage of gas occurred, Run No. 144. This stoppage of gas flow is believed due to buoyancy of the 70 mm cup and internal pressure holding the cup against the outlet holes. The canister containing six cups was redesigned with a rod being soldered inside to prevent the cups from sealing off the holes. A second test, Run No. 146, was made using the same configuration except the rod was inserted and 40.2 cu ft of H_2 was evolved in 15 min. This test with

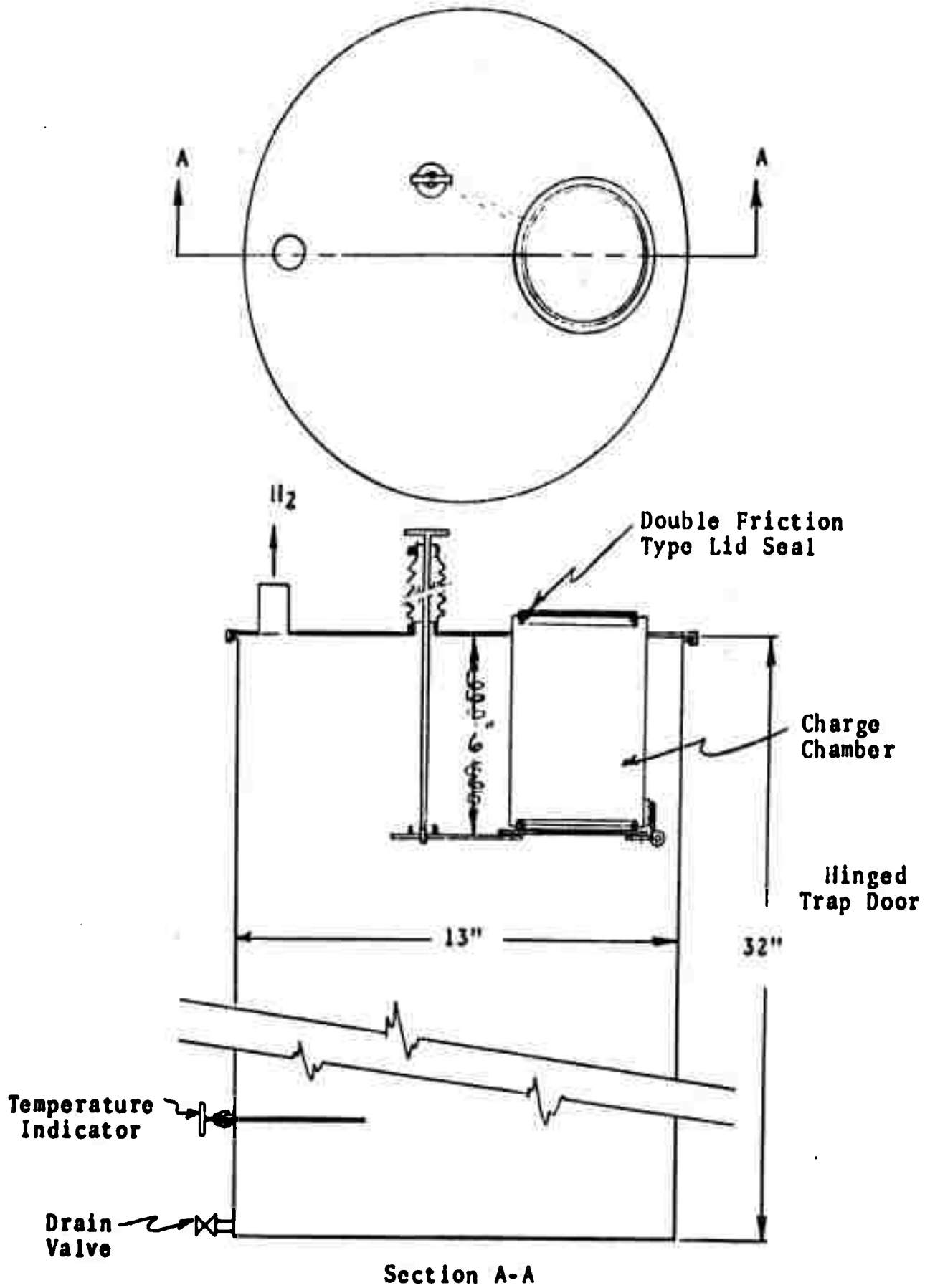


FIG. 11 - MODEL VI HYDROGEN GENERATOR

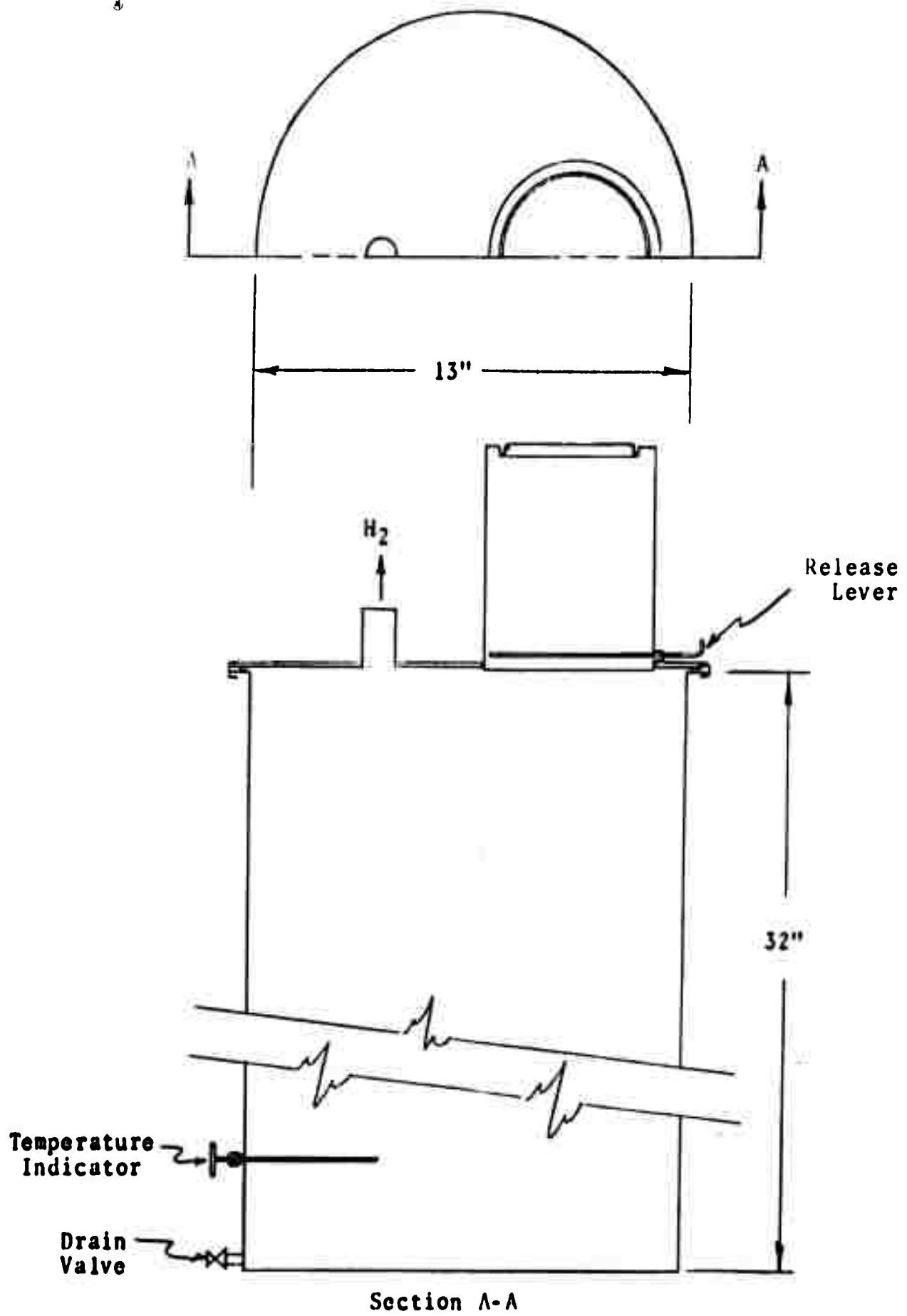


FIG. 12 - MODEL VII HYDROGEN GENERATOR

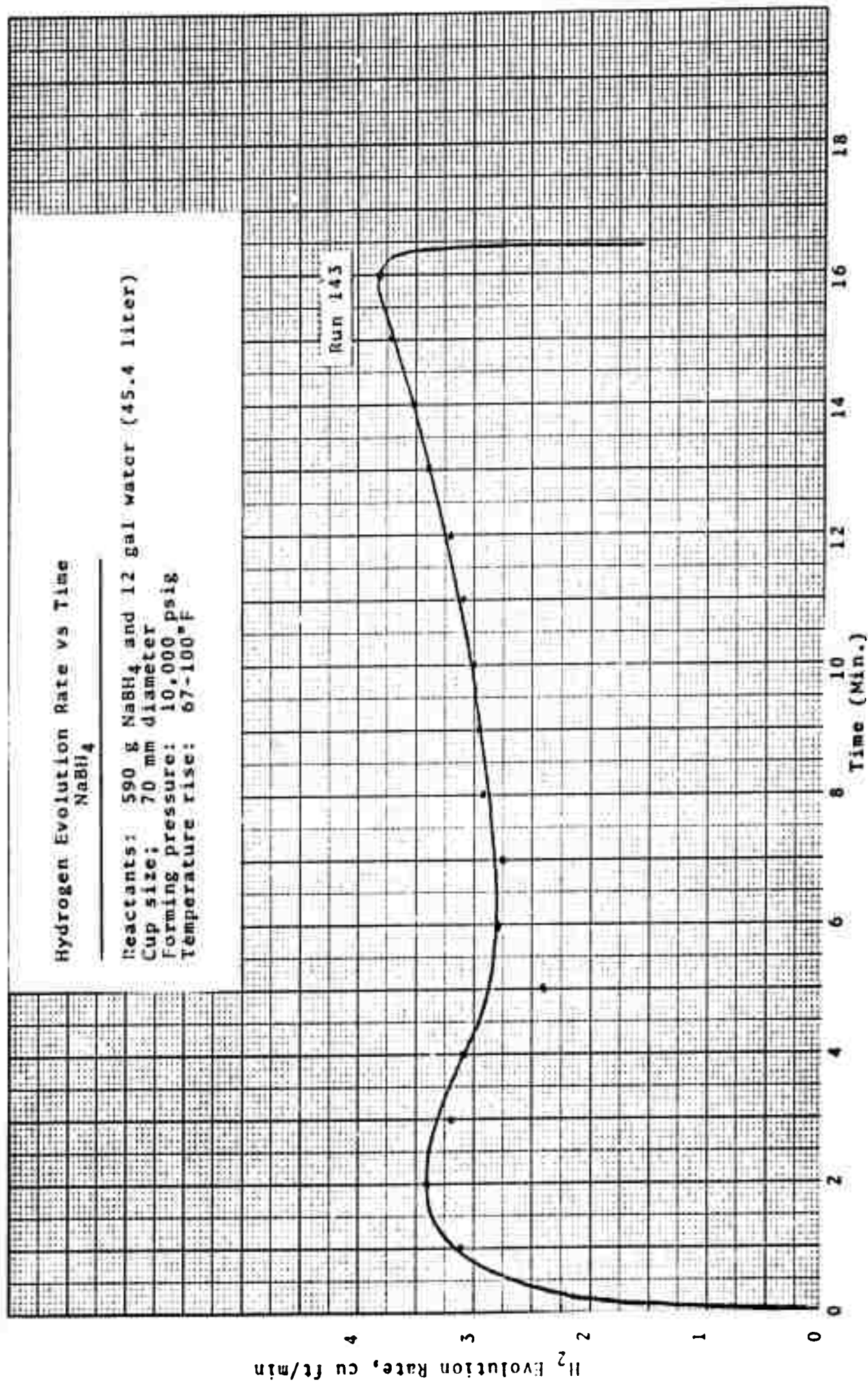


