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FUEL CELL

RESEARCH & DEVELOPMENT

**RESEARCH ON COMPACT
FUEL CELL POWER SUPPLIES**

PROGRESS REPORT NUMBER 6

MAY 30, 1961 - JULY 30, 1961

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CONTRACT NO. DA19-129-QM-1705 (OI 5022)

U. S. ARMY

QUARTERMASTER RESEARCH AND ENGINEERING COMMAND

NATICK, MASSACHUSETTS

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AIRCRAFT ACCESSORY TURBINE DEPARTMENT

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RESEARCH ON COMPACT
FUEL CELL POWER SUPPLIES

BY

AIRCRAFT ACCESSORY TURBINE DEPARTMENT
FUEL CELL LABORATORY

PROGRESS REPORT NO. 6

MAY 30, 1961 - JULY 30, 1961

FOR

U.S. ARMY

QUARTERMASTER RESEARCH AND ENGINEERING COMMAND
NATICK, MASSACHUSETTS

AIRCRAFT ACCESSORY TURBINE DEPARTMENT
GENERAL ELECTRIC COMPANY
LYNN, MASSACHUSETTS

INTRODUCTION

This is a technical progress report of a research program directed toward improvement of the Ion Exchange Membrane Fuel Cell concept. This work is conducted by the General Electric Company under Contract No. DA-19-129-QM-1705 (OI 5022) with the U.S. Army Quartermaster Research and Engineering Command. The objective of this program is to:

"Initiate research studies to establish those factors which currently contribute the high energy losses of the ion exchange membrane and establish approaches for minimizing such losses to achieve substantially greater power densities per unit weight of cell."

In this series of reports, progress is reported on work directed toward the above objective. In addition, progress is reported on certain related work being conducted at the Aircraft Accessory Turbine Department. This is made possible by virtue of the similarity of the objective of this contract with that of other work being conducted concurrently. The Work Plan for the total work reported is given in Section 0.4 of this report. Guidance and approval of the direction of the work under this contract is given by Mr. L. A. Spano - Chief Advanced Projects Office, Quartermaster Research and Engineering Command, Natick, Massachusetts.

These progress reports are issued on a bi-monthly basis and special summary reports will be issued as indicated. The reader should recognize that this is a progress report covering a particular period of time. The experiments reported are factual, but not necessarily complete, and any conclusions made must be considered tentative until a summary report is issued. Comments and suggestions on these reports are most welcome.

SUMMARY

Task III - Study of the External Generation and Storage of Hydrogen

Task III A - Electrolytic Methods

In order to increase the current density, it was found necessary to eliminate the monel type of separator in the electrolyzer and substitute for it a membrane type separator for the electrode compartments. By adopting this design change, current densities have been increased to 1200 ma/cm².

Task III B - Chemical Methods of Hydrogen Generation

The task involving chemical methods for the generation of hydrogen is completed. Various systems for generating hydrogen were investigated and compared. The results for the past work period are indicated in this report and are self-explanatory.

0.3 STATEMENT OF WORK

A. SCOPE:

The Contractor shall, commencing on October 1960 and continuing through 2 October 1961 furnish necessary services, labor, materials, tools, equipment and supplies, and will furnish his best efforts to do what is deemed necessary to:

Initiate research studies to establish those factors which currently contribute the high energy losses of the ion exchange membrane and establish approaches to minimizing such losses to achieve substantially greater power densities per unit weight of cell. The principal effort under this contract should include but not be limited to a study of the factors that impede the conduction of electronic and ionic charges. Investigations of resins, membrane and catalyst formulations and their incorporation into a cell should be conducted to achieve maximum performance.

B. REPORTS:

Reports shall be submitted in accordance with the following:

1. The Contractor shall submit ten (10) copies of bi-monthly reports within fifteen (15) days following the end of each reporting period, indicating progress of work to date and significant developments. These quarterly reports shall include:
 - a. An estimate of the percentage of work completed to date.
 - b. An estimate of the percentage of costs incurred to date.
 - c. A statement that to the Contractor's best knowledge the funds remaining unexpended are sufficient to complete the work called for by the contract, or a revised estimate setting forth the costs required to complete the contract and the reason (s) for the contemplated excess.
2. Upon completion or termination of the contract, the Contractor shall furnish fifty (50) copies of a complete final report or summary report which shall consolidate all findings, notes, data, computations, test procedures, evaluation of all data, test results, principles and techniques relative to the objective indicated herein.

REPORTS (Cont'd)

The Contractor shall include specific conclusions and recommendations concerning work done and detailed information and recommendations relative to further work that may be required. All reports shall be identified with Project No. 7X8-01-New. The final report shall be submitted within thirty (30) days after the expiration or termination date of the contract. Reports shall be of a paper bound brochure type using commercially available bond paper. All reports shall be submitted to the responsible Project Officer, Chemicals and Plastics Division HQ QMR & E Center Natick, Massachusetts.

PRIORITY RATING:

DD C9E CERTIFIED UNDER DMS REGULATION
#1 IS ASSIGNED TO THIS CONTRACT.

PLACE OF PERFORMANCE:

CONTRACTOR'S PLANT, WEST LYNN,
MASSACHUSETTS

0.4 WORK PLAN

REGULATED, COMPACT FUEL CELL POWER SUPPLIES

General Objective

Conduct a research and development program to achieve the capability of designing compact, lightweight and reliable, regulated fuel cell power supplies.

Research Approach

Task I

Studies of Contributions to and Efforts to Minimize Electronic and Ion Impedances

- A. Quantitative studies of contributions to electrical losses in present cells.
 1. Separate ohmic losses from electrochemical irreversibilities by use of an adaptation of the Kordesch-Marko bridge and other electronic techniques.
 2. Determine magnitude of various contributions to ohmic impedance by independent measurements of electrode-layer resistance, and electrolytic conductivity of the membranes. Establish apparent ohmic resistance attributable to the electrolytic conductivity of the membranes by other methods, including multi-frequency A.C. bridge measurements and driven cell D.C. resistance with two hydrogen electrodes.
- B. Minimization of Electrolytic Conductance Losses
 1. Investigate ion exchange resin formulations with varying exchange capacities, ionization constants and water content with regard to their effect on cell performance.
 2. Investigate the effect on cell performance of membrane thickness along with varying the amount and distribution of resin and reinforcing material.

3. Evaluate various reinforcing materials from the standpoint of cell performance, strength, compatibility, and ease of membrane manufacture.

C. Minimization of Electronic Losses

1. Investigation of catalyst impregnated carbon electrodes to minimize electrode layer resistance.
2. Evaluate various carbon blacks with different catalyst loadings and physical properties with regard to cell performance and life.

Task II

Study of the Internal Generation and Storage of Hydrogen Fuel

- A. Studies at the Electronics Laboratory, HMED, Syracuse, of palladium and other absorbents.
- B. Provision of data by the AAT Laboratory on the electrolysis characteristics of membrane fuel cells, including details of supplying the water required for such operation.

Task III

Study of the External Generation and Storage of Hydrogen

and Oxygen

A. Electrolytic Methods:

Engineering work leading to the development of a prototype model of a small high pressure electrolyzer. The initial design concept is that of a compartmented vessel for simultaneous generation and containment of hydrogen and oxygen in quantities sufficient for operation of a fuel cell for a specified duty cycle. Included will be studies of pressure reduction methods including minimum weight regulators of conventional design.

B. Chemical Methods:

Studies of various chemical systems for minimum volume and cost hydrogen generators. The size will be set by the required duty cycle.

Chemical Methods (Cont'd)

Noxious reaction products are to be avoided. Feasibility of the process using the best of the chemical system (s) will be demonstrated by constructing a laboratory model generator including pressure regulators as required.

