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NO. 078

**FINAL REPORT**

**LOW TEMPERATURE FUEL CELL BATTERY**

**U. S. ARMY  
ELECTRONICS RESEARCH AND DEVELOPMENT LABORATORY  
FORT MONMOUTH, NEW JERSEY**

**CONTRACT NO. DA-36-039-SC-78367  
TASK NO. 3A-99-09-002-03  
1 July 1960 - 28 February 1961**

**UNION CARBIDE CONSUMER PRODUCTS COMPANY  
PARMA RESEARCH CENTER  
PARMA, OHIO  
ELECTROCHEMICAL PRODUCT DEVELOPMENT LABORATORY  
CLEVELAND, OHIO**

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UNION  
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**LOW TEMPERATURE FUEL CELL BATTERY**

**FINAL REPORT**

for the period

1 July 1960 - 28 February 1961

Prepared by E. A. Schumacher and G. T. Jobe

U. S. ARMY  
ELECTRONICS RESEARCH AND DEVELOPMENT LABORATORY  
FORT MONMOUTH, NEW JERSEY

CONTRACT NO. DA-36-039-SC-78367  
TASK NO. 3A-99-09-002-03

The object of this program was to carry on investigations leading to the fabrication of a 28-volt, 200-watt fuel cell battery operating on hydrogen and air with an over-all package weight not to exceed 35 pounds, including mechanical auxiliaries, and fuel for 24 hours' operation.

UNION CARBIDE CONSUMER PRODUCTS COMPANY  
PARMA RESEARCH CENTER  
PARMA, OHIO

ELECTROCHEMICAL PRODUCT DEVELOPMENT LABORATORY  
CLEVELAND, OHIO

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## LOW TEMPERATURE FUEL CELL BATTERY

### I. Purpose

To carry on investigations leading to the fabrication of a 28-volt, 200-watt fuel cell battery operating on hydrogen and air with an over-all package weight not to exceed 35 pounds, including mechanical auxiliaries, and fuel for 24 hours' service. A life of 1000 hours was established as the desired service target for the battery. The investigation covered study of fuel sources adapted to meet the service and weight requirement, and means for generation of the fuel gas.

### II. Abstract

The following approach to design feature studies and laboratory investigations was followed to meet the technical requirements of the contract:

1. Carbon thickness was minimized and battery construction features revised with the prime objective of developing a 35-pound battery system including mechanical auxiliaries and fuel generating facilities.
2. The problem of air diffusion was investigated in relation to cathode structure and other factors which influence battery operation with air under various ambient conditions.
3. Battery operation at elevated temperatures was examined to achieve maximum effectiveness with respect to stability, voltage, and water vapor removal.
4. Electrolyte composition and concentration were studied to achieve highest operating voltages consistent with stability, and water vapor removal.
5. Portable hydrogen sources capable of operating a battery for 24-hour periods were subjected to laboratory tests. Consideration was given to fuel sources in the form of solids, liquids and compressed gas, meeting weight, cost, hazard, portability and control criteria.

The engineering studies falling within the scope of the contract requirements showed that the conventional baked-carbon electrodes with the thinnest dimension compatible with structural soundness would not reproduce with air depolarization the excellent performance consistently obtained with oxygen. Under these conditions it was not possible to meet the current density demand imposed by the 35-pound battery weight stipulation. These determinations were conveyed to the U. S. Army Electronics Materiel Agency and agreement reached that since the prime objective of the contract was not within the state-of-the-art at that time, contract termination was in order to conserve residual funds.

Concurrent work by the Research Laboratory of the Union Carbide Consumer Products Company during the later stage of the contract period was concerned with new electrode fabrication techniques which offered large reduction in weight and volume as well as the possibility of easier operation through simplification of by-product water removal. This work was called to the attention of the U. S. Army Electronics Materiel Agency and subsequently incorporated in a new proposal for the development of a lightweight, thin electrode making the 35-pound battery weight a sound technical goal.

### III. Publications, Lectures, Conferences

#### A. Publications

H. W. Holland, "Carbon Electrode Fuel Cell Battery," Proceedings, Fifteenth Annual Power Sources Conference (1961).

#### B. Conferences

1. Meeting at Fort Monmouth, 4 August 1960. Purpose: to discuss approach to technical problems; procedures; technical areas requiring clarification; arrangements for shift in contract starting date.

2. Visit by Mr. J. Lyons, 8 December 1960. Purpose: for reorientation and review of progress; revision of report numbering; inclusion of "work accomplished statement" in reports.

3. Meeting of personnel from U. S. Army Electronics Research and Development Laboratory (USAELRDL) and Union Carbide Consumer Products Company (UCCPC), 7 April 1961, at Fort Monmouth. Purpose: to discuss status of project and possible revision of objectives.

a) Gas generation - to discontinue work on hydrides; examine other sources.

b) Weight problem - best foreseeable figure with baked-carbon electrodes well above 35-pound requirement; other electrode configurations indicated.

c) Contract time extension requested without additional funding to permit study of new Research Laboratory approach to lightweight battery.

4. Meeting of USAELRDL and UCCPC personnel, 18 July 1961, at Fort Monmouth. Purpose: to outline UCCPC's technical position on the contract and to discuss specifically:

a) Termination of expenditures on contract.

b) Continuation of work by UCCPC on new electrodes.

c) Specifications for proposed bench test battery.

5. Meeting of USAELRDL and UCCPC personnel, 2 February 1962, at Fort Monmouth. Purpose: to outline contract status.

a) Complete review of UCCPC's state-of-the-art; advised that the battery weight specifications of Technical Requirements No. SCL 753 could not be met with the baked-carbon electrode.

b) The battery weight requirement is a practical objective based on UCCPC's research progress on "fixed-zone" electrodes.

c) Discuss preparation of a Technical and Cost Proposal covering research and development work on "fixed-zone" electrodes.

6. Meeting of USAELRDL and UCCPC personnel, 17 May 1962, at Fort Monmouth. Purpose: summation of contract studies and outline of UCCPC's research progress in the general area of lightweight fuel cell batteries. Proposal made to utilize residual contract funds for the construction of a bench type battery for laboratory tests.

\* \* \*

#### IV. Battery Development

##### A. Basic Design Concept of UNION CARBIDE Fuel Cell Battery

The first laboratory models of the Union Carbide hydrogen-oxygen fuel cell battery utilized tubular, carbon electrodes processed to develop active surfaces, wetproofed and catalyzed to obtain acceptable hydrogen and oxygen potentials.\* These were quite effective for preliminary laboratory examination of process and operating parameters and a number of such unit cells are still in operation after 12,000-18,000 hours at discharge rates of the order of 10-20 amperes per square foot. Under single electrode test conditions to establish current:voltage relationships, stable voltages were obtained with such electrodes for current densities up to 500-1000 amperes per square foot. However, the problems of assembling tubes into large multicell batteries were sufficiently onerous to require investigation of more practical methods of battery fabrication. Figure 1 illustrates double and multi-tube electrode arrangements.

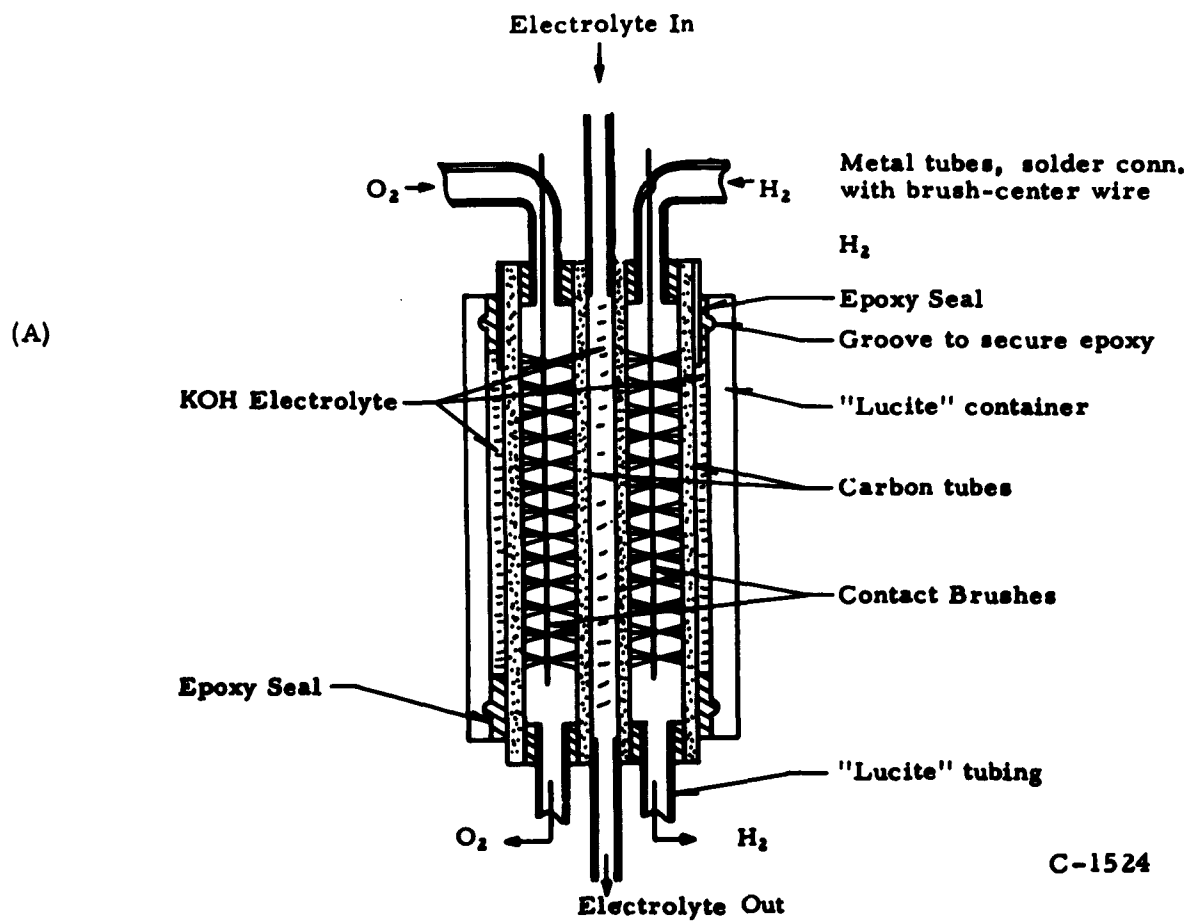
The simpler concept of construction, involving bipolar, flat plate carbon electrodes, was introduced soon thereafter, eliminating the difficult electrical harnessing of multi-tube cells and problems of gas distribution. The present plate-type electrodes are now used to achieve maximum weight and space economy and to obtain low resistance intercell electrical connections. With battery design generally pointed to use of gas pressures below 1 psig, a leading design feature for the battery components is resistance to attack by strong alkali in moist oxidizing and reducing environment. Because of the tendency of alkaline electrolytes to creep through fissures and capillaries, adequately designed liquid seals are imperative. This feature is of major importance when batteries are subjected to the added hazards of shock and vibration stresses.

The electrodes are generally mounted in plastic or rubber frames either by injection molding, or through use of special sealing compounds to effect bonding between electrodes and separately molded frames. Distribution of the hydrogen, oxygen or air and electrolyte is obtained by through-ports molded as integral parts of the frames to serve as main laterals and

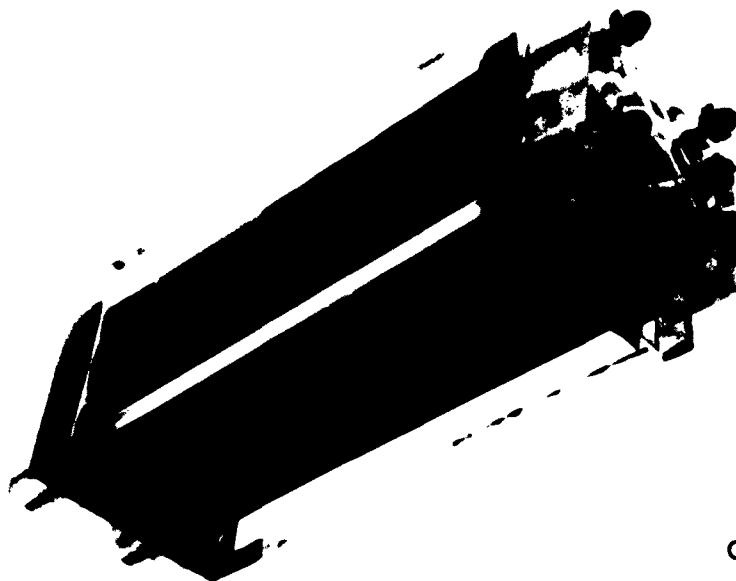
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\*G. E. Evans, Proceedings, 13th Annual Power Sources Conf., p 109 (1959)

**FIGURE 1**  
**CONSTRUCTION WITH TUBULAR ELECTRODES**  
**(A) DOUBLE AND (B) MULTI-TUBE ARRANGEMENTS**



(B)



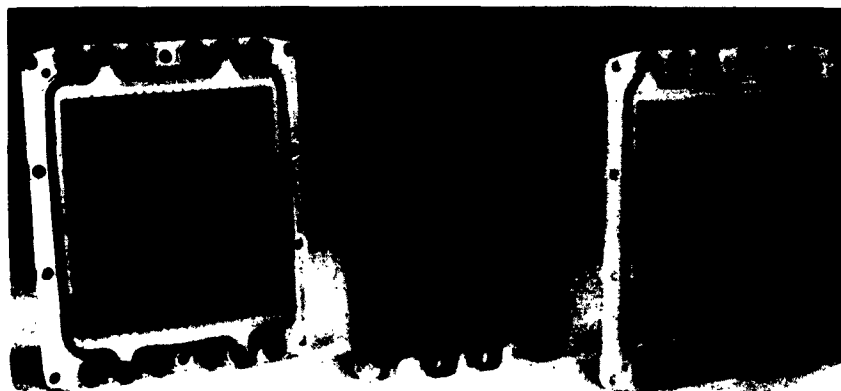
through molded-in branch lines leading to the appropriate gas and electrolyte cavities. The intercell electrical connection is made via metal gas barriers separating the two gases with conducting means to bridge the barrier-electrode gaps constituting the gas cavities.