FIG. 13 - 45 cu ft NaBH₄ CHARGE

NaAlH₄ in a canister design similar to the ML-305/TM canister used with CaH₂ and the AN/TM γ -3 generator, has shown that a NaAlH₄ charge is also workable.

The feasibility of using the Model VI generator with the NaAlH₄ charge was proven in Run No. 145 and indicates the generator design is also feasible with NaAlH₄ as well as NaBH₄. This also indicates any generator design capable of generating H₂ from NaBH₄ can also be used to generate H₂ from NaAlH₄.

3. Shelf Life Tests

Samples of NaBH₄ and NaAlH₄ have been packaged in the two forms which may result in the final packaging. The initial form was 50 and 70 gm pellets of NaBH₄ and NaAlH₄ pressed to 6000 psi and the second, a 45 mm cup containing 25 gm each of NaBH₄ and NaAlH₄. Each NaBH₄ charge contained 8% CoCl₂ and each NaAlH₄ charge contained 20% parafin wax. Half the samples were stored at room temperature (20°C) in sealed containers and the other half were stored at 50°C in an oil bath.

Samples will be evaluated at 1, 3, 8 and 18 month intervals. Fresh samples will be prepared and analyzed with each set of tests. Each sample will be tested for H₂ evolution rate and efficiency under identical conditions at each time interval. Results will be plotted on a semi-log scale to enable extrapolation of data to a 10 year life estimate.