3.0 TASK III - External Generation and Storage of Hydrogen and Oxygen

3.1 TASK IIIA - Electrolytic Methods

Sufficient data was obtained during this work period such that the effect of the current density on the operation of the electrolyzer is now clear. In Progress Report #5, it was reported that increasing current density caused sufficient agitation to allow mixing of the hydrogen and oxygen. However, after plotting the data of runs #5 thru #13 (Figures 1 thru 6, Pages 2 - 4), it becomes obvious that the mixing of the gas is not due to the increasing current density. In runs #5 thru #8, Page:2 in which the monel separator plate was not covered by porous polyethylene, the ratio of the volume of hydrogen to the volume of oxygen varied from 1.0 to 2.0 with a change in current density from 85 ma/cm² to 20 ma/cm². Runs #9 thru #11, in which the separator plate was covered with porous polyethylene (75 - 150 μ pore) the data are practically identical to the data of runs #5 thru #8. However, in runs #12 and #13 in which the monel plate is removed from the electrolyzer and separation of the electrode compartments is accomplished by only porous polyethylene (75-150 μ pore), the ratio of volumes remains near theoretical (i.e. 1.9-2.0) with a current density range of 20.9 to 1,200 ma/cm². Since the mixing of the H₂ and O₂ is apparently not taking place across the membrane, the only conclusion that can be drawn is that the monel separator is behaving as a bi-polar electrode causing the hydrogen and oxygen to be produced on each side of the membrane. That is, due to the monel separator, each of the electrode compartments is behaving as a cell. This becomes more evident if we examine the data plotted in Figures 7 and 8, Page 5. In these data we can see as high as 70% more gas being generated than can be calculated from the number of coulombs passing through the cell.

In obtaining data for runs #12 and #13 no difficulties were encountered in the operation of the cell at the higher current densities except for some carry-over of the potassium hydroxide at 1200 ma/cm².

From these data it can be concluded that:

- (1) The use of a metallic separator, such as the monel separator, is not practical in this particular design of a cell.
- (2) Operation of this cell design at current densities of 1000 to 1200 ma/cm² is entirely possible. Perhaps with slight modifications in design, current densities somewhat above 1200 ma/cm² are possible.
- (3) The porous polyethylene, (75-150 μ pore) will serve as an excellent membrane material.

It has not as yet been possible to evaluate the pressure equalizing system due to delays in obtaining the expansible bags from the vendor.