Figure 2 shows both sides of one electrode in its frame. In this case, a liquid-tight peripheral seal is obtained by injection-molding a plastic

FIGURE 2

FRAMED CARBON ELECTRODES

- (a) Electrolyte Side
- (b) Gas Side



(a)

barrier

(b)

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compound around the carbon plate. In (a), the electrolyte side is shown with flow-distributing channels molded at both top and bottom. The two through-ports at each end of the frame provide flow of electrolyte through a battery in the direction perpendicular to the working surfaces of the electrodes. Neoprene "O" rings provide seals around each port, and a large "O" ring surrounds the entire liquid space of the cell. In assembly, a matching electrode of opposite polarity is placed face down against the electrode shown, forming a cell except for the gas supplies. In (b), the gas

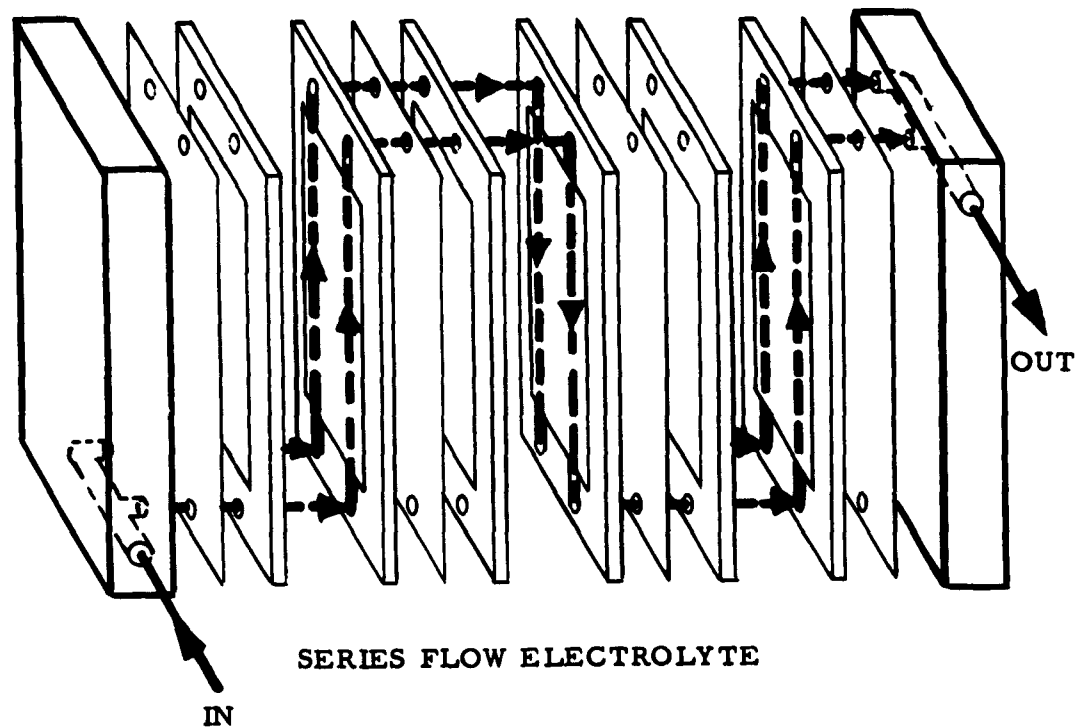
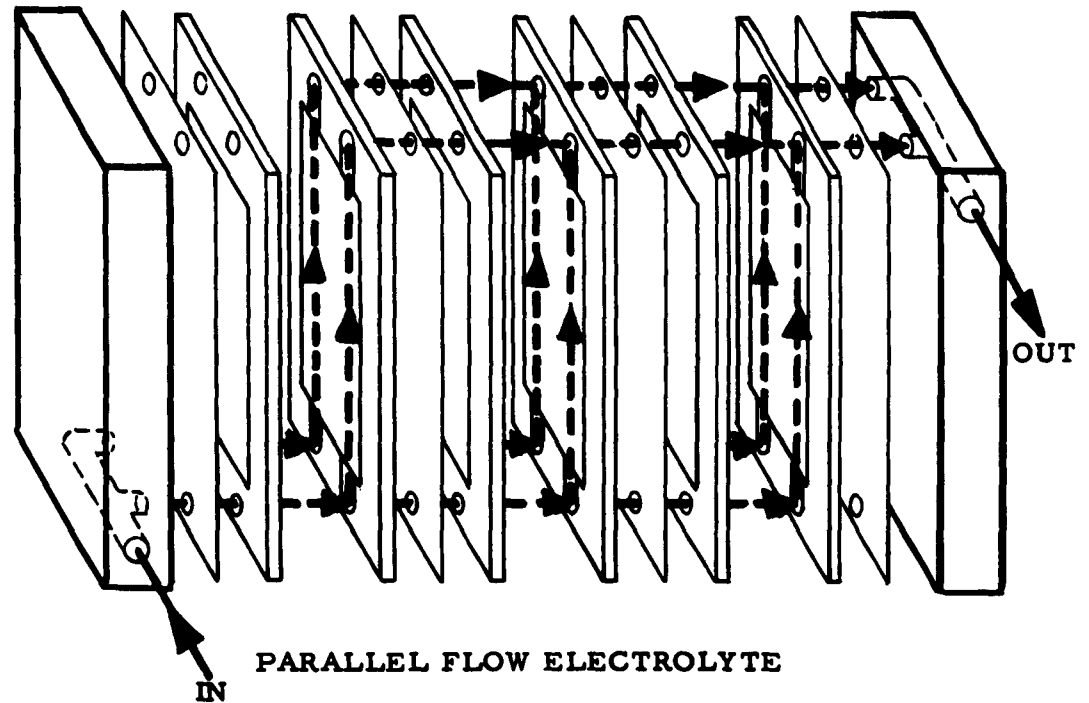
side of an electrode is shown. An arrangement similar to that just described is used to lead the gas into and out of the space defined by the large 'O' ring. After assembly, the gas side of another electrode faces the electrode shown but with a thin metal sheet placed between the two. The metal sheet serves as a gas barrier to separate fuel gas and oxidant and also acts to conduct current between the cells. In the configuration shown in Figure 2, the metal plate has been corrugated to serve, in addition, as the electrode contact.

When the framed electrodes of Figure 2 are assembled into batteries the fluid passages can be arranged to provide either parallel or series flow of solution as Figure 3 illustrates. Parallel flow minimizes pressure drop in the fluid system, avoids gas entrapment, and with proper porting design ensures uniform temperature and fluid distribution. A similar arrangement of the gas passages (not shown) provides parallel feed of hydrogen and oxygen.

Under some conditions of operation, series flow of electrolyte and gas is advantageous because the same fluid velocity through each cell is ensured. Series flow of electrolyte has the disadvantage of supplying more dilute electrolyte to downstream cells, since reaction product water can appear at least partly in the electrolyte. Variation in temperature and pressure head throughout an array of cells are added problems when using series electrolyte flow.

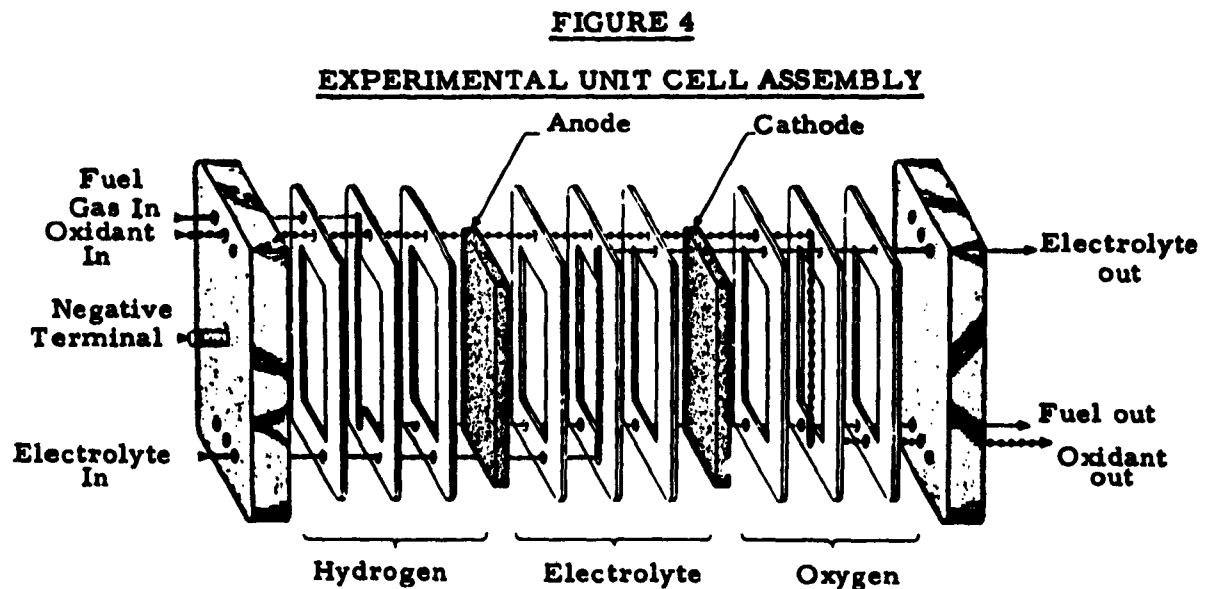
The decision for parallel or series flow thus depends upon the number of cells in series, the battery current, the power assigned to pumping, and the tolerance of the electrodes to differences in electrolyte concentration, temperature and pressure. Series flow of the gases presents other problems, for the presence of water vapor in varying amounts in the gas cavities alters the partial pressure of the reacting gases. In either mode of operation, condensation of moisture in gas passages must be avoided for water clogged ports can interrupt gas flow to one or more cells. Such problems are minimized by a choice of operating conditions to maintain the gases safely above their dew points. For the present contract work the parallel arrangement of fluid flow was used.

**FIGURE 3**  
**SCHEMATIC DRAWING OF FUEL CELL BATTERIES ILLUSTRATING**  
**PARALLEL AND SERIES FLOW OF ELECTROLYTE**



Investigation of the electrical contact problem continued through much of the contract studies. Although total resistance through the thickness of the carbon electrodes is quite small, occasional uneven current distribution over the electrode surfaces observed in the early stages of the contract studies resulted in localized higher current density areas. These were subject therefore to higher polarization and over long periods appeared to show more rapid penetration of electrolyte into the electrode pore structure. It should be noted that the carbon electrodes now available are quite uniform and do not change voltage significantly over a fairly wide range of current density, as discussed in a later section of this report. With the improvement in electrode technology established over the past several years, e. g., uniform activation, flatness, better electrical contact methods, the problem of contact variability apparently no longer exists.

Variations of the bipolar construction were developed by substitution of other framing procedures to simplify the assembly of experimental cells. This approach was used in the present contract program as a means of expediting the testing of a variety of experimental electrodes preliminary to the assembly of batteries.



Note: For simplicity, electrical connections and gaskets surrounding edges of electrodes not shown. C-1528

## B. Specific Design Considerations

### 1. Weight Calculations

Early weight calculations postulated on satisfactory air operation at 35-50 ASF current density with available 0.235 inch carbon stock led to the following estimated values for a 35 cell, 28 volt, 200 watt battery.

TABLE I  
BATTERY WEIGHTS VS CURRENT DENSITY  
(0.235" carbonelectrode)

|                                   | 35 ASF    | 50 ASF <sup>1</sup> | 70 ASF <sup>2</sup> |
|-----------------------------------|-----------|---------------------|---------------------|
|                                   | lbs       | lbs                 | lbs                 |
| Battery and Electrolyte           | 60        | 42                  | 30                  |
| Accessory Components              | 12        | 12                  | 12                  |
| Fuel System and Fuel <sup>3</sup> | <u>14</u> | <u>14</u>           | <u>14</u>           |
| Total Plant                       | 86        | 68                  | 56                  |

<sup>1</sup> Operation at 50 ASF for periods of 400-500 hours has been achieved by moderate temperature elevation.

<sup>2</sup> This current density has not yet been validated for air operation.

<sup>3</sup> Represents weight of compressed hydrogen and lightweight cylinder for 14 hrs operation.

From this analysis it was evident that several avenues should be pursued to achieve the desired goal of a complete battery plant weighing not in excess of 35 pounds:

- a) To reduce the electrodes well below the regular 0.235 inch thickness;
- b) To improve performance with air, and
- c) To reduce the weight of fuel, fuel tank and other mechanical components.

Experimental work had indicated that the state-of-the-art electrodes at the time of contract initiation, might be reduced in thickness to about 1/8". However, for the easily assembled type of construction envisaged for the battery, this reduction in electrode thickness was not sufficient to achieve

the desired weight. Using baked 1/8" electrodes as the basis for further weight estimating, the following data were established for battery operation on air and hydrogen at 35-40°C.

**TABLE II**  
**BATTERY WEIGHTS VS CURRENT DENSITY**  
(0.125 inch carbon electrode)

|                                   | 35 ASF    | 50 ASF    | 70 ASF <sup>1</sup> |
|-----------------------------------|-----------|-----------|---------------------|
|                                   | lbs       | lbs       | lbs                 |
| Battery and Electrolyte           | 35        | 24        | 15                  |
| Accessory Components              | 12        | 12        | 12                  |
| Fuel System and Fuel <sup>2</sup> | <u>14</u> | <u>14</u> | <u>14</u>           |
| Total Plant Weight                | 61        | 50        | 41                  |

<sup>1</sup>This current density has not yet been validated for air operation.

<sup>2</sup>Represents weight of compressed hydrogen and lightweight cylinder for 14 hrs operation.