NaBH₄ - The first month samples of NaBH₄ were evaluated in Runs 67, 107 and 108 stored at room temperature. Results of these tests are shown in Table 1. Run 67, a 50 gm pellet evaluated in 1900 ml H₂O (26 g/l) gave better yield but at a slightly slower rate than its control, Run No. 66. When Runs 107 and 108 were compared to Runs 105 and 106, the freshly prepared controls, there was no significant difference.

Samples stored in the oil bath at 50°C for one month, Runs 109 and 110 when compared with freshly prepared control samples and other one month shelf samples, gave yields which were slightly less, but reaction rates required, significantly, twice the normal reaction time. Samples with anhydrous CoCl₂ will be packaged for similar tests to determine if the moisture affects this reaction rate, or if the elevated temperature is responsible.

NaAlH₄ - One month shelf samples of NaAlH₄ stored at room temperature and in an oil bath at 50°C showed no significant difference from the freshly prepared controls. The results are shown in Runs 129 and 130, fresh controls; Runs 131 and 132 shelf samples at room temperature; and Runs 133 and 134 samples stored at 50°C.

4. Other Chemicals

Small scale tests with Li-Al alloy and Li-CaH₂ (5% Li) were made to evaluate their use in hydrogen generation. H₂ evolution rates were so slow and yields so low that further investigations are not warranted with progress being made with NaBH₄ and NaAlH₄.

LiBH₄ was investigated by the American Potash Corporation as a hydrogen source and an economic evaluation proved that it would not be practical to investigate this chemical now. It is also highly pyrophoric until it is pelletized.

The Clevite Corporation was contacted about activated aluminum which they make. Without specifying the nature of their product, they requested our hydrogen flow rate which was submitted.

The blending of NaBH₄ and NaAlH₄ in a cup may also be tried as hydrogen generating source. This may eliminate the use of cobalt as a catalyst and replace it with NaAlH₄.

CONCLUSIONS

Work has been directed toward establishing the parameters affecting H₂ generation rate with the chemical NaBH₄ and NaAlH₄ in a pressed package form.

The parameters most significant to H₂ generation are exposed surface, concentration (gm/liter) and water temperature.

1. Data obtained from small scale tests with NaBH₄ has been applied to perform a full scale 45 cu ft H₂ generation. Data from successive small scale charges has been obtained and is being applied to the design of H₂ generations up to 270 cu ft.
2. The chemical package to generate a 45 cu ft (STP) from NaBH₄ will contain 600 gm NaBH₄ and 48 gm of anhydrous CoCl₂. The packing density of this charge is about 50 lb/cu ft.
3. A full scale NaAlH₄ charge was successfully generated in the Model VI generator, Run No. 145 and in the AN/TMQ-3 generator, Run No. 146. The chemical charge contained 762 gm NaAlH₄ and was prepared by mixing it with 152 gm of parafin wax.
4. A suitable canister to generate 45 cu ft has been designed using NaAlH₄ in either the generators discussed in this report, or the present AN/TMQ-3 generator.
5. Foaming which appeared to be a problem when pellets were used has been reduced in 45 cu ft generations when the charge was in the pressed cup form. Presumably the more uniform H₂ generations obtained with a cup and sufficient void space in the new generators has contributed to the reducing of the foaming problem.

FUTURE WORK

Immediate goals are to examine full scale NaBH_4 successive charges with various concentrations. Plans are to add 6 charges immediately to a large quantity of water (about 30 gal) and obtain a 30 min reaction time, or successive charges will be added and with each charge a specified amount of water will be added. The end result will be a 30 min period of smooth H_2 evolution. For example, on a large scale, 1 charge would require 5 gal H_2O , 2 charges would require 10 gal H_2O , etc. Following successful small scale tests and data extrapolation, the full 600 gm NaBH_4 charges would be tried, selecting the best method.

Small scale successive charges of NaAlH_4 will be attempted, followed by full scale tests. Since few generator or reaction rate problems are envisioned, these full scale charges can be accomplished in the very near future.

Following these tests, the simplest, workable generator will be constructed and 50 charges each of NaBH_4 and NaAlH_4 will be prepared in cups or a canister design to give 30 min H_2 evolution in specified quantities of H_2O . The generator and charges will be submitted to U. S. Army Electronics Research and Development Laboratory for evaluation on February 28.

PERSONNEL ASSIGNED TO CONTRACT

	<u>Hours Charged</u>
Carter, William Julian	193
Kunard, David J.	0
McGoff, Miles J.	68
Spencer, Richard A.	<u>240</u>
TOTAL	501

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