FIGURE 1

PERCENT DEVIATION VS CURRENT DENSITY

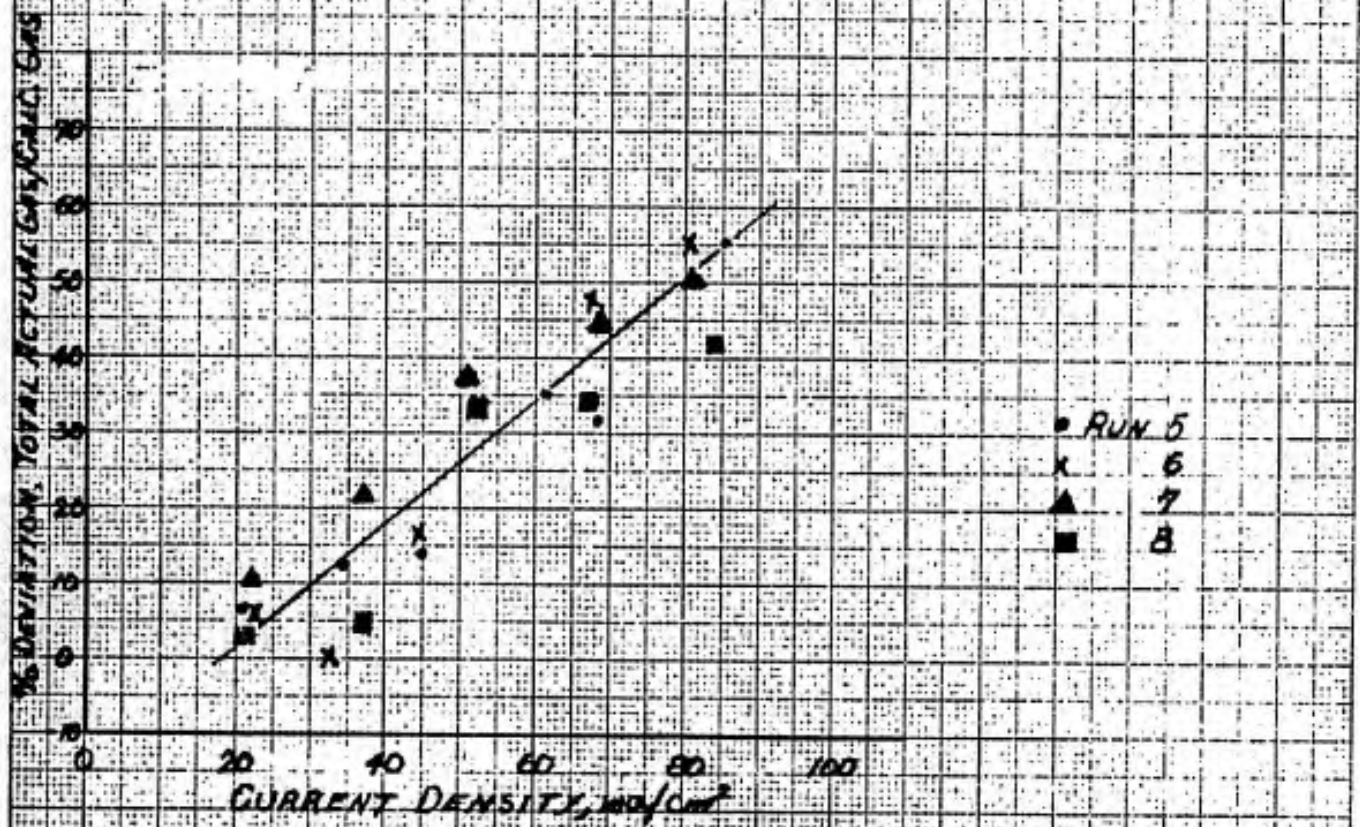
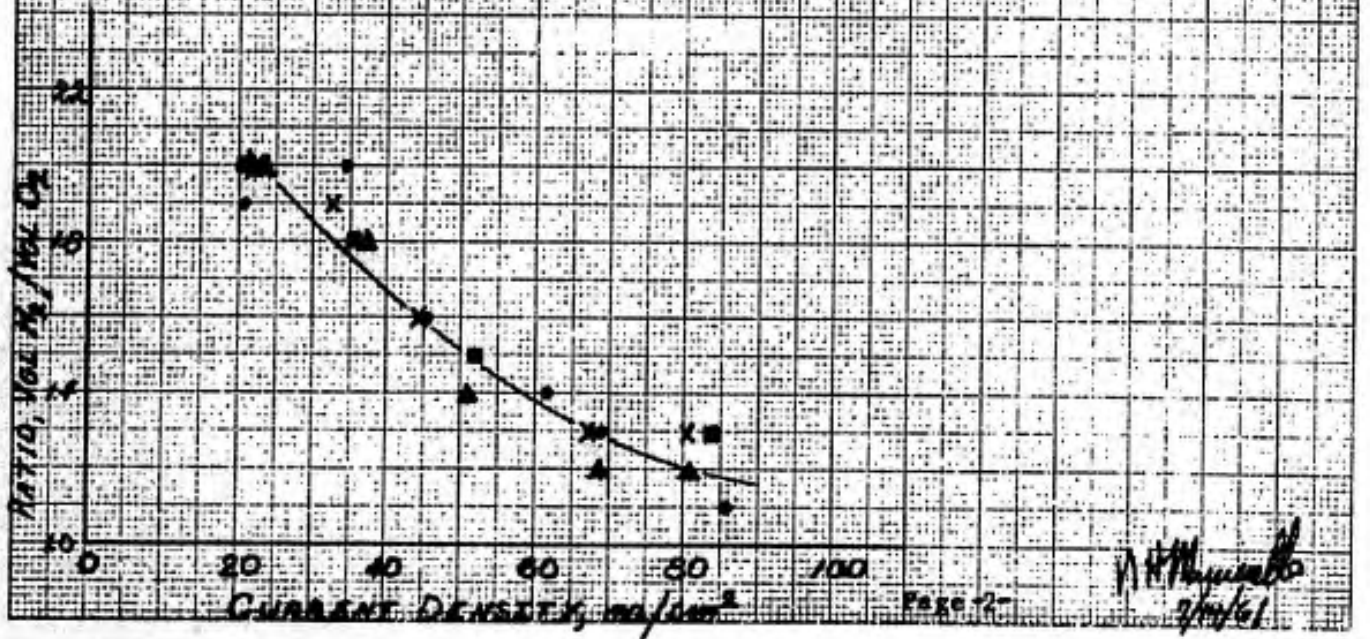
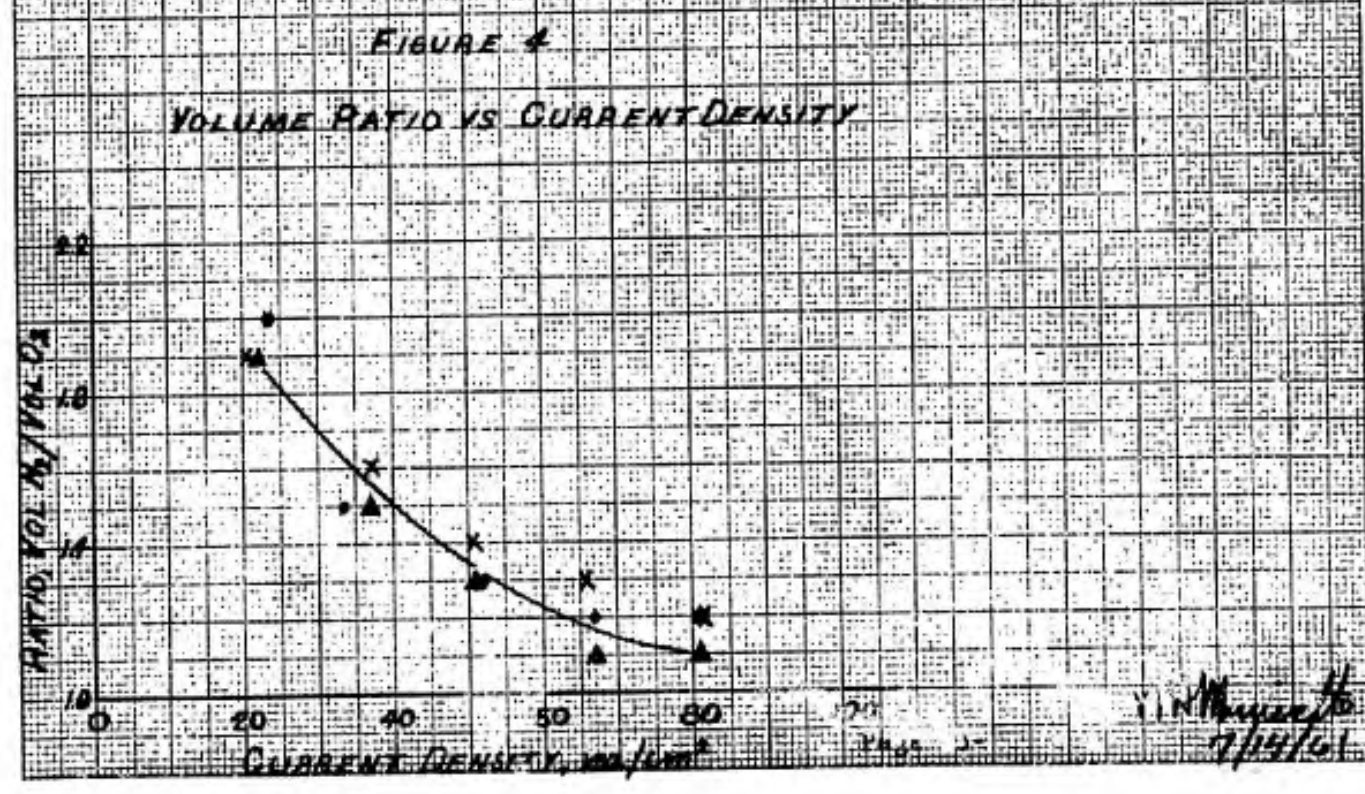
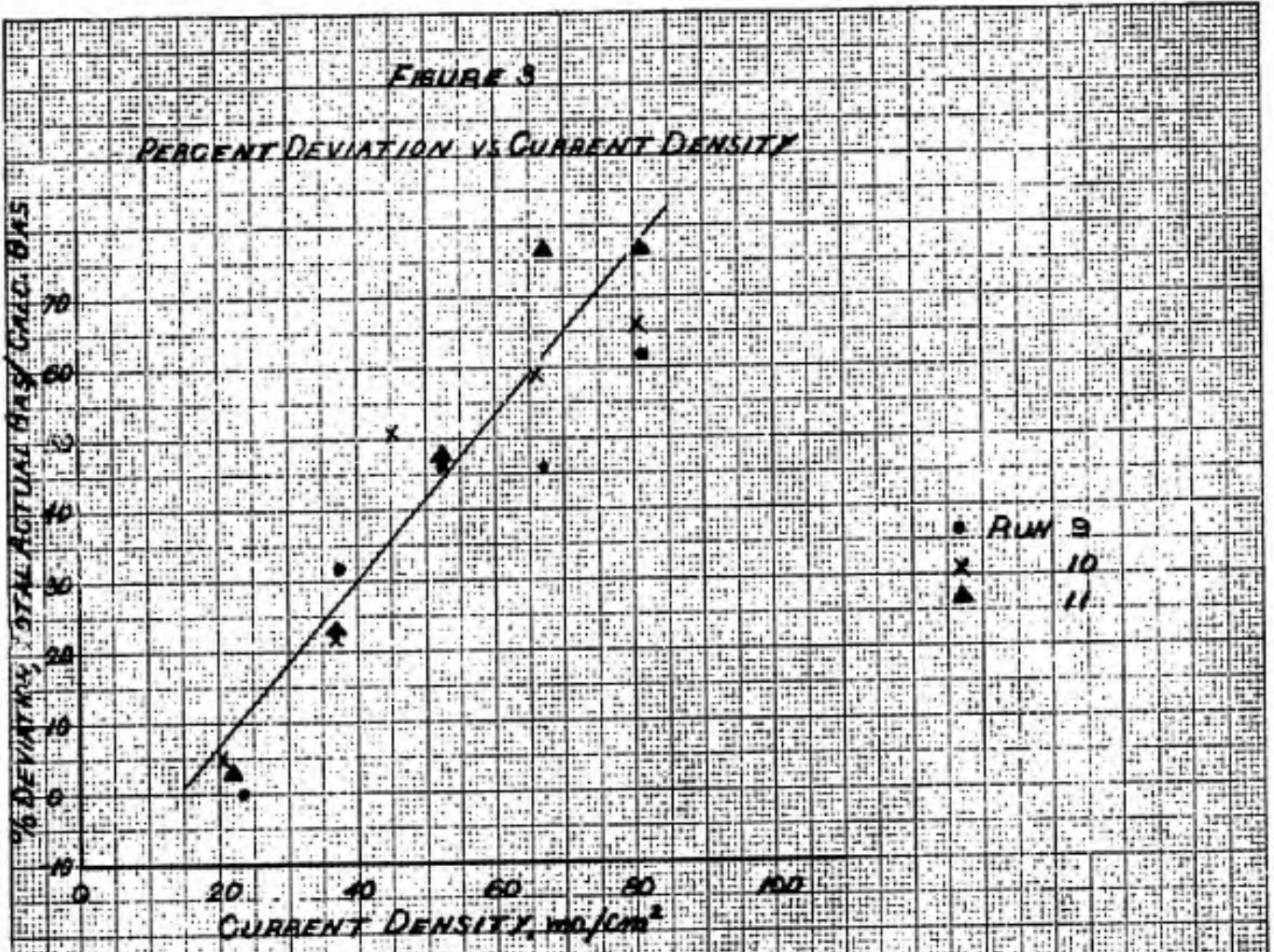