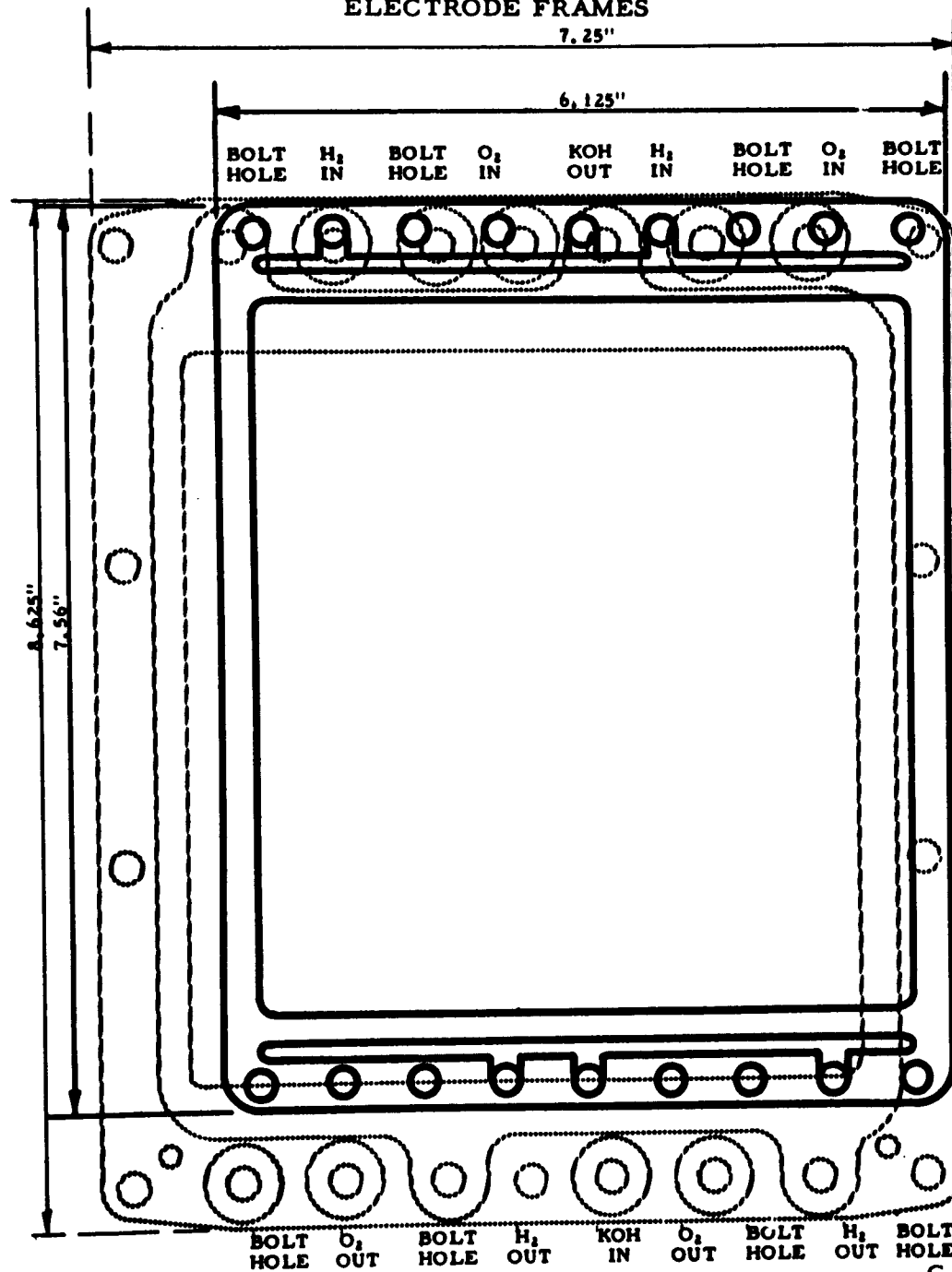
At 35 amperes per square foot, the total battery using 1/8" baked carbon electrodes, will be roughly 175 per cent in excess of the required 35 pound upper weight limit. By extrapolation, a current density of about 100 ASF at elevated temperature and significantly improved air electrodes would be necessary to achieve the weight target. It is evident also that a more intensive engineering study of the mechanical components and fuel generation means is needed since at 100 ASF these would represent approximately 75 per cent of the 35 pound limit.

## 2. Design Studies

Subsequent design studies showed that a moderate reduction in weight was available through frame redesign and end plate design revision. However, the over-all weight economy would still be insufficient to bring the total battery plant within the desired weight limit, without coincident upgrading in performance; i. e., unless operability were raised to 75-80 ASF operation with air. The frame redesign resulted in reduction in the frame dimensions from 8-5/8" length down to 7-1/2" and 7-1/4" width down to 6-1/8". The regular and revised new frames are shown in Figure 5.

FIGURE 5

COMPARISON RIGID PLASTIC AND ELASTOMERIC  
ELECTRODE FRAMES



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These changes were predicated on two assumptions:

a) That the "O" rings could be eliminated through substitution of elastomeric materials (rubber, neoprene, polypropylene, etc.) for rigid plastics for frame fabrication, and

b) Omission of side tie bolts since the battery would be used at atmospheric pressure.

Elimination of the "O" rings required only that the frame could be molded of material having sufficient elasticity to permit stretching over the electrodes and subsequent sealing to the electrodes with a sealant pre-applied to the electrode edges. Both features were tested in module type units with every evidence of successful applicability to larger battery construction. The neoprene frames permitted flexing during battery assembly so that the cell to cell joints remained tight against electrolyte and gas leakage and showed no distortion of the normal fluid passageways.

With no reduction in the dimensions of the gas and electrolyte gaps, the thickness of a half-cell (single framed electrode) in the revised frames was 0.203 inch, equivalent to 0.406 inch for a complete cell as compared with 0.625 inch for the usual cell. The volume of the conventional and new cell designs thus became 39 inches<sup>3</sup> and 18.6 inches<sup>3</sup>, respectively, amounting to a reduction in gross volume of 52 per cent. The corresponding weight reduction of the new frames is estimated to be about 35-40 per cent, equivalent to nearly 2 pounds of the weight of an activated battery operating at 35 ASF. Figure 6 compares two batteries of equivalent output: "A" utilizes the 0.125" electrodes with the revised frame construction and "B" utilizes the conventional injection molded plastic frame with 0.235" thick carbon electrodes.

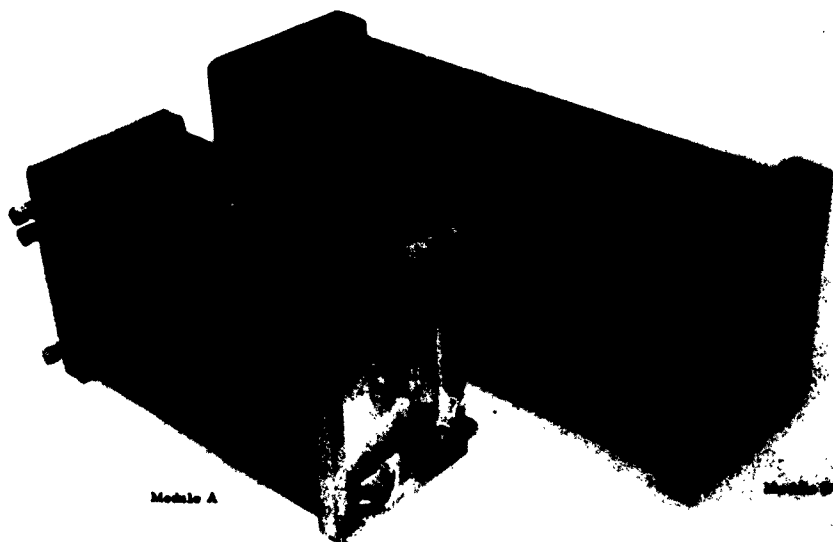
### 3. Advanced Design

Another type of battery construction, developed at a later stage of Union Carbide sponsored research on the general problem of electrode fabrication and battery assembly, involves cast type plastic housings which permit use of electrodes much thinner than the conventional 1/8" baked carbons. These thin "fixed-zone" electrodes consist of several layers of bonded carbon or metal powders with the electrolyte-facing layer very thin, microporous and completely wetted, and the layer or layers underneath the

FIGURE 6

COMPARISON 32 CELL BATTERIES

- (A) Elastomeric Frame - 1/8" Carbons  
(B) Plastic frame - 1/4" Carbons



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surface material non-wetting, macroporous and highly conducting. The top layer may consist of a catalyzed, active carbon but this is not a necessary restriction and other materials such as catalytic metals may be employed where they offer advantage over carbon. The same broad approach applies to the porous, electrolyte-repellent layers. Plastic-bonded graphite or sintered nickel have been used, but there are no arbitrary limits on materials that can be employed.

As a result of this Union Carbide work, a number of advantages predicted for the "fixed-zone" principle have already been demonstrated:

- a) Use of precatalyzed carbon powders for greater uniformity and catalyst effectiveness;
- b) Thinner electrode structure, i. e., down to 0.011"-0.035";

- c) Improved rates of water vapor transpiration, and
- d) Greater insensitivity to discharge abuse.

Based on this latest development in electrode fabrication and on other major foreseeable improvements in battery design; such as, vapor transpiration of by-product water; elimination of electrolyte circulation; and reaction of chemical hydrides to produce hydrogen which are discussed in later sections of this report, a 35 pound battery system operating at 35 ASF becomes a completely realistic target. The indicated weight would include battery, electrolyte, mechanical auxiliaries, and a fuel system for 24 hours of operation daily.

Application of the same new principles of battery operation to a battery constructed with 0.125 inch thick baked carbon electrodes suggests that current density capability of the order of 70 ASF would be required to meet the 35 pound specification for the total system.

TABLE III

PROJECTED TOTAL PLANT WEIGHT VS CURRENT DENSITY

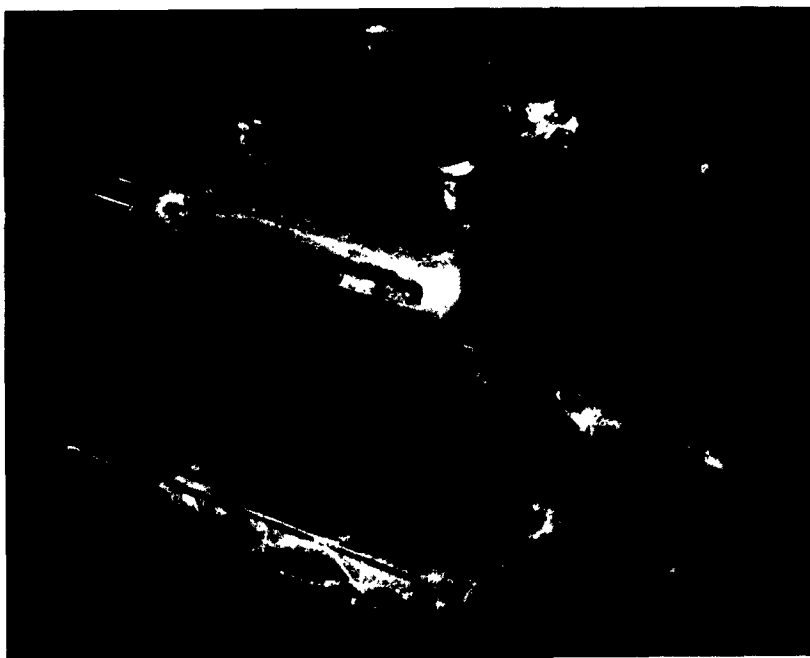
| Current Density ASF | 1/8" Baked Carbon | "Fixed-Zone" Electrode |
|---------------------|-------------------|------------------------|
|                     | <u>lbs</u>        | <u>lbs</u>             |
| 70                  | 35                | 29                     |
| 50                  | 43                | 31                     |
| 35                  | 54                | 35                     |
| 25                  | 69                | 40                     |

Note: Data for both battery types are based on water transpiration elimination of electrolyte circulation, and reaction of battery water vapor with hydride to make hydrogen.

Figure 7 illustrates a Research Laboratory battery featuring the "fixed-zone" electrodes assembled in a cast epoxy housing without the usual plastic or neoprene frames. Ports and channels in the electrolyte and gaseous reactants were "built in" prior to the casting operation.

FIGURE 7

RESEARCH TYPE BATTERY - THIN FLEXIBLE ELECTRODES



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4. Electrolyte and Gas Porting

Electrode frames used in the first experimental cells provided electrolyte entry into the electrolyte cavity by means of two inlets, located at the lower edge of each cavity, and two similar exits at the upper edge. Any maldistribution of electrolyte through the solution cavity resulting from this porting geometry permitted concentration and dilution areas to become established.

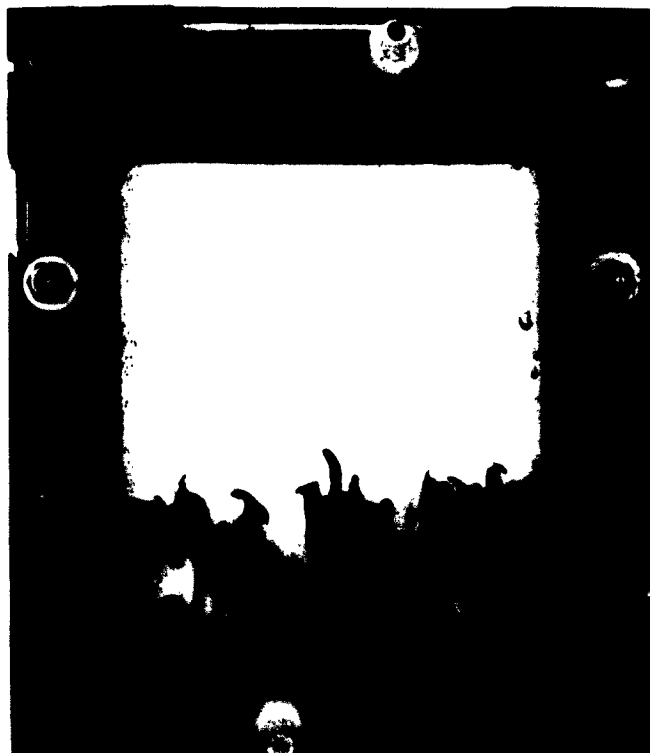
Electrolyte circulation through the electrolyte cavity was determined experimentally by injecting ink or dye into a solution stream introduced between transparent plastic plates simulating the battery electrolyte cavity and having the same inlets and outlets as a standard cell. The distribution pattern, Figure 8 (A) shows two columns of electrolyte rising vertically from inlets to outlets with some slow sideways diffusion, but for battery purposes, an undesirable circulation situation.

FIGURE 8  
SOLUTION FLOW PATTERNS IN ELECTROLYTE GAP

(A)



(B)



C-1532

Figure 8 (B) shows flow through an inlet manifold designed to sweep the entire electrolyte cavity. The improvement is obvious.

Application of the modified entry-exit arrangement in connection with other investigations involving batteries having up to 18 cells in series array showed that electrolyte distribution was good throughout the test as evidenced by measurements of temperature rise in the individual cells. Although the good performance observed for these batteries was the result of better electrode quality as well as efficient electrolyte circulation, the latter undoubtedly made a significant contribution to long life (up to 1700 hours per battery were obtained at 50 ASF continuous operation with oxygen supplied to the cathodes).

Additional improvement in electrolyte circulation is anticipated as a result of another recent design revision in the electrolyte system scheduled for early incorporation in experimental batteries. Accurately machined orifices, positioned in each of the individual branches leading from the main electrolyte manifolds, provide a positive pressure drop between inlet and outlet ports and minimize the effect of dimensional differences occurring between cells as previously fabricated and assembled. Experimental verification of this design feature has been demonstrated on much larger cells than required for SC-78367, and, therefore, are expected to be equally valid for use on this contract.

Similar results have been observed in the study of gas distribution uniformity in the hydrogen and oxygen gas cavities supplying the anodes and cathodes respectively. Redesign of the porting also makes for better gas movement. This may be expected to be of greater significance for the cathode than for the anode in view of the low partial pressure of the oxygen in the air supply and possibility of developing pockets of nitrogen.

##### 5. Parasitic Currents in the Electrolyte System

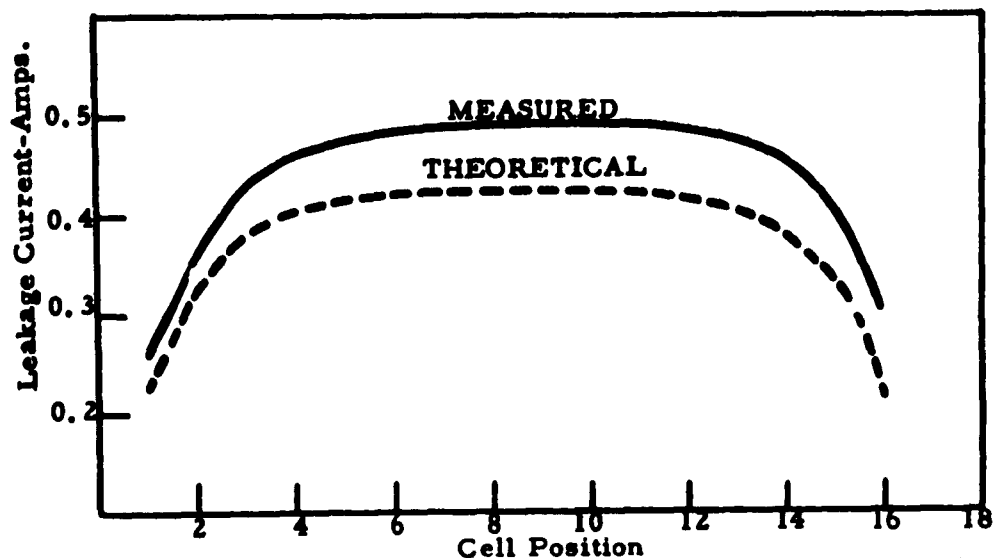
With common electrolyte feed manifolds located along the lower edge of the carbon:frame assemblies to supply the individual cells and a similar arrangement at the upper edge for movement of electrolyte from the cells into a common discharge manifold, an electrolytic path is set up within a battery leading to self-discharge of the individual cells. An analysis

of the leakage currents has been made and mathematical models derived corresponding to the electrochemical and physical parameters for batteries containing up to 40 cells. Computer calculations were confirmed experimentally on a 17-cell, 125 watt, hydrogen-oxygen battery based on plastic framed, nominal 6" x 6" x 1/4" electrodes, and 12 M KOH electrolyte at 60°C.

Comparison of the calculated values and experimental data is given in Figure 9. Theoretical values range from 0.22 ampere up to 0.425 ampere

**FIGURE 9**

**CALCULATED AND EXPERIMENTAL VALUES  
FOR PARASITIC CURRENT**



C-1533

per cell with approximately half of the cells lying in the region of high parasitic current (0.40-0.425 ampere). The measured values are slightly larger, possibly due to the use of elastic "O" rings which do not permit accurate definition of passage cross-sections. Using the experimental values, the average parasitic current amounts to about 0.42 ampere or 3.8 per cent of the load at 50 ASF and proportionately more as the load is reduced. The absolute current density value for the parasitic current is 1.9 ASF for the plastic frame construction.

The magnitude of the internal short is influenced by a number of factors subject to corrective steps which can materially reduce the internal shorting condition. Manifold cross-section contributes most importantly to the magnitude of the bypass current. Reduction in manifold diameter is an immediate approach to correction but must be weighed against the additional power needed to move electrolyte through a more restricted passageway. Other improvements include use of orifices in the feed manifold and longer laterals within each cell before the solution enters the electrolyte cavity. These also will add somewhat to the electrolyte pumping load. Electrolyte concentration change toward higher alkali content cannot be used as an off-setting approach, since less conducting solutions will raise the IR drop through the cells, and perhaps more than overshadow the gains realized by reduction in parasitic current.

In the event that electrolyte circulation requirements can be moderated by special battery design features, as discussed in a later section of this report, for example, the introduction of electrodes to run at temperatures permitting water vapor transpiration into the gas streams, then power for circulation will also be lowered. Such power reduction can be used to offset the effect of smaller electrolyte manifolds.

To eliminate residual parasitic current demand for hydrogen and air during long standby periods, the hydrogen lines to and within the battery are purged with air until potential differences are eliminated between the anodes and cathodes, i. e., until the anodes become cathodic. At this point the gas supplies can be disconnected. Further steps to 'mothball' a battery may include electrolyte removal from the internal cavities. Reactivation then consists simply of a solution refilling operation and flushing the anode gas lines with fresh hydrogen.

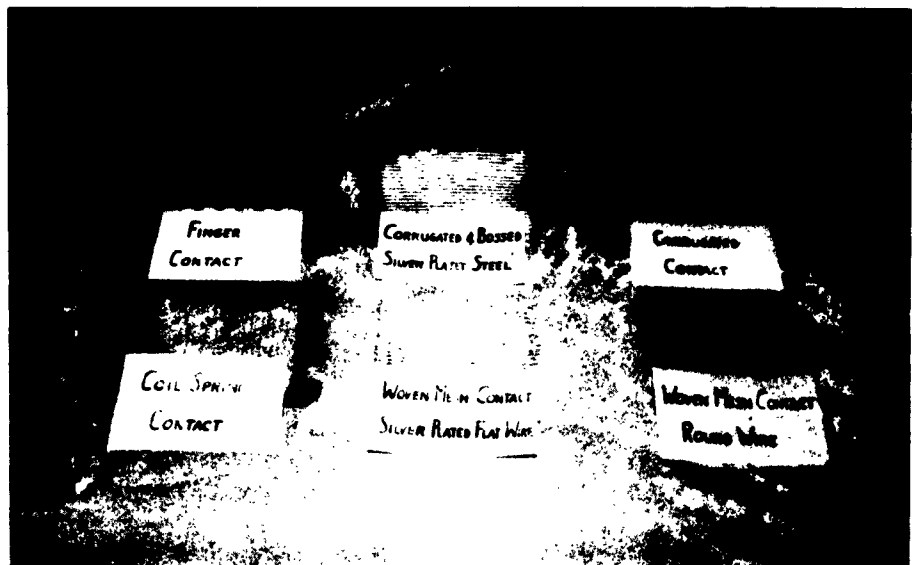
## 6. Electrical Contacts

Electrical contact between the metallic gas barrier and its adjacent anode-cathode pair, as previously stated, was originally achieved by corrugating the metal gas barrier so that when assembled, electrical continuity was established with the contacting carbon faces. Used alone, this contact proved inadequate and nonuniform. However, in combination with

a thin porous metal coating applied to the gas side of the electrodes, a more effective contact was obtained which was quite satisfactory at relatively low current densities. Such porous metal coating may be varied over a fairly broad thickness-range without interference with gas flow. Many metals can be used for this purpose: copper, silver, gold, lead, nickel, etc., but preferably should be of the same species as the metal barrier.

With demand for higher current output from fuel cell batteries, other contact methods were investigated some of which are illustrated in Figure 10. The top row shows two versions of the corrugating treatment

FIGURE 10  
ELECTRICAL CONTACT ELEMENTS FOR ELECTRODES



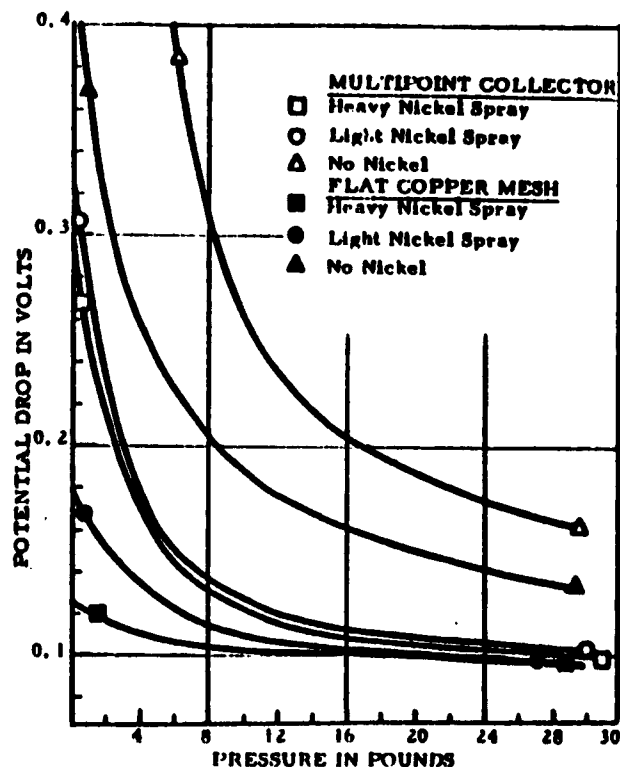
C-1534

embossed corrugations, and silver-plated barrier members were distinct improvements. Pierced sheets having raised spring-like or "finger contacts" were fairly effective. Two such sheets sandwiched an imperforate sheet of metal to retain the gas barrier function. A particularly novel method was achieved with the contact illustrated in the low left corner of Figure 10, "Coil Spring Contact."

Copper strands, either flat or round, and loosely woven to make a metal mesh provide excellent contact and adaptability to variations in gas gap dimension. Plain copper was superseded by silver-plated material to overcome the oxidation effects encountered in moist oxygen and air. Less expensive but equally effective is a related form of mesh made of round nickel wire titled "Woven Mesh Contact-Round Wire" in Figure 10. In conjunction with nickel barrier sheet and nickel spray, it displays outstanding

**FIGURE 11**

**CONTACT RESISTANCE VS. ASSEMBLY PRESSURE-  
TYPICAL CONTACT MEMBERS**



C-1535

resistance to oxidation and prevents formation of electrochemical couples in the event that electrolyte fortuitously finds its way into a gas gap.

Figure 11 illustrates the effect of assembly pressure, collector form and carbon preparation. It is clearly shown that the use of a porous nickel coating on the gas faces is a desirable part of the current distributor element, and that the flat copper mesh is somewhat better than a multipoint collector. With proper nickel coverage of the carbon surface, both collectors show about the same values at 1 psi assembly pressure; however, some advantage is offered for the copper mesh below 0.5 psi. Later work with special nickel mesh contacts demonstrates that these function as good electrically as silver-plated copper, show less deformation tendency under load application, and are considerably lower in cost than the silver-plated meshes.

Figure 12 is a schematic representation of the fuel cell system with experimentally determined values for IR drop at the various interfaces in a cell. It is evident that when properly assembled, the electrode:metal coating:metal mesh combination has an IR drop at 50 ASF far below that of the electrolyte, or the polarization loss at the electrodes. It is evident also that further elevation in cell voltage may be expected from reduction in the electrolyte gap.

## 7. Environmental Testing

The UNION CARBIDE Fuel Cell battery with baked-carbon electrodes has been subjected to a number of dynamic environmental conditions with gratifying evidence of stability in all aspects of these tests.

### a) Shock

A unit cell comprising plastic-framed, nominal 6" x 6" x 1/4" electrodes with standard gas barriers, metal mesh contactors, neoprene "O" ring seals and electrolyte was mounted in a Barry Corporation Component Part Shock Machine - Type 20 VI. The electrolyte head was 14 inches and gas pressure 1/2 psi. The load on the cell was held at 11 amperes throughout three successive shock tests of 40 G for 6 milliseconds, 100 G for 3.5 milliseconds and 200 G for 5 milliseconds. The current was maintained for 4-minute periods while each shock level was attained, and

FIGURE 12

SCHEMATIC OF RESISTANCE VALUES IN CELL

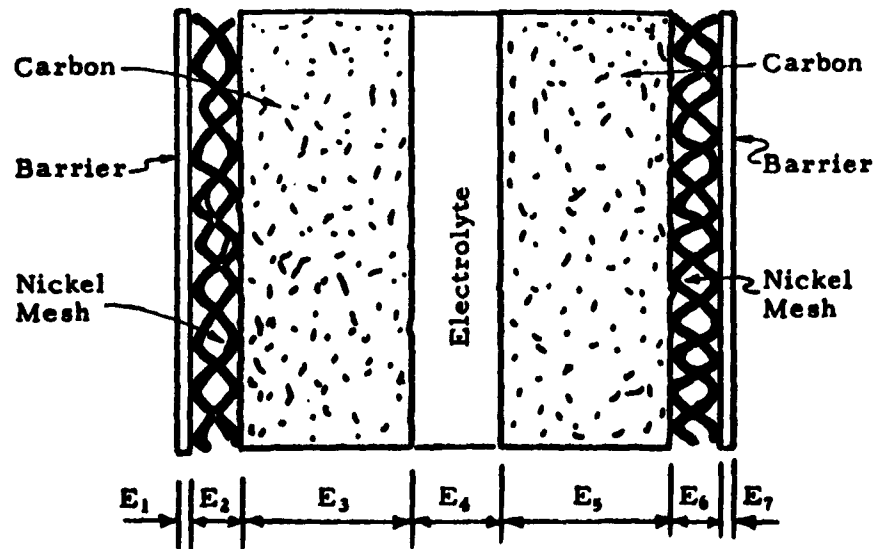


TABLE IV

TYPICAL IR DROP OF CELL COMPONENTS

| <u>Component</u>             | <u>IR Drop at 50 ASF</u> |
|------------------------------|--------------------------|
| E <sub>1</sub> - barrier     | < .0001                  |
| E <sub>2</sub> - metal mesh  | .002-.005                |
| E <sub>3</sub> - carbon      | < .0002                  |
| E <sub>4</sub> - electrolyte | .067-.082                |
| E <sub>5</sub> - carbon      | < .0002                  |
| E <sub>6</sub> - metal mesh  | .002-.005                |
| E <sub>7</sub> - barrier     | < .0001                  |
| Total IR Range               | .068-.093                |

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in each instance load voltage remained essentially unchanged at 0.72 volt (25°C). No observable changes could be detected in the physical condition of these cells at the conclusion of the measurements.

Polarization data before and after shock testing were as follows:

TABLE V  
EFFECT OF SHOCK TESTING ON CELL DISCHARGE

|           | Before Shock Test<br>(40-45°C) | After Shock Test<br>(40-45°C) |
|-----------|--------------------------------|-------------------------------|
| O. C. V.  | 1.065                          | 1.050                         |
| 2.8 amps  | 0.890                          | 0.880                         |
| 5.0 amps  | 0.855                          | 0.850                         |
| 10.0 amps | 0.785                          | 0.780                         |
| 15.0 amps | 0.735                          | 0.725                         |

Note: 11 amps = 50 ASF

Shock testing using larger carbon electrodes of the 12" x 14" x 1/4" size was restricted to individual cell components because of equipment limitations. Shock force effects up to 200 G were determined on the carbon electrodes, framed electrodes with battery end plate supports, and framed electrodes taken from test batteries after 200-2000 hrs of discharge testing. Shock was applied 3 times to each test specimen in each direction at 50 G, 100 G and 200 G forces. In no instance was damage evident in any of the test specimens. It is particularly gratifying that long discharge usage had not reduced shock resistance.