FIGURE 2

VOLUME RATIO VS CURRENT DENSITY



N. H. ...
3/1/61



J. N. Murray
7/19/61

FIGURE 5

PERCENT DEVIATION VS CURRENT DENSITY

REDUCTION, TOTAL GAS FACTOR GAS

● RUN 12
x 13

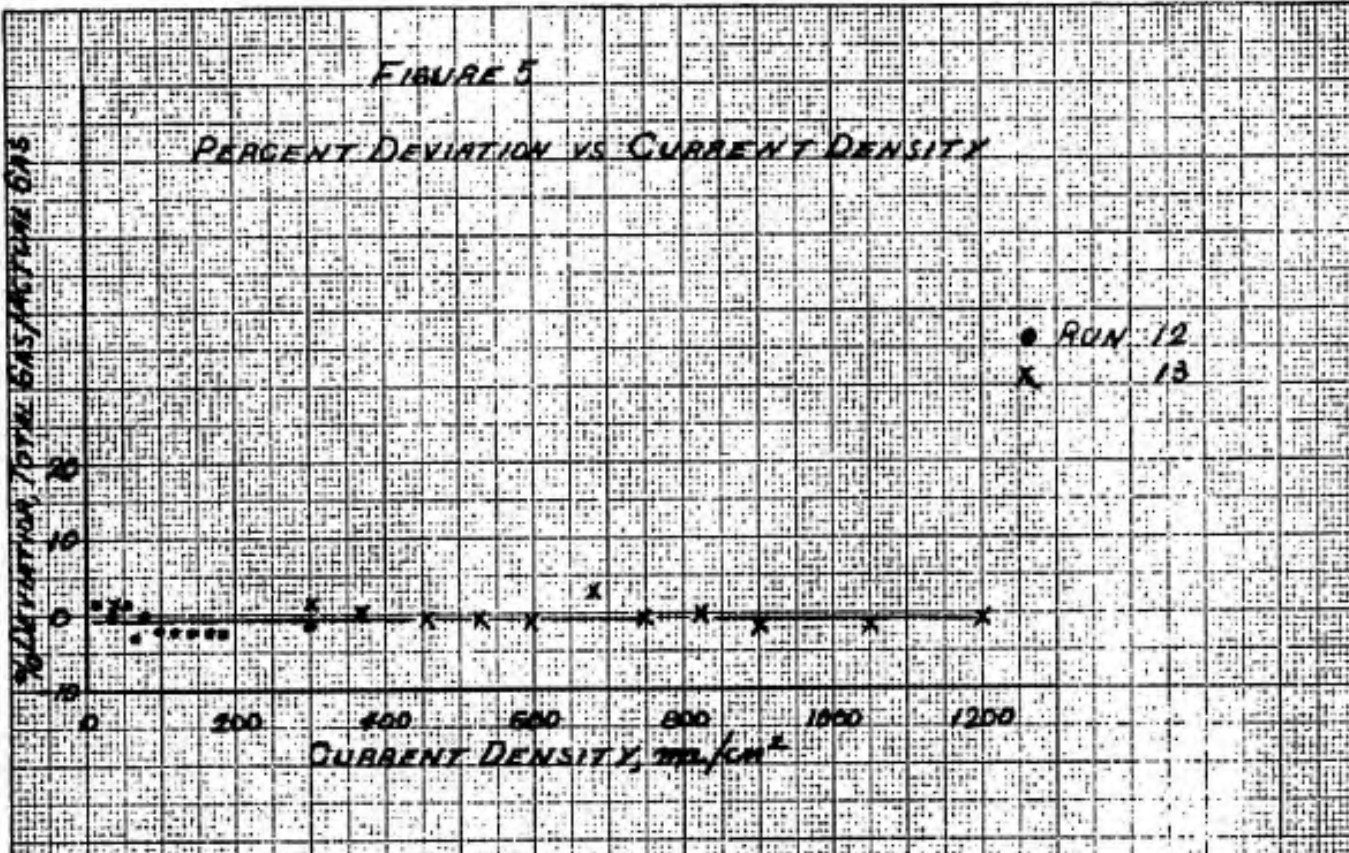
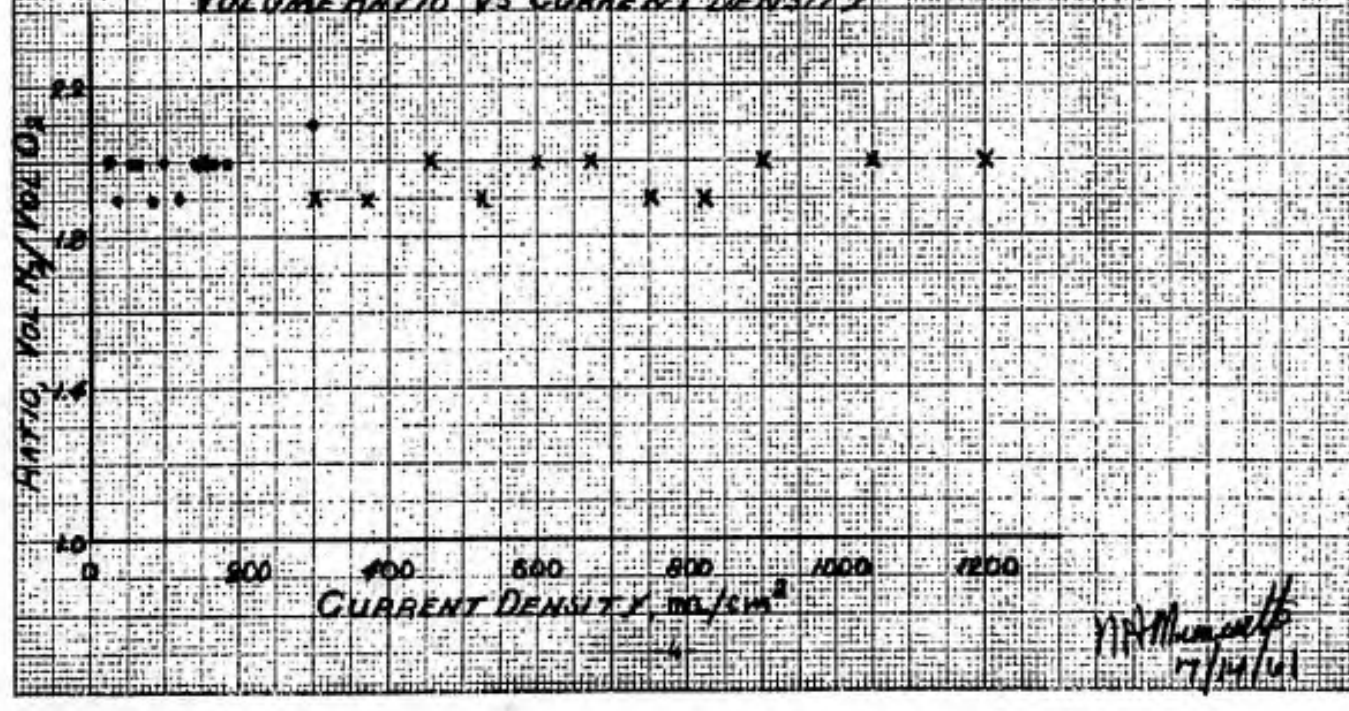