Further testing on large electrodes involved shock forces of 200 G applied perpendicularly to the large face of a specimen carrying a normal head of electrolyte. The surface of the electrode was prewet with the alkaline electrolyte to establish a wet interface as in an operating cell. No physical damage could be detected. Electrode weight measurements before and after the shock test showed no increase in weight and cross-section examination following the test failed to reveal any evidence of accelerated solution penetration into the electrode pores.

TABLE VI  
SHOCK TESTING

| Specimen                                                                                   | Axis* | Shock Force |     |     | Repeat Cycles |     |     | Results   |
|--------------------------------------------------------------------------------------------|-------|-------------|-----|-----|---------------|-----|-----|-----------|
|                                                                                            |       | (1)         | (2) | (3) | (1)           | (2) | (3) |           |
|                                                                                            |       | g           | g   | g   |               |     |     |           |
| 1) Dry carbon electrode                                                                    | A-A   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | B-B   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | C-C   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
| 2) Dry carbon electrode in rubber frame                                                    | A-A   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | B-B   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | C-C   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
| 3) Dry carbon electrode in rubber frame and battery end plate                              | A-A   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | B-B   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | C-C   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
| 4) Carbon electrode in rubber frame after 200 hrs battery service                          | A-A   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | B-B   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | C-C   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
| 5) Carbon electrode in rubber frame and battery end plate after 2000 hours battery service | A-A   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | B-B   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |
|                                                                                            | C-C   | 50          | 100 | 200 | 3             | 3   | 3   | No damage |

\*Axis A-A, B-B, C-C are mutually perpendicular.

#### b) Vibration and Shock Testing

A plastic-framed cell using 6"x 6" x 1/4" nominal size carbon electrodes, activated with electrolyte and connected to hydrogen and oxygen supplies, was mounted on a Model C 10-C Exciter (M-B Manufacturing Co. ) with the accelerating forces normal to the electrodes. Model No. 2213 Crystal Accelerometers (Endeoco Corp. ) were used to monitor acceleration during vibration.

Current was varied from 2 to 11.5 amperes. The frequency range was 380 to 5000 cycles per second; acceleration up to 27 G. Test performance remained normal throughout the test and the cell was found to be in good physical condition in the subsequent post-test examination.

**TABLE VII**  
**VIBRATION AND ACCELERATION DURING DISCHARGE**

| Time of Test | Current amps | Voltage | Vibration (cps) | Acceleration (G) |
|--------------|--------------|---------|-----------------|------------------|
| 12 min       | 2            | 0.890   | 380             | 7.5              |
|              |              | 0.895   | 560             | 27.0             |
|              |              | 0.895   | 3000            | 27.0             |
| 5 min        | 5            | 0.820   | 580             |                  |
|              |              | 0.820   | 3000            |                  |
| 6 min        | 11.5         | 0.720   | 560             |                  |
|              |              | 0.720   | 3000            |                  |
| 30 sec       | 11.5         | 0.725   | 5000            | 10.0             |

Note: 11.5 amps ~ 50 ASF

Vibration tests at relatively low amplitudes on three 6-cell batteries (6"x 6"x 1/4" electrodes) were conducted on a L. A. B. Corporation Type RVH Vibration Test Stand. The batteries were vibrated in three mutually perpendicular directions for a total test time of 4-1/2 hours. Vibration amplitude was 0.06", the frequency 10 cps. Post-test examination of the three batteries (36 electrodes) showed no damage to frames or electrodes.

Similar excellent resistance to vibration forces was obtained for a 12-cell battery assembled with 12"x 12"x 1/4" rubber frames, and subjected to the same vibration forces and amplitude as were employed for the preceding tests on the smaller dimensioned batteries; i. e., 10 to 60 cps and 0.06" amplitude. The time for the tests was 1.5 hours per axis, or 4.5 hours total.

Shock testing of three 6-cell batteries (6"x 6"x 1/4" electrodes) after completion of the vibration test series was carried out on the Barry Corp. Type 20 VI Component Shock Machine. Each shock level was applied once along three mutually perpendicular axes. Twenty G's deceleration shock was sustained for a duration of 5.5 milliseconds and 40 G's for a period of 7.5 milliseconds. No damage was observed in any of the 36-framed electrodes.

### c) Acceleration Testing

For this measurement an activated test cell was mounted in a rotary accelerator Type G-6-B Serial No. 180 (Schaevitz Machine Works) 24-3/8" from the center of rotation, electrode faces being normal to the accelerating forces. A head of 15" of KOH electrolyte was applied to the cell parallel to the accelerating forces to obtain maximum hydraulic pressure on the electrodes. Gas supplies maintained at about 0.5 psi were mounted on the opposite end of the rotating arm; exhaust gases were vented through rotary gas connections on the rotary column.

Voltage and current taps were made on the column to permit controlled variation in cell operation during rotation. Acceleration from zero to 12 G was uniformly attained over a 4 minute period and held for one minute at the maximum value. A 3.0 ampere load was applied to the cell for the entire 5-minute test period. The cell voltage held constant at 0.850 volt over this time. Polarization data obtained prior to rotation and immediately after conclusion of this test gave no evidence of change in operational characteristics. Examination of the cell after acceleration disclosed no damage or appearance change.

TABLE VIII  
EFFECT OF ACCELERATION TESTING ON CELL PERFORMANCE

| <u>Current Load</u><br><u>(amps)</u> | <u>Cell Voltage (Terminal)</u> |                       |
|--------------------------------------|--------------------------------|-----------------------|
|                                      | <u>Before Rotation</u>         | <u>After Rotation</u> |
| 3.0                                  | 0.840                          | 0.845                 |
| 5.0                                  | 0.790                          | 0.800                 |
| 10.0                                 | 0.685                          | 0.695                 |
| 12.3                                 | 0.645                          | 0.680<br>(11 amps)    |

With the current through the cell maintained at the 11 ampere level, rotation was increased to 14 G acceleration in 30 seconds and held for 1.5 minutes. The voltage remained unchanged at 0.690 volt during this time. No physical damage was observed at the conclusion of this test.

Further measurement involved a 20 G acceleration in 2 minutes; then, during application of a 3 ampere load, increases to 30, 40 and finally 50 G during the succeeding 1 minute period. The cell potential remained unchanged at 0.850 volt. At this point a gas bottle connection developed a leak necessitating termination of the test. No cell damage was observed and the unit continued to operate satisfactorily when normal test conditions were restored.

d) Torque Testing

Torque tests were made on 6-cell batteries (6"x 6"x 1/4" plastic framed electrodes) to determine the effect of repeated assembly and disassembly as a measure of design suitability and mechanical strength of the battery components. The stacks of 12 electrodes making up each battery were held together by means of ten tie bolts passing through the peripheral edges of each plastic frame. Each assembly operation involved tightening the tie-bolt nuts with a torque wrench to approximately 45-inch pounds. After three cycles at 24-hour intervals, the batteries were again dismantled for careful examination of the frames and electrodes. No failures were observed in any of the 36 framed electrodes.

e) Temperature Testing

After equilibration at room temperature, five groups of 6-cell batteries, again featuring the plastic-framed 6"x 6"x 1/4"carbons, were placed into a cold test chamber at temperatures of -20°C and -40°C for periods up to 115 hours. The batteries were returned to room temperature periodically to check for electrode and frame condition. No failures were observed in the 24 framed electrodes representing the preferred molding material.

f) Leakage Testing

Following exposure to temperature, vibration, shock and torque testing, a group of five 6-cell batteries were filled with electrolyte and stored at 115°F for periods up to 96 hours. At the conclusion of this exposure, the batteries were drained and dismantled for examination of the

bond between the plastic frames and the carbons. Chemical tests applied to the bond surfaces completely around each carbon failed to indicate any leakage points in the 60 electrodes tested.

#### 8. Schematic Arrangement of Battery Complex Elements

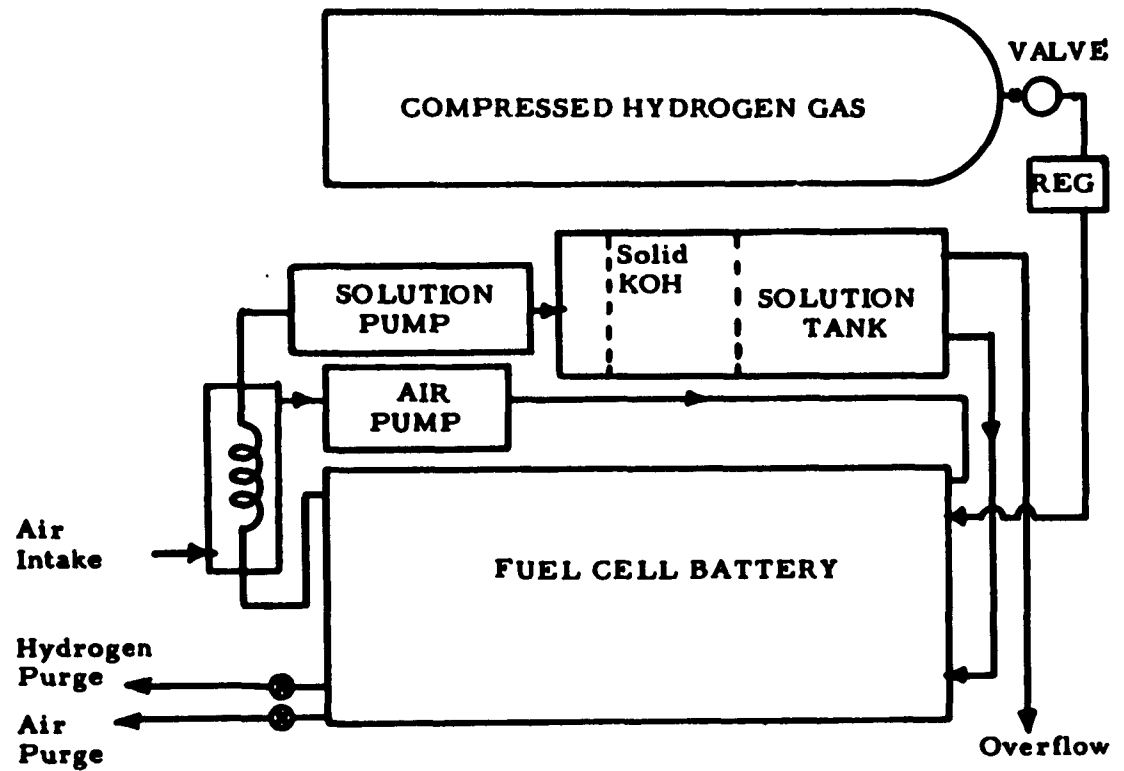
Figure 13 portrays a generalized arrangement of components comprising a complete fuel cell battery plant utilizing a cylinder of compressed hydrogen gas as the anode reactant source. Such hardware was not assembled during the contract period in view of the greater need for intensive study of other aspects of the project. The dimensions therefore are not final and in all likelihood are subject to substantial downward revision when a "close-packed" engineering design is initiated.

Progress in other areas of UNION CARBIDE Fuel Cells has called attention to the practicality of compact two-stage gas pressure regulators weighing less than 1/2 pound (Linde Division of Union Carbide Corporation) and capable of holding low pressures (5'-16" H<sub>2</sub>O) within very close limits. Small, leakproof, maintenance-free electrolyte pumps have been used in the laboratory for hundreds of hours of continuous operation without difficulty. Other components, e. g., plastic piping, plastic fittings, plastic valves have been evaluated with excellent results. Where metal components are used as part of the electrolyte system they are of nickel to avoid the possibility of metallic contaminants entering the electrolyte.

Figure 14 illustrates more specifically the interrelationships of the various elements of the battery complex. The precise location of any particular component is not fixed, and may be changed as required for greater compactness or effectiveness of the battery complex. Heat exchanger design and location will be determined in the final analyses by the handling of the by-product water entering the electrolyte, the gas streams, etc. The heat exchangers may be used for preheating entering gases, or the electrolyte may serve as a heat source for the gases, depending on current densities used, IR loss within the cells, and insulation factors. Since hardware was not constructed in prototype form, particularized methods have not been developed for dealing with batteries in storage, preparation for activation,

FIGURE 13

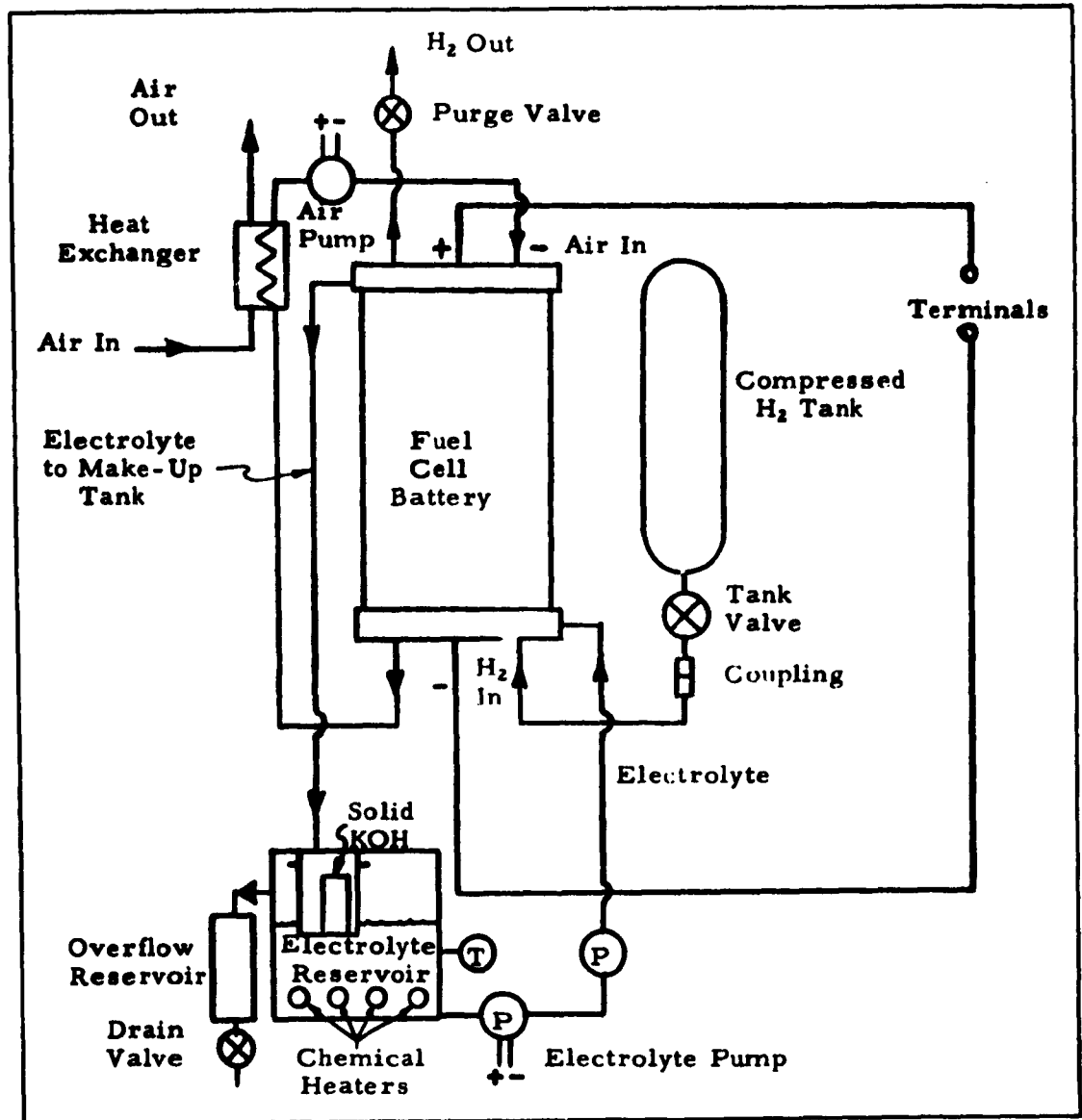
SCHEMATIC ARRANGEMENT OF FUEL CELL BATTERY  
SYSTEM COMPONENTS



C-1537

FIGURE 14

SCHEMATIC FUEL CELL BATTERY SYSTEM -  
GAS, FLUID AND ELECTRICAL CIRCUITS



C-1538

introduction of gases, initiation of operation, guides to efficient operation, and for intermittent battery shutdown. Many of these details were dependent on the unclarified situation regarding water removal during the period of the contract.