FIGURE 6

VOLUME RATIO VS CURRENT DENSITY

RATIO, VOL H₂/VOL O₂



M. M. M. M. M.
7/14/61

FIGURE 7
% DEVIATION VS RATIO

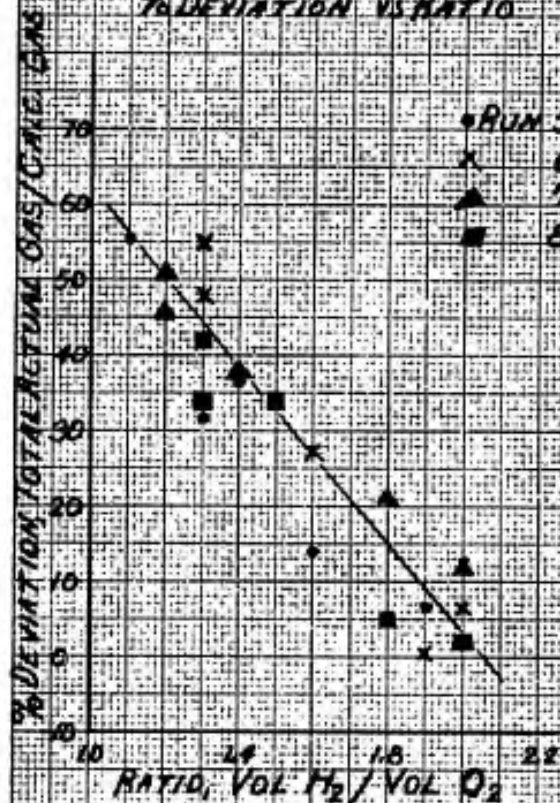
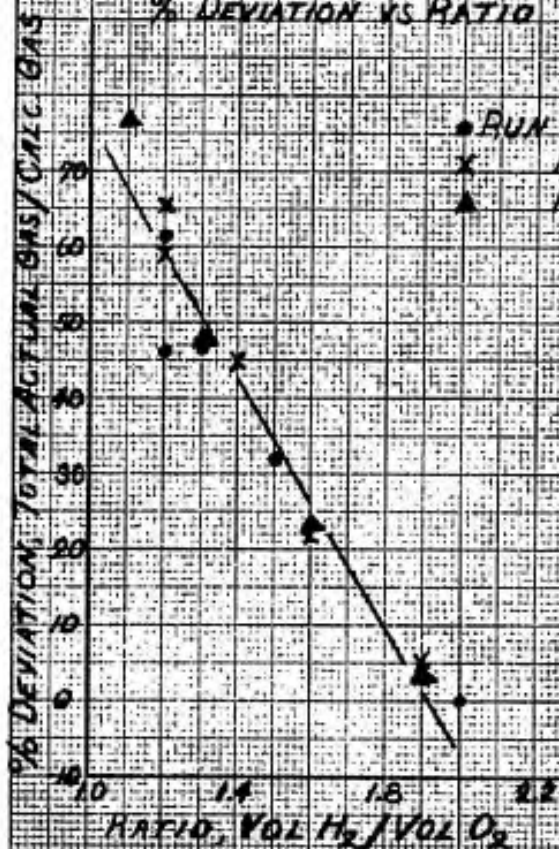


FIGURE B
% DEVIATION VS RATIO



M. W. Minette
8/7/61

RUN #5

Monel separator in system; no porous polyethylene electrode spacing: 1.5 in³

Test	1	2	3	4	5	6
E, Volts	3.61	3.90	3.80	2.95	4.0	4.1
I, Amps	0.23	0.41	0.305	0.14	0.46	0.57
Time, Minutes	49	25	38	83	21	18
Vol. H ₂ , cc	89	86	85	75	80	89
Vol. O ₂ , cc	45	61	53	39	63	79
Total Vol. Gas, cc	134	147	138	114	143	168
Ratio Vol. H ₂ /Vol. O ₂	2.0	1.4	1.6	1.9	1.3	1.1
Number Coulombs	680	616	697	698	580	616
Current Density, ma/cm ²	34.4	61.3	45.3	20.9	68.7	85.2
Calc. Vol. H ₂ , cc	79	72	81	81	67	72
Calc. Vol. O ₂ , cc	40	36	40	41	34	36
Total Calc. Vol. Gas, cc	119	108	121	122	101	108
% Deviation Total Actual Gas to Total Calc. Gas	+12.6	36.2	14.0	6.6	31.7	55.4

RUN #6

Monel separator in system; no porous polyethylene electrode spacing; $1\frac{1}{4}$ "

Test	1	2	3	4	5
E, Volts	3.55	3.05	3.75	3.90	4.05
I, Amps	0.22	0.15	0.30	0.45	0.54
Time, Minutes	60	80	36	22	17
Vol. H ₂ , cc	90	89	88	88	85
Vol. O ₂ , cc	47	45	56	66	67
Total Vol. Gas, cc	137	134	144	154	152
Ratio Vol. H ₂ /Vol. O ₂	1.9	2.0	1.6	1.3	1.3
Number Coulombs	793	722	648	595	552
Current Density, ma/cm ²	32.9	22.4	44.8	67.2	80.6
Calc. Vol. H ₂ , cc	92	84	75	69	64
Calc. Vol. O ₂ , cc	46	42	38	35	32
Total Calc. Vol. Gas, cc	138	126	113	104	98
% Deviation Total Actual Gas To Total Calc. Gas	0.7	6.3	27.4	48.0	55.0

RUN #7

Monel separator in system; no porous polyethylene electrode spacing: 1 in.

Test	1	2	3	4	5
E, Volts	3.15	3.72	3.85	3.88	3.95
I, Amps	0.15	0.25	0.34	0.46	0.57
Time, Minutes	75	45	30	23	17
Vol. H ₂ , cc	86	90	86	89	89
Vol. O ₂ , cc	43	51	62	72	73
Total Vol. Gas, cc	129	141	148	161	162
Ratio Vol. H ₂ /Vol. O ₂	2.0	1.8	1.4	1.2	1.2
Number Coulombs	676	676	613	635	583
Current Density, ma/cm ²	22.4	37.3	50.7	68.7	80.6
Calc. Vol. H ₂ , cc	77	77	71	74	67
Calc. Vol. O ₂ , cc	39	39	36	37	34
Total Calc. Vol. Gas, cc	116	116	107	111	101
% Deviation Total Actual Gas to Total Calc. Gas	11.2	21.5	38.3	45.0	50.6

RUN #8

Monel separator in system; no porous polyethylene electrode spacing: 5/8 in.

Test	1	2	3	4	5
E, Volts	2.95	3.62	3.75	3.85	3.93
I, Amps	0.14	0.25	0.35	0.45	0.56
Time, Minutes	80	47	30	23	18
Vol. H ₂ , cc	80	83	82	85	83
Vol. O ₂ , cc	40	46	56	65	66
Total Vol. Gas, cc	120	129	138	150	149
Ratio Vol. H ₂ /Vol. O ₂	2.0	1.8	1.5	1.3	1.3
Number Coulombs	673	706	630	622	605
Current Density, ma/cm ²	20.9	37.3	52.2	67.2	83.6
Calc. Vol. H ₂ , cc	78	82	74	76	70
Calc. Vol. O ₂ , cc	39	41	37	36	35
Total Calc. Vol. Gas, cc	117	123	111	112	105
% Deviation Total Actual Gas to Total Calc. Gas	2.6	4.9	34.3	34.0	41.9

RUN #9

Monel separator in System Covered with high density
Porous polyethylene, Type 3011,
75-150 pore size

Electrode spacing: $1\frac{1}{2}$ "

Test	1	2	3	4	5
E, Volts	3.38	3.55	3.78	3.85	3.92
I, Amps	0.16	0.25	0.35	0.45	0.54
Time, Minutes	70	39	27	20	17
Vol. H ₂ , cc	78	80	81	80	83
Vol. O ₂ , cc	39	55	64	68	72
Total Vol. Gas cc	117	135	145	148	155
Ratio Vol. H ₂ /Vol. O ₂	2.0	1.5	1.3	1.2	1.2
Number Coulombs	672	586	568	541	552
Current Density, ma/cm ²	23.9	37.3	52.2	67.2	80.6
Calc. Vol. H ₂ , cc	78	68	66	63	64
Calc. Vol. O ₂ , cc	39	34	33	31.5	32
Total Calc. Vol. Gas, cc	117	102	99	94.5	96
% Deviation Total Actual Gas to Total Calc. Gas	0	+32.4	+46.5	+46.1	+61.5

RUN #10

Monel separator in system, covered with high density
porous polyethylene, type 3011
75-150 pore size

Electrode spacing: 1 1/4"

Test	1	2	3	4	5
E, Volts	3.18	3.72	3.82	3.92	3.98
I, Amps	0.14	0.25	0.34	0.44	0.54
Time, Minutes	80	42	27	20	16
Vol. H ₂ , cc	81	82	80	81	81
Vol. O ₂ , cc	42	52	59	65	68
Total Vol. Gas, cc	123	134	139	146	149
Ratio Vol. H ₂ /Vol. O ₂	1.9	1.6	1.4	1.2	1.2
Number Coulombs	672	631	552	528	518
Current Density, ma/cm ²	20.9	37.3	50.7	65.6	80.6
Calc. Vol. H ₂ , cc	78	73	64	61	60
Calc. Vol. O ₂ , cc	39	37	32	31	30
Total Calc. Vol. Gas, cc	117	110	96	92	90
% Deviation Total Actual Gas to Total Calc. Gas	5.1	21.6	44.8	58.8	65.5

RUN #11

Monel separator in system, covered with high density
porous polyethylene, type 3011
75-150 pore size

Electrode spacing: 5/8"

Test	1	2	3	4	5
E, Volts	3.23	3.69	3.82	8.91	4.00
I, Amps	0.15	0.25	0.35	0.45	0.54
Time, Minutes	75	42	27	19	15
Vol. H ₂ , cc	79	82	83	85	79
Vol. O ₂ , cc	42	53	63	75	72
Total Vol. Gas, cc	121	135	146	160	151
Ratio Vol. H ₂ /Vol. O ₂	1.9	1.5	1.3	1.1	1.1
Number Coulombs	675	632	438	514	487
Current Density, ma/cm ²	22.4	37.3	52.2	67.1	80.6
Calc. Vol. H ₂ , cc	78	73	51	60	57
Calc. Vol. O ₂ , cc	39	37	25.5	30	28
Total Calc. Vol. Gas, cc	117	110	76.5	90	85
% Deviation Total Actual Gas to Total Calc. Gas	3.4	22.7	47.6	77	77

Run #12

Monel separator not in system; using high density porous polyethylene, type 3011 material as separating diaphragm

Electrode spacing: 5/8"

Test	1	2	3	4	5	6	7	8	9	10	11
E, Volts	1.99	2.12	2.20	2.25	2.33	2.40	2.48	2.55	2.62	2.70	3.06
I, Amps	0.14	0.25	0.35	0.44	0.55	0.64	0.80	0.95	1.12	1.22	2.00
Time, Minutes	84	47	34	30	22	19	16	26	23	22	15
Vol. H ₂ , cc	83	81	82	89	83	83	86	167	175	183	201
Vol. O ₂ , cc	42	42	41	45	43	42	45	85	89	91	98
Total Vol. Gas, cc	125	123	123	134	126	125	131	252	264	274	299
Ratio Vol. H ₂ /Vol. O ₂	2.0	1.9	2.0	2.0	1.9	2.0	1.9	2.0	2.0	2.0	2.1
Number Coulombs	707	707	695	793	725	730	768	1480	1550	1610	1800
Current Density, ma/cm ²	20.9	37.3	52.2	65.6	82.1	99.5	120	142	168	182	300
Calc. Vol. H ₂ , cc	82	82	81	92	84	85	89	172	180	187	208
Calc. Vol. O ₂ , cc	41	41	40	46	42	42.5	45	86	90	94	105
Total Calc. Vol. Gas, cc	123	123	121	138	126	127.5	134	258	270	281	313
% Deviation Total Actual Gas to Total Calc. Gas	+1.6	0	+1.7	-3.0	0	-2.0	-2.2	-2.3	-2.1	-2.5	-1.3

RUN #13

Monel separator not in system; using high density porous polyethylene, type 3011 material as separating diaphragm
 Electrode spacing; 1/4"

Test	1	2	3	4	5	6	7	8	9	10	11	12
E, Volts	2.41	2.86	2.92	3.08	3.30	3.35	3.55	3.62	3.75	3.85	3.95	4.10
I, Amps	1.00	2.00	2.48	3.05	3.52	4.00	4.50	5.01	5.50	6.03	7.04	8.01
Time, Minutes	50	30	25	21	18	30	20	24	22	19.5	17	15
Vol. H ₂ , cc	354	421	428	442	435	825	643	828	834	806	820	828
Vol. O ₂ , cc	177	217	222	226	226	419	328	425	434	408	412	422
Total Vol. Gas, cc	531	638	650	668	661	1244	971	1253	1268	1214	1332	1250
Ratio Vol. H ₂ /Vol. O ₂	2.0	1.9	1.9	2.0	1.9	2.0	2.0	1.9	1.9	2.0	2.0	2.0
Number Coulombs	3,000	3,600	3,720	3,850	3,810	7,200	5,410	7,220	7,270	7,060	7,190	7,210
Current Density, ma/cm ²	149	299	370	455	526	598	670	748	822	900	1050	1200
Calc. Vol. H ₂ , cc	348	418	432	446	442	835	628	837	845	820	834	836
Calc. Vol. O ₂ , cc	175	210	216	224	222	419	315	420	423	411	418	420
Total Calc. Vol. Gas, cc	523	628	648	670	664	1254	943	1257	1268	1231	1252	1256
% Deviation Total Actual Gas to Total Calc. Gas	+1.5	+1.6	+0.3	-0.3	-0.5	-0.8	+3.0	-0.25	0	-1.4	-1.3	-0.5

3.2 TASK III-B - Chemical Methods of Hydrogen Generation

- 3.2.1 Using the batch hydrogen generator described in Report #5, tests were run using CaH_2 and water to generate about 12 watt hours of hydrogen. To refresh the reader's memory, the laboratory generator was a cylinder separated into two chambers to store water above the CaH_2 with a plastic separator which could be pierced by a plunger to let the water run into the hydride chamber.

By using a simple "spike" plunger, the generator gave good yields of hydrogen but the plunger had to be pulled back, away from the separator, to allow reactant mixing. A different plunger with a large cutting head containing crossed knife edges allowed the generator to function properly, giving 90% hydrogen yield in 20 minutes of operation. The gas was expanded into a thin rubber weather balloon. The generator, then, is a simple device to be used with small size fuel cell units where complicated controls or high pressure gas bottles and regulators would be troublesome.