### C. Operating Characteristics

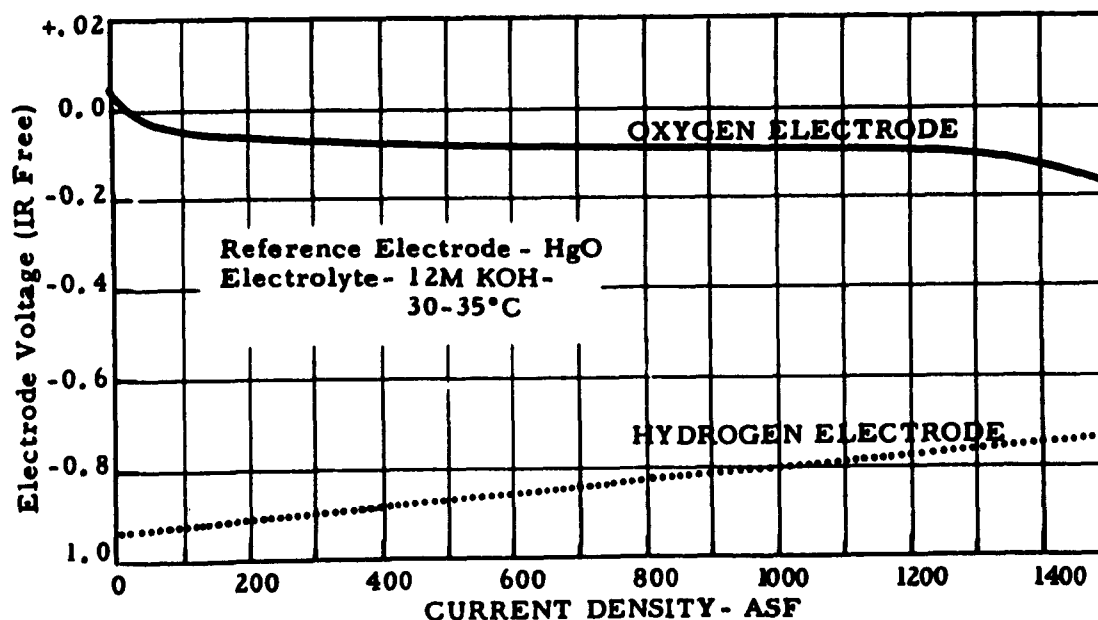
#### 1. Performance with O<sub>2</sub> and H<sub>2</sub> Gas

Most of the operational experience in the early stages of Union Carbide Fuel Cell development were concerned with pure oxygen depolarization of the cathodes. Depolarization by oxygen from atmospheric air differs primarily in the lower partial pressure of oxygen and the corollary problem of nitrogen counter diffusion. To establish a base line for performance evaluation, single electrodes, unit cell and battery tests were conducted with oxygen as well as air.

Single electrode tests on anodes and cathodes incorporating the latest improvements in fabrication have shown major advances in terms of current/voltage relationship. The oxygen supplied cathode now shows a limiting current density of 1400-1600 ASF in 12 M KOH at 35°C against an inert nickel anode using an electronic interrupter for IR-free voltages. The anode has been taken to better than 2000 ASF under the same conditions.

**FIGURE 15**

#### SINGLE ELECTRODE TESTS ON ANODE AND CATHODE (E vs. I)



C-1539

Single electrode discharge tests on cathodes at 200 ASF have been carried as high as 3000 hours and up to 4000 hours at 100-200 ASF. Anodes have been operated continuously at 100 ASF for 2000 hours, and for 1500 hours at 200 ASF.

Many single cells are currently delivering 3000-4000 hours at 40-50 ASF with individual units indicating possibilities for operation beyond 5000-6000 hours. Life expectancy currently is approximately 2000 hours for 80 per cent of the cells subjected to test and 95 per cent to 500 hours.

**TABLE IX**  
**OPERATIONAL LIFE EXPECTANCY OF FUEL CELLS WITH**  
**1/4" FLAT CARBON ELECTRODES**

| Date of Testing | Percentage of Cells Surviving* |          |          |          |
|-----------------|--------------------------------|----------|----------|----------|
|                 | 500 hrs                        | 1000 hrs | 2000 hrs | 4000 hrs |
| 1959            | 30%                            | 10%      | ---      | ---      |
| 1960            | 50%                            | 20%      | 10%      | ---      |
| 1961            | 80%                            | 60%      | 10%      | ---      |
| Early 1962      | 90%                            | 80%      | 50%      | 10%      |
| Mid 1962        | 95%                            | 90%      | 80%      | 20%**    |

\* The percentage figure indicates the portion of surviving cells on a continuous load of 40 to 50 ASF operated on H<sub>2</sub> and O<sub>2</sub>. A test is considered terminated for mechanical reasons (breakage, gas or electrolyte loss, etc.) or when terminal voltage drops under 0.75 volt under load.

\*\*Incomplete; in latest test series one cell operated for 4700 hrs, another cell operated for 6400 hrs; other tests are still in progress.

## 2. Cathode Operation with Air-General

Substitution of air for oxygen greatly reduces the limiting current density at the carbon cathode. Whereas oxygen-supplied electrodes sustain current loading of 200 ASF for thousands of hours as indicated before, the same electrode stock maintains only 35-50 ASF with air under otherwise

similar conditions of operation.

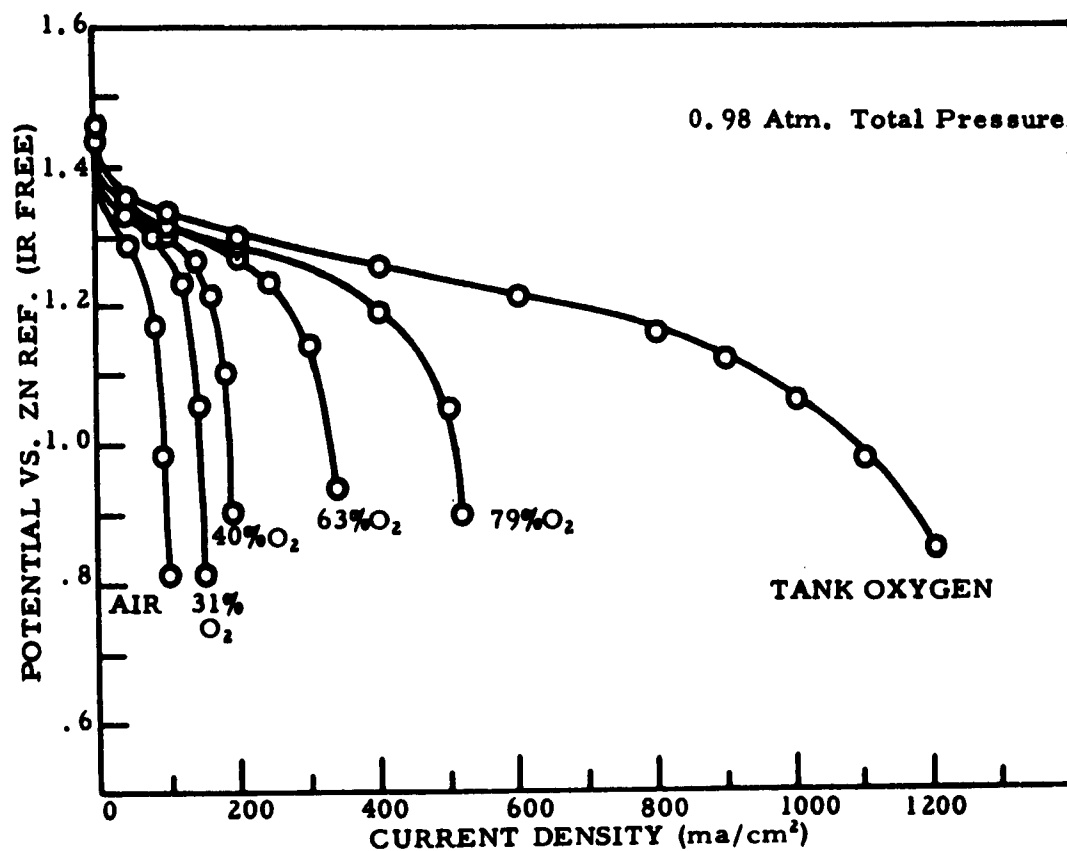
Examination of the obvious physical parameters of permeability and porosity over the range likely to be considered for porous type electrodes, reveals no important generalization regarding electrochemical performance. Permeability data furnish strong evidence regarding the nature of gas flow through carbon pores but do not particularize the precise transfer mechanism by which oxygen is adsorbed on active sites at a working electrode surface. While at the present time diffusion of oxygen through the associated nitrogen of atmospheric air may offer a partial mechanical approach to the problem of oxygen migration, it is also evident that permeability or porosity, per se, or diffusion are not adequate in themselves for prediction of electrochemical behavior. A more useful indication of performance apparently is the measure of surface "activity," again recognizing that there are practical upper and lower limits for this property.

Activation, or the process of developing a surface, has no noticeable effect on gross permeability to oxygen or nitrogen in the range of electrode processing of interest in the development of gas electrodes. Wetproofing treatment used for added repellency to electrolyte penetration into an electrode, likewise is without effect on the free flow of oxygen or air through the pore structure.

When carbon cathodes are operated with oxygen-nitrogen mixtures of varying proportions for the study of limiting current densities, the anticipated sharp breaks are replaced by a more gradual "rounded" drop, reflecting the heterogeneity of the customarily encountered active carbon surface, and the relatively broad range of active site effectiveness. This is illustrated in Figure 16 for gas mixtures running from 760 pp  $O_2$  down to 160 mm pp  $O_2$  plus 600 mm pp  $N_2$ . In this particular series of Research measurements, the air composition curves (EMF vs. pp  $O_2$ ) show a definite break starting at about 50-60 ASF.

FIGURE 16

CURRENT-CATHODE POTENTIAL CURVES (vs. pp O<sub>2</sub>)



C-1540

### 3. Half-Cell Test Data with Air

Half cell current density/voltage tests made on the carbon cathodes in 12M potassium hydroxide at 35°C point to reasonably good voltages for the relatively short period of the tests. Over longer periods of operation, the potentials drop as a result of the nitrogen blanket built up in the carbon pores and slowness of oxygen diffusion through the nitrogen to replace the electrochemically reacted oxygen at the electrolyte:carbon surface. Continued operation under these conditions accelerates electrolyte penetration and premature failure of the electrodes, as indicated in the following outline of experimental measurements.

TABLE X

**HALF-CELL AIR CATHODE TESTS (VOLTAGE VS. CURRENT DENSITY)**

| Electrode No. |      |         | Mv. vs. HgO Electrode (35°C) |     |     |      |      |      |        |
|---------------|------|---------|------------------------------|-----|-----|------|------|------|--------|
|               |      |         | O.C.                         | 10  | 25  | 50   | 75   | 100  | 150ASF |
| 1             | 1/8" | 89-P90  | +30                          | -30 | -55 | -75  | -90  | -115 | -320   |
| 2             | 1/4" | 89-P90  | +30                          | -30 | -50 | -65  | -80  | -170 | -400   |
| 3             | 1/4" | 103-P90 | +25                          | -50 | -75 | -100 | -120 | -180 | -400   |
| 4             | 1/4" | 85-LB   | +15                          | -45 | -70 | -90  | -105 | -140 | -400   |
| 5             | 1/4" | Res.    | +100                         | +30 | +15 | -10  | -20  | -25  | -25    |

Continued on air operation at 50 ASF continuous discharge, the electrodes gradually lost voltage. Test termination occurred between 6-7 days.

|   |      |         | Mv vs. HgO Electrode (35°C) |        |        |        |         |         |
|---|------|---------|-----------------------------|--------|--------|--------|---------|---------|
|   |      |         | 1 hr                        | 24 hrs | 48 hrs | 96 hrs | 140 hrs | 170 hrs |
| 1 | 1/8" | 89-P90  | -95                         | -75    | -80    | -85    | -90     | failed  |
| 2 | 1/4" | 89-P90  | -95                         | -85    | -85    | -85    | -90     | -90     |
| 3 | 1/4" | 103-P90 | -90                         | -95    | -100   | -100   | -100    | failed  |
| 4 | 1/4" | 85-LB   | -90                         | -80    | -80    | -85    | -90     | -90     |
| 5 | 1/4" | Res.    | 0                           | -25    | -5     | failed | ---     | ---     |

**4. Cell Operation with Air**

Early laboratory cell tests with baked-carbon electrodes running on air at 25°-30°C are summarized in Table XI. These cells were assembled in accordance with the rubber gasket type framing procedure illustrated in Figure 5 (p 12 of this report). A schematic arrangement of the electrolyte and gas flow circuits for the cell testing is outlined in Figure 17. A typical test bench for the testing of experimental cells is shown in Figure 18; this bench is provided with circulating electrolyte systems, pumps, surge tank for the electrolyte, filter, gas lines, pressure regulators. Resistance-free voltage measurements, IR drop determination and single electrode data are obtained by means of the portable electronic interrupter shown in the foreground.

TABLE XI  
UNIT CELL LIFE TESTS\* WITH AIR AND OXYGEN CATHODES

|       |                 | AIR CATHODES             |       |        |         |         |         |         |         |         |         |         |  |  |
|-------|-----------------|--------------------------|-------|--------|---------|---------|---------|---------|---------|---------|---------|---------|--|--|
|       |                 | Hours on Continuous Test |       |        |         |         |         |         |         |         |         |         |  |  |
| Tests | Current Density | <50                      | 50-75 | 75-100 | 100-125 | 125-150 | 150-175 | 175-200 | 200-225 | 225-250 | 250-275 | 300-325 |  |  |
| 4     | 10              | ---                      | 1     | 2      | ---     | ---     | ---     | ---     | 2       | ---     | ---     | ---     |  |  |
| 3     | 20              | ---                      | ---   | 1      | 1       | ---     | 1       | ---     | ---     | ---     | ---     | ---     |  |  |
| 18    | 25-30           | 11                       | 2     | ---    | 1       | ---     | 1       | 1       | 1       | ---     | 1       | ---     |  |  |
| 6     | 40              | ---                      | 1     | ---    | ---     | 2       | 2       | ---     | ---     | 1       | ---     | ---     |  |  |
| 1     | 50              | ---                      | ---   | ---    | ---     | ---     | ---     | ---     | 1       | ---     | ---     | ---     |  |  |
| ---   | ---             | ---                      | ---   | ---    | ---     | ---     | ---     | ---     | ---     | ---     | ---     | ---     |  |  |
| 32    | ---             | 11                       | 3     | 3      | 2       | 2       | 4       | 1       | 4       | 1       | 1       | ---     |  |  |

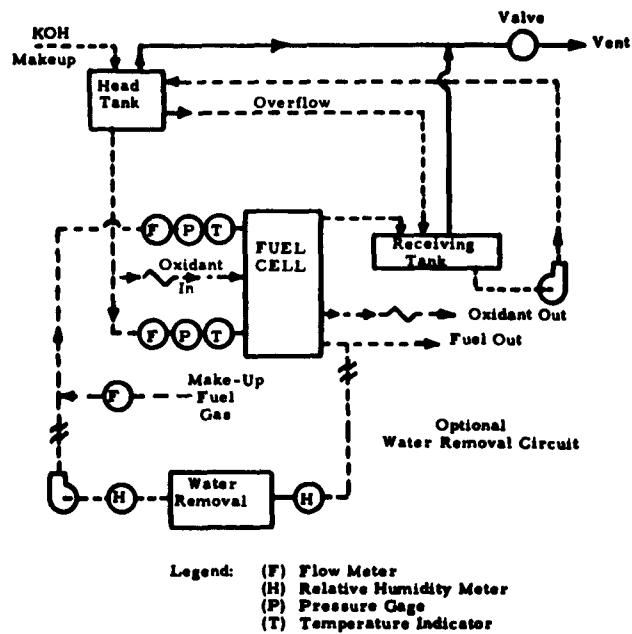
OXYGEN CATHODES

|       |                 | Hours on Continuous Test |       |        |         |         |         |         |         |         |         |         |     |  |
|-------|-----------------|--------------------------|-------|--------|---------|---------|---------|---------|---------|---------|---------|---------|-----|--|
|       |                 | Hours on Continuous Test |       |        |         |         |         |         |         |         |         |         |     |  |
| Tests | Current Density | <50                      | 50-75 | 75-100 | 100-125 | 125-150 | 150-175 | 175-200 | 200-225 | 225-250 | 250-275 | 300-325 |     |  |
| 2     | 20              | 1                        | 1     | ---    | ---     | ---     | ---     | ---     | ---     | ---     | ---     | ---     |     |  |
| 20    | 25-30           | 11                       | 1     | 2      | 1       | 1       | ---     | ---     | ---     | 1       | ---     | ---     | 3** |  |
| 68    | 50              | 31                       | 9     | 8      | 3       | 4       | 7       | 1       | ---     | ---     | 2       | ---     | --- |  |
| 3     | 100             | 2                        | ---   | 1      | ---     | ---     | ---     | ---     | ---     | ---     | ---     | ---     | --- |  |
| ---   | ---             | ---                      | ---   | ---    | ---     | ---     | ---     | ---     | ---     | ---     | ---     | ---     | --- |  |
| 93    | ---             | 45                       | 11    | 11     | 4       | 5       | 7       | 1       | ---     | 1       | 2       | 3       | 3   |  |

\* Room temperature with KOH electrolyte.

\*\* 450, 560, and 980 hours.

FIGURE 17  
SCHEMATIC DRAWING OF FUEL CELL TEST ARRANGEMENT



C-1541

FIGURE 18  
LABORATORY BENCH FOR UNIT CELL TESTS



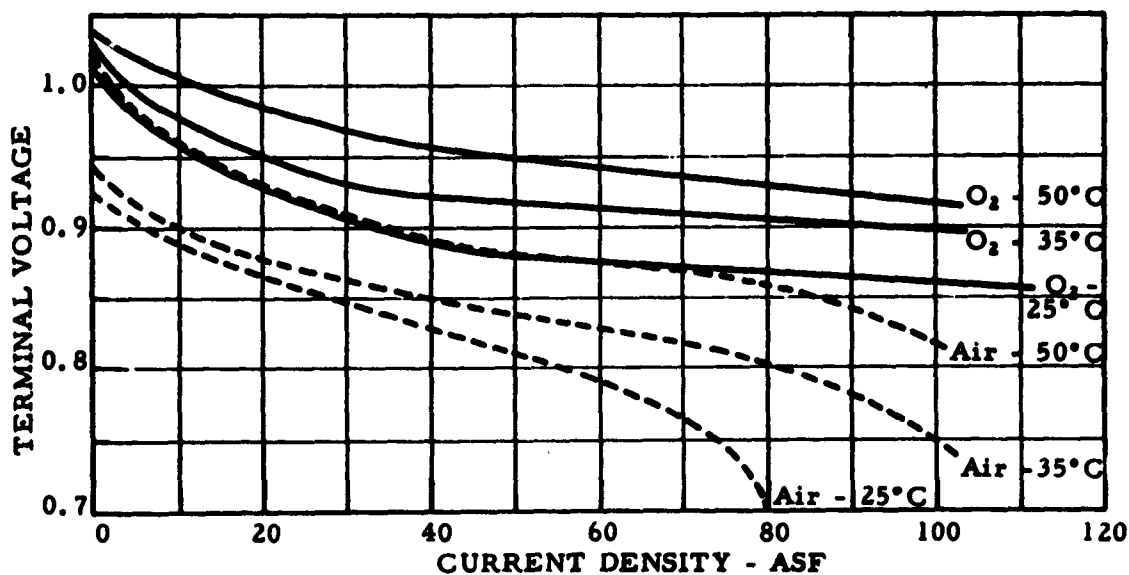
C-1542

Examination of the data above reveals no clear-cut relationship between current density and hours on test, and suggests that factors other than use of air were overriding in determining test termination. A scan of the performance obtained with oxygen supplied cells of the same general period points to a slightly more regular distribution. In both cases however, the majority of tests fell into the short service category. Single cell performance with air during this early period reached a peak of 300 hours at 35-40 ASF; with oxygen cathodes service maxima of 500-900 hours were obtained.

A comparison of current density:voltage relationship for unit cells featuring baked-carbon cathodes operated with air and with oxygen is given in Figure 19. The tests with oxygen show the non-linear type curve at low current densities, characteristic of cathodic reduction of oxygen to peroxy ion, followed by approximately linear curves for the limited current density range covered by the measurements.

FIGURE 19

COMPARISON OF  $H_2-O_2$  AND  $H_2$ -AIR CELLS



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Corrected for IR drop, the discharge curve would be relatively flat, in line with expected electrode behavior. Rising temperature brings about a rise in cell voltage due principally to a drop in electrolyte resistance, and to a lesser extent, to reduction in polarization by increased gas diffusion.

Even with a large excess of air flowing over the cathode breathing surface, the performance remains characteristically lower than with oxygen. Gas diffusion related breaks occur earlier at 25°C than at 35°C or 50°C, with indication that at 50°C, the limiting current density is about 65-75 ASF.

Other Research Laboratory tests show that on continuous discharge with air depolarization, the baked-carbon cathodes can operate at 50 ASF for periods as long as 400-500 hours. For the present these are the exception but should be considered valid targets for a further development program based on baked carbons which does not impose such severe weight limitation on the battery as does the present contract. Continuous operation with baked carbon electrodes currently may be considered confined to an upper range of 25-35 ASF. At 25 ASF, performance with air can be expected to yield 1000 hours at 30°-40°C.

Sodium hydroxide appears to be a better electrolyte than KOH for use with air in view of the much lower solubility of CO<sub>2</sub> in the former, and offers the further advantage of somewhat greater cathode stability. While permissible current density is restricted to about 15 ASF used continuously at room temperature, elevation of operating temperature or intermittent testing allows use of somewhat higher current densities. A small, 6-cell, 1.5 ampere hydrogen-air battery operated intermittently at about 20 ASF at room temperature for hundreds of hours over a 5-month period.

Substitution of 1/8" baked carbons for the usual 1/4" electrodes permits a reduction in weight but has revealed no outstanding trend toward improved operation. The new "fixed-zone" electrode is a basically sounder approach to the problem of lightweight, air-depolarized batteries, and promises to broaden the spectrum to higher current densities. Combining higher temperature and "fixed-zone" electrode construction, battery operation with air at 30 ASF has reached 500 hours at 50°C and 500 hours at 50 ASF and 60-70°C.

The problem of air usage continues to receive intensive research and development attention to establish a sound theoretical basis for gas mixtures, as well as practical approaches to optimization of operation. Studies of base materials, structure, surface development, catalysts and electrolyte repellency treatments for electrodes are in this category.

#### D. Removal of By-Product Water

Because the weight of battery auxiliaries is a large factor in weight control of battery systems, Union Carbide sponsored research efforts have been intensively directed to the elimination of electrolyte circulation. Results are highly encouraging.

Using baked-carbon electrodes with oxygen depolarized cathodes, a 5-cell battery was on 50 ASF discharge for 500 load hours in a temperature range of 65°-70°C, then continued on a 35 ASF load for an additional 1400 hours at 45°-50°C for a total of 1900 load hours with an average cell potential of 0.8 volt at this service point.

A second battery (7 cells) using baked carbon electrodes with oxygen cathodes was on 50 ASF operation for better than 3600 load hours including excursions to 100 ASF. Average cell voltage at the 2800 hour point was 0.8; at the 3600 hour termination point, 5 cells were still operable.

Data obtained in the course of the foregoing tests and other related bench measurements on water vapor transpiration through 1/4" baked-carbon electrodes, using a 9N KOH electrolyte are summarized in Table XII.

TABLE XII  
EQUIVALENT WATER TRANSPIRATION RATES

| Cell Temperature<br>°C | Equilibrium*<br>Current Density |
|------------------------|---------------------------------|
| 35                     | 10 ASF                          |
| 50                     | 25 "                            |
| 65                     | 50 "                            |
| 80                     | 100 "                           |
| 100                    | 150                             |

\* By-product water balances water transpiration.

Note: - Hydrogen flow maintained at 10 times the theoretical rate plus small purge flow to eliminate inerts.  
- Oxygen flow maintained at theoretical rate plus small purge flow to eliminate inerts.  
- Condenser temperature (water cooled) 15°C.

If a constant current density is employed, e.g., 50 ASF, the indicated electrolyte concentration:temperature relationship required for invariant composition when using 1/4" baked carbon electrodes will vary between 40°C for a 7N KOH solution, 50°C for 8N KOH, and 70°C for 12N KOH.

The cathode also transpires water into the gas stream although at a lesser rate than through the anodes. Under comparable flow conditions this may amount to 25 to 50 per cent of the anodic transpiration value. Therefore, if two circulating gas systems are used ( $H_2$  and  $O_2$ ) either a higher electrolyte concentration can be employed at any particular temperature, or a commensurate increase in current density may be utilized. However, the latter feature may not apply to the same extent to operation with air in view of the further diluent effect of the water vapor on the oxygen partial pressure.

Water transpiration measurements using "fixed-zone" electrodes are equally encouraging. Using an oxygen cathode, a cell has been on test for over 2200 load hours at a current density of 35 ASF and a temperature of 50°-60°C. In this instance electrolyte circulation was used and water removed continuously by transpiration. Terminal voltage at the 2200 hour point was 0.8 volt. Small size multicell batteries featuring "fixed-zone" electrodes have been operated intermittently at 10-25 ASF for periods up to 2500 hours.

Elimination of electrolyte circulation introduces the added feature of narrower electrolyte gaps, hence higher permissible current density to bring about the rise in temperature needed for air operation and water transpiration.

Exact data on water removal is still lacking in view of the many dependent variables influencing the water vapor movement, e.g., electrode permeability, wetproofing, electrolyte concentration, gas flow rates, temperature, influence of transpiration through the cathode in addition to the anode. A presently unknown quantity is the actual water vapor pressure above the electrolyte present in the pores of the electrode, and the concentration gradients and varying viscosities, etc., existing in the pore system. A program covering an intensive examination of the foregoing parameters should provide the necessary answers to a practical portable battery.

## V. Portable Hydrogen Source

### A. Analysis of Requirements

Fuel sources eligible for consideration were in the form of compressed gas liquid or solid. Further, converters or generators were considered for converting a primary fuel to hydrogen. Attention in the selection of the fuel storage system was given to weight, hazards, cost, portability and control. A broad range of environmental conditions were required including shock, vibration, acceleration, humidity, ambient temperatures, and high altitude. The most imposing problem was the weight requirement since the fuel sub-system was considered part of the total system limit weight of 35 pounds.

### B. Experimental

#### 1. Equipment and Methods

In view of work done elsewhere and from initial calculations, it appeared that chemical sources of hydrogen would be most suitable for a portable type unit. Accordingly, two laboratory reactors were developed for experimental work. One was based on the concept of a fluidized bed reaction since there appeared to be a possibility that the battery by-product water might be transpired from the cell into the gas streams, thus permitting generation of hydrogen by passage of wet hydrogen gas through the chemical hydrogen source. The other reactor was intended for the more conventional approach of dripping a liquid onto the chemical hydrogen source. This liquid could be water, the cell electrolyte or possibly other liquids indicated by the requirements of the system.