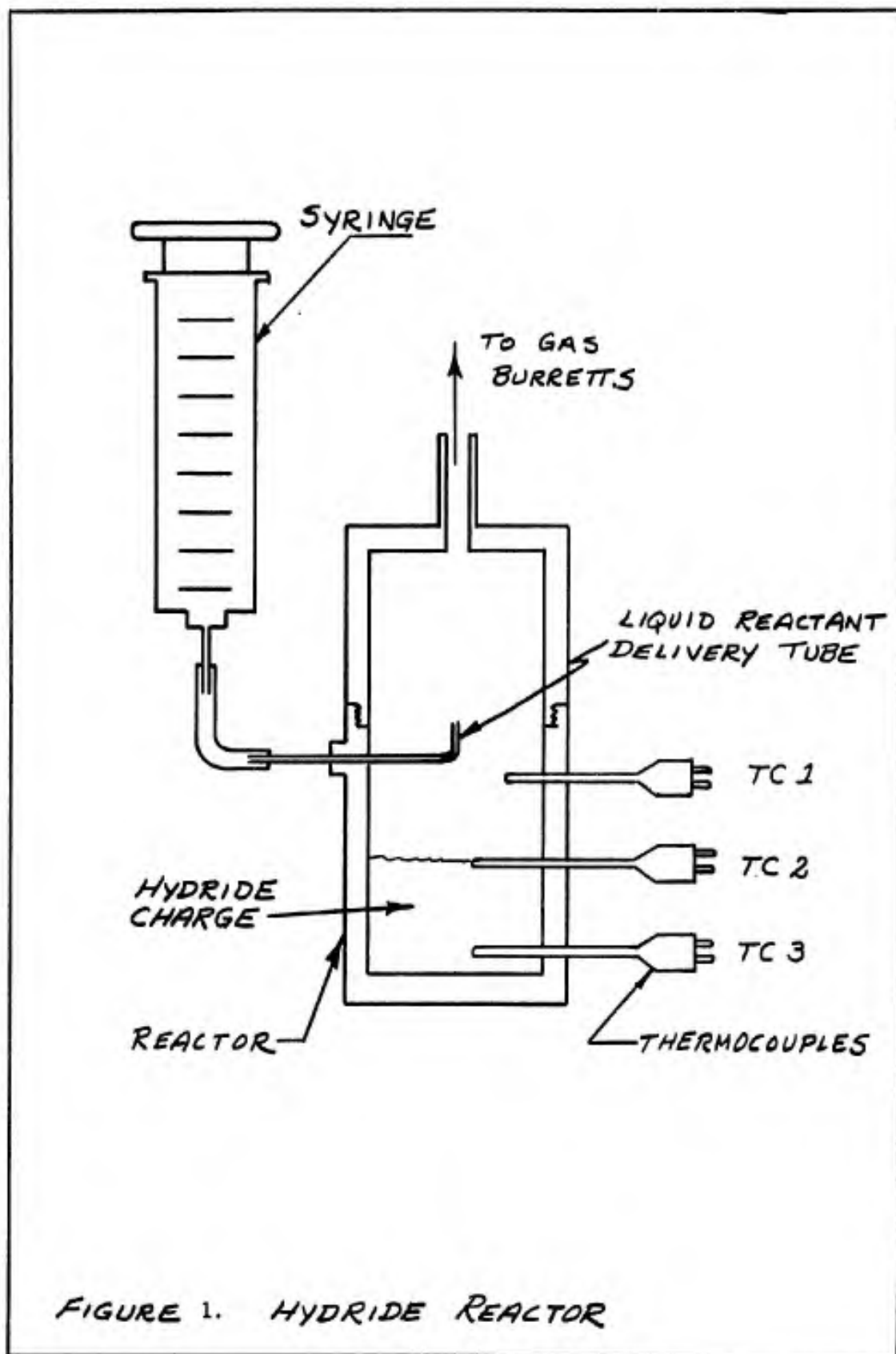
- 3.2.2 An experimental study was made to get an idea of the behavior of various systems containing solid reactants in a cylindrical type container, with liquid reactants simply poured on the solids at a low rate for relatively long periods of time. This would somewhat simulate a generator mission and given an idea of the problems arising in the masking of reactive solids by reaction products. Three systems were studied:

1. $\text{CaH}_2 + \text{H}_2\text{O}$
2. $\text{CaH}_2 + 35\% \text{H}_2\text{SO}_4$
3. $\text{NaBH}_4 + 35\% \text{H}_2\text{SO}_4$

The apparatus used consisted of the hydrogen generator, modified so that liquid introduction could be controlled by a syringe, and also for the purpose of measuring the temperatures. A diagram showing the apparatus is shown in Figure I, Page 16.

The following controls and conditions existed in the testing:

1. Tests were run with a constant volume charge of solid reactants, since masking would be dependent on the geometry of the charge.
2. The granule size of the solid reactants was held constant at about 1/16 inch.
3. Total liquid reactants used per test was 200% of the theoretical required.



4. The liquid reactants were added in 5 cc portions every ten minutes.
5. The liquid reactants were added to the solids by gravity feed, with no provision made for distribution of the liquid throughout the solids. This was done to get a clearer comparison of the distribution problem with the various systems.
6. Tests were allowed to terminate by themselves as evidenced by the lack of reaction rate.
7. Temperature traces were made from copper - constantan shielded thermocouples (1/16" dia.) placed in the solid reactant, and in the gas space above it.

Table I, Page 18, gives the results of the tests. One sees that $\text{CaH}_2 + \text{H}_2\text{SO}_4$ gives relatively low yields, probably caused by the masking of the reactants by the reaction product, which is CaSO_4 (Plaster of Paris). The best system seems to be $\text{CaH}_2 + \text{H}_2\text{O}$. The reaction product, Ca(OH)_2 (lime), does not impede further reaction of the unreacted CaH_2 in the small sized generator.

TABLE I

Comparison of Various Hydrogen Generation Chemical Systems with Stepwise Addition of Liquid Reactants

System	% Theoretical Yield of H ₂	Length of Test (min.)	Solid Expansion Ratio	Temperatures					
				TC#1 (min.)	TC#2 (min.)	TC#3 (min.)	TC#1 (min.)	TC#2 (min.)	TC#3 (min.)
CaH ₂ + 35% H ₂ SO ₄	62%	95	2	307°F	2	207°F	10	517°F	10
NaBH ₄ + 35% H ₂ SO ₄	67%	73	4	146°F	2	170°F	10	146°F	30
CaH ₂ + H ₂ O	100%	90	2	152°F	2	344°F	2	356°F	30

- 3.2.3 Temperature traces show that much higher gas temperatures and reaction temperatures are associated with the CaH_2 reaction. With the CaH_2 reaction, the maximum temperatures were found in the bottom of the chamber which indicates a local reaction with a limited amount of water in that region.

The volume of the charge was measured before running the tests, and the volume of the solid reaction product measured at the end of the tests. The reaction products were allowed to expand, unconfined, except for the cylindrical wall of the reactor and its bottom. The ratio of final to initial volume gives the "expansion ratio" as shown in Table I, Page 18. Calcium hydride expands approximately twice its original volume, whereas sodium borohydride expands about four to one.

- 3.2.4 A final study was made on the $\text{CaH}_2 - \text{H}_2\text{O}$ system to determine the effect of particle size on rates of hydrogen evolution and hydrogen gas yields. The range of particle size ran from 1/16 in. to 325 mesh powder. The water was added in 5 cc portions every five minutes, and an average rate of hydrogen gas was determined over the five minute interval. Figure II, Page 20, shows the results of the tests. With particles ranging from 0.010 to 0.060 inch, the yields are essentially the same. With 325 mesh CaH_2 powder, trouble was experienced in grinding and sifting the hydride which was done in ambient air. The powdered hydride produced was only about 40% reactive; 60% probably reacted with the moisture in the air during processing.