#### 2. Materials.

Table XIII lists the compounds which have been considered as hydrogen sources for this work. While there is considerable variation of the liters of hydrogen available per gram of chemical, other factors are involved in choosing a suitable source. These included safety, convenience, cost, availability, etc. Calcium hydride, for instance, while not particularly high in contained hydrogen appeared well worth considering from all other viewpoints. With a battery of reduced weight and free from the need for electrolyte circulation, the calcium hydride, despite its relatively poor

yield, remains a potentially useful source of hydrogen. The alkali aluminum hydrides were not known to be available until experimental work on hydrogen generation from chemical sources was discontinued. They are included in the table however to show the considerable promise they would have if price and other factors were favorable. Further, since all of these compounds required addition of liquid to generate the hydrogen, a more correct comparison is based on total system weight, rather than on the hydrogen containing compound alone. This comparison is shown in Column 2 of Table XIII.

TABLE XIII  
THEORETICAL HYDROGEN YIELDS FOR VARIOUS COMPOUNDS

| Compounds          | Column 1                                | Column 2                              |
|--------------------|-----------------------------------------|---------------------------------------|
|                    | Liters H <sub>2</sub> Per Gram Material | Liters H <sub>2</sub> Per Gram System |
| LiH                | 2.82                                    | .86                                   |
| NaH                | .94                                     | .53                                   |
| KH                 | .56                                     | .38                                   |
| CaH <sub>2</sub>   | 1.06                                    | .57                                   |
| MgH <sub>2</sub>   | 1.71                                    | .72                                   |
| NaBH <sub>4</sub>  | 2.40                                    | 1.21                                  |
| LiBH <sub>4</sub>  | 4.15                                    | 1.55                                  |
| Al + NaOH          | 0.50                                    | 0.40                                  |
| NaAlH <sub>4</sub> | 1.66                                    | 1.00                                  |
| LiAlH <sub>4</sub> | 2.36                                    | 1.21                                  |

Experience with sodium borohydride confirmed difficulties reported elsewhere in hydrogen generation from this material. The reaction barely started with water, even when cobalt chloride was added as a catalyst. With 18 per cent H<sub>2</sub>SO<sub>4</sub> the reaction started smoothly, but stopped completely at about 60 per cent conversion, leaving a badly caked residue.

Reaction of methanol with calcium hydride, sodium borohydride and sodium hydride-calcium hydride mixtures was very poor. The significance of this for low temperature operation of the battery system is that

the liquid used would have to be a water solution and would be restricted to such a composition that it would not freeze at the minimum temperature required for system operation.

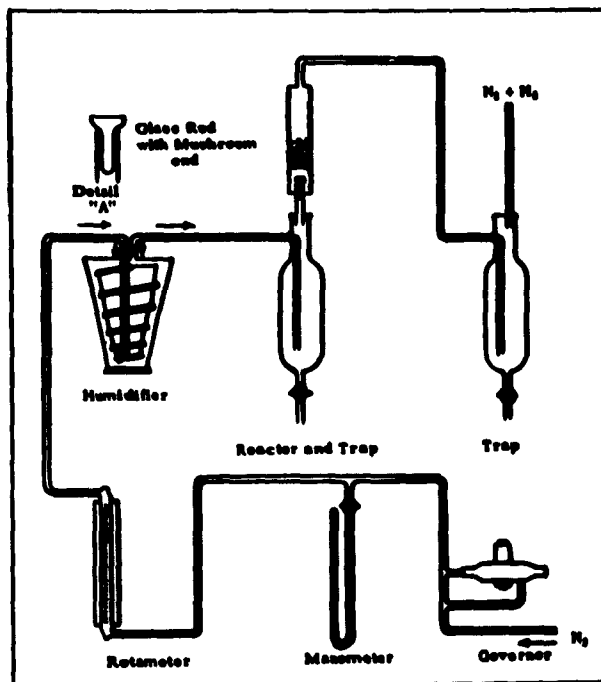
In passing, attention is directed to certain organic compounds having possibilities for chemical generation of hydrogen. These include, for example, diborane, or hydrazine. These materials would require quite different techniques for the release of their hydrogen, techniques which might not be feasible within the present framework of minimum weight and convenience demands.

### 3. Gas-Solid Reactor

The fluidized bed or gas-solid reaction as shown in Figure 20, consisted of a 36-millimeter diameter glass tube about 12 inches high,

FIGURE 20

#### EXPERIMENTAL GAS-SOLID HYDROGEN GENERATOR



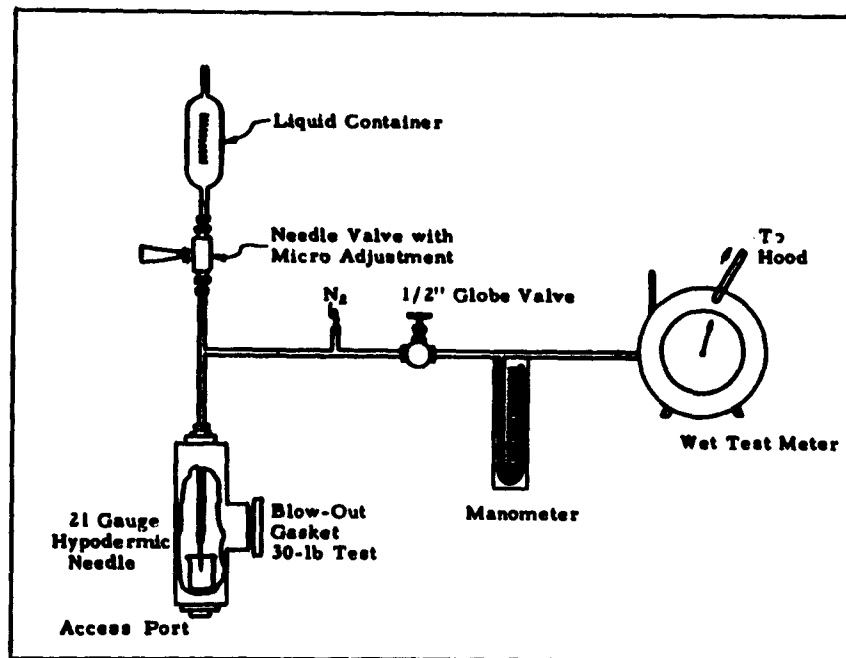
mounted vertically. In the bottom a rubber stopper containing a smaller diameter glass tube served as gas inlet. At the top of the small diameter tube and inside the larger tube a short plug of solid glass rod with a mushroom shaped head was dropped into the tube. This device served as a check valve permitting the gas pressure to push it up and allow gas inflow, but when the gas pressure was shut off, the weight of solid in the larger tube pushed the head down against the smaller tube, thus shutting it off. Operation of the reactor consisted of charging the large tube with about an inch or two of the solid hydrogen source in powdered form. For preliminary work the moisture source was nitrogen bubbled through a flask of water at a temperature such that the vapor pressure of water would produce a predetermined humidity in the gas stream.

#### 4. Liquid-Solid Reactor

The liquid-solid reactor is shown in Figure 21. The body of the reactor is simply a 4-inch pipe tee. The side of the tee is closed with a ring flange and a full-face gasket which acted as a blowout flange for the reactor, calibrated to relieve at approximately 30 psi.

FIGURE 21

#### EXPERIMENTAL LIQUID-SOLID HYDROGEN GENERATOR



C-1545

The solid charge was placed in the bottom of the reactor in a small boat or 50 cc beaker. The liquid was added from the top from a calibrated glass container through a Hoke needle valve with a calibrated stem, and through a hypodermic needle inside the reactor which assured excellent control of the rate of liquid addition. The evolved gas passed through the side piping through a trapped line with shut-off valve, and into a wet test meter and manometer. This reactor proved simple and safe to operate. For handling the reactive alkali hydrides a dry box was used. The containers were opened, charges weighed out, and the resealed containers were kept stored in the dry box. The charge container for the reactor was taken in and out through a panel at one end while nitrogen was flushed through the box.

### C. Results

#### 1. Gas-Solid Reaction

In a preliminary experiment with the gas-solid reactor, 55 per cent conversion of calcium hydride was indicated, based on the change in weight of the charge. The reactor was operated as a column reactor and not in a fluidized condition. Progress of the reaction zone through the bed of  $\text{CaH}_2$  was readily detectable and the reaction stopped as soon as the top layer showed reaction. In a second run a conversion of 62 per cent was obtained, verified by reacting the residue with acid and measuring the evolved hydrogen. No appreciable reaction was obtained using methyl alcohol vapor instead of water. Addition of water to the alcohol led to a brief, steady reaction presumably limited to the water content. Lithium hydride was found to react with alkaline methyl alcohol.

Two runs had been made in the fluidized bed reactor when it became apparent that the then current state of development of the fuel cell favored a system in which the generated water would be absorbed almost entirely in the electrolyte. The fluidized bed approach was therefore set aside to permit consideration of other aspects of the hydrogen generation problem.

## 2. Liquid-Solid Reaction

Preliminary experiments indicated that the very reactive alkali hydrides would react smoothly and steadily with concentrated hydroxides, at least when the hydride was added to a large volume of the liquid. In a test using a small charge of calcium hydride in the liquid-solid reactor, the reaction went smoothly to essentially 100 per cent completion. The solid residue had expanded several fold in volume but remained granular. Conversely, considerable difficulty was encountered in a run with sodium borohydride. The reaction barely started with water even with cobaltous chloride added as a catalyst. With 18 per cent sulphuric acid the reaction proceeded smoothly for a while but stopped completely at about 60 per cent conversion; the residue was badly caked. Several runs were then made using mixtures of solid sodium hydride and sodium hydroxide such as would exist in the later stages of a reaction and using alkaline electrolyte as the liquid reactant. Very low yields were obtained in relation to the amount of liquid used.

A standard method and procedure for carrying out the reaction was based on production of 1 gram mol of hydrogen, or 0.85 cubic foot NTP or the volume required by the 200-watt battery every 12.8 minutes. The experimental reactor was operated by half this rate by adding the liquid phase to the reactor over a time of 25 minutes. Early experiments with the reactant materials indicated that low yields were obtained from a stoichiometric amount of water in the form of electrolyte. The custom was established therefore by adding an additional 100 per cent stoichiometric quantity of water as electrolyte and then finally adding 50 per cent of the stoichiometric requirements as pure water in order to ensure complete reaction of the potentially dangerous charge in the reactor before it was dismantled and washed out.

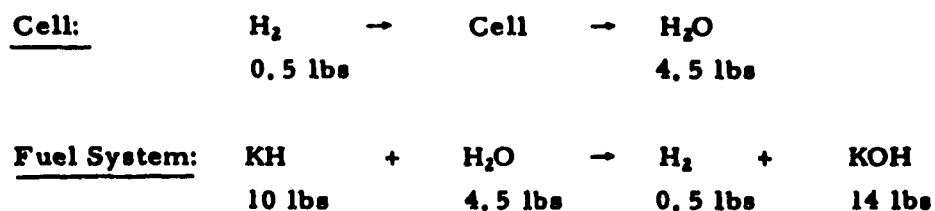
Using this procedure, a run was made using sodium hydride as a reactor charge. Only 25 per cent completion of the reaction was obtained from the stoichiometric quantity of electrolyte. Inspection of the residues from the reaction, and analysis of the results showed clearly that the problem was due to the tendency of the reaction mass to absorb water both physically and as hydrates. As soon as any significant quantity

of hydroxide was formed this prevented the added liquid from contacting hydride. The extent of the problem is indicated by the fact that even after the addition of a stoichiometric quantity of water, conversion was only about 50-60 per cent complete.

Runs were also attempted with methanol and mixtures of  $\text{NaH-CaH}_2$  and with methanol and sodium borohydride. In both cases the reactions proceeded very slowly.

Since the fuel cell produces water as the end product of the reaction, a cyclic system was examined based on the electrolyte from the fuel cell as the second reactant for the hydrogen generating system. Thus, potassium hydride contacted with electrolyte containing excess water from the fuel cell forms potassium hydroxide to regenerate the electrolyte and produce hydrogen fuel.

However the experimental work showed that this approach has limited applicability to present requirements. Thus the daily weight balance of the fuel generation system using KOH as a reactant shows:



The weight of KOH produced is found to be three times that required to produce a saturated solution of the daily water production of the cell. The net effect is a continuous increase in concentration of the electrolyte. From a practical viewpoint 25 per cent of the indicated amount of hydride can be reacted with 25 per cent of the water production in the battery to produce enough KOH to reconstitute the remaining 75 per cent of the water. The rest of the hydrogen would require generation by other methods. In a system requiring a minimum weight such a dual generation approach is not particularly attractive.

### 3. Additives

Two approaches were tried to overcome incomplete conversion. One was the use of a catalyst material similar to the use of cobalt salt in the sodium borohydride reaction. Second, was the use of a porous expander-type material to produce a more uniform spread of the liquid throughout the reaction mass. The addition of 8 per cent by weight of cobalt chloride to the hydride charge did indeed improve the yield in one run. Surprisingly, somewhat the same result was obtained from the use of 8 per cent sodium chloride which is not listed in the literature as a catalyst for such reaction.

First experiments with the expander-type material were based on cellulose fibers and cellulose mat materials. The gas yields were respectively 28 and 33 per cent. It appeared that this type of material absorbed moisture in such a way that it did not contribute to the distribution of the liquid through the reaction. Other fibers are indicated therefore in place of cellulose materials or in conjunction with these to provide a more porous reaction mass in which the water remains available for reaction.

Consideration was also given to the possibility of using mixtures of hydrides and non-porous expanders such as powdered aluminum or shredded aluminum foil. This material would have the added advantage that a reaction could take place between the aluminum and the generated hydroxide to provide still further hydrogen generation. Experiments confirmed this reasoning. With addition of aluminum powder amounting to 8 per cent of the charge, yields were doubled at the end of the addition of a stoichiometric quantity of 36 per cent KOH solution.

Table XIV summarizes the experiments with NaH and additives. From the viewpoint of improved reactivity, the use of aluminum and chloride salts showed most promise. However, the conversion of the sodium hydride is not sufficient to render this approach promising, at least for a system which demands minimum weight. Use of  $\text{CaH}_2$  with catalysts and fibrous extenders appears to be a useful combination for further study.

TABLE XIV  
EFFECT OF ADDITIVES ON HYDROGEN YIELD FROM NaH  
AND 36% KOH SOLUTION

| Additive |                                   | % Conversion | Max. Temp.<br>°F |
|----------|-----------------------------------|--------------|------------------|
| Expander