- 3.2.5 Table II, Page 21, shows the rates of reaction of hydride averaged over five minute intervals. It can be seen that the rates are of the same order of magnitude for all sizes of particles studied. It must be remembered that these rates are a function of the hydride geometry as well as the geometry of the hydride charge. Higher rates are most likely possible by "shaping" the hydride charge to allow more intimate contact of the solid and liquid reactants.

FIGURE II - PERFORMANCE OF CALCIUM HYDRIDE AND WATER SYSTEM FOR VARIOUS SIZES OF HYDRIDE GRANULES

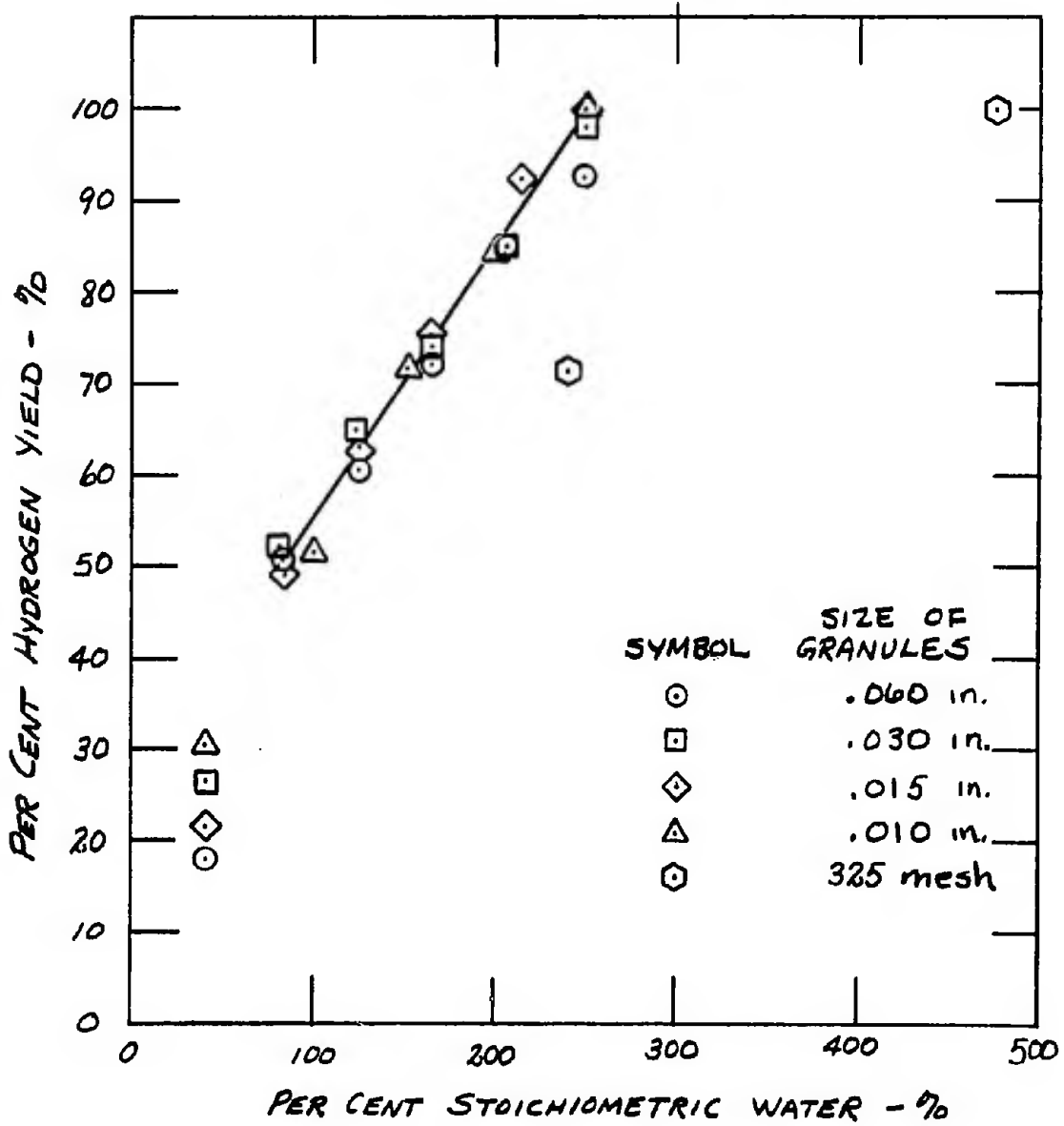


TABLE II

Results of Testing $\text{CaH}_2 + \text{H}_2\text{O}$ with CaH_2 Granules of Various Sizes.

Test Number: 56

Weight of $\text{CaH}_2 = 14.18$ grams

Theoretical $\text{H}_2 = 0.556 \text{ ft}^3$ (STP)

Size of granules = 0.060 in. - nominal

<u>Time</u> <u>min.</u>	<u>Total Vol.</u> <u>H_2O</u> <u>added</u> <u>ml.</u>	<u>%</u> <u>Stoich</u> <u>H_2O</u> <u>%</u>	<u>Volume</u> <u>H_2</u> <u>evolved</u> <u>ft^3</u>	<u>% of theo-</u> <u>retical</u> <u>yield</u> <u>%</u>	<u>Rate of</u> <u>H_2 evolu-</u> <u>tion</u> <u>$\text{ft}^3/\text{min.}$</u>
5	5	41	.100	18.0	.020
10	10	83	.282	50.7	.036
15	15	124	.336	60.5	.011
20	20	165	.401	72.1	.015
30	25	206	.473	85.0	.007
35	30	248	.515	92.6	.010

Test Number: 57

Weight of $\text{CaH}_2 = 14.20$ grams

Theoretical $\text{H}_2 = 0.560 \text{ ft}^3$ (STP)

Size of granules = 0.030 in. - nominal

5	5	41	.143	26.6	.029
10	10	83	.292	52.1	.030
15	15	123	.362	64.7	.030
20	20	164	.416	74.4	.011
25	25	206	.490	87.5	.017
30	25	206	.505	90.2	.003
35	30	248	.546	97.5	.008

TABLE II (Cont'd)

Results of Testing $\text{CaH}_2 + \text{H}_2\text{O}$ with CaH_2 Granules of Various Sizes

Test Number: 58

Weight of $\text{CaH}_2 = 14.08$ grams

Theoretical $\text{H}_2 = .540 \text{ ft}^3$ (STP)

Size of Granules = .015 in. nominal

Time min.	Total Vol. H_2O added ml.	% Stoich H_2O %	Volume H_2 evolved ft^3	% of theo- retical yield %	Rate of H_2 evolu- tion $\text{ft}^3/\text{min.}$
5	5	41	.117	21.7	.023
10	10	83	.264	49.0	.029
15	15	124	.338	62.6	.015
20	20	165	.409	75.7	.014
25	26	214	.500	92.6	.018
30	26	214	.515	95.5	.003
35	30	248	.534	98.8	.004
45	30	248	.540	100	.001

Test Number: 59

Weight of $\text{CaH}_2 = 6.472$ grams 42% active hydride

Theoretical H_2 yield = .102

Size of granules = 325 mesh (powder)

5	5.6	238	.0735	72	.015
10	11.1	476	.102	100	.006

Test Number: 60

Weight of $\text{CaH}_2 = 12.30$ grams 92% active hydride

Theoretical H_2 yield = .430 ft^3

Size of granules = .0165 to .0035 in. nominal

5	5	41	.131	30.5	.026
10	10.5	1.00	.221	51.4	.018
15	16	152	.307	71.4	.017
20	21	200	.362	84.2	.011
25	26	248	.420	97.7	.012
30	26	248	.430	100	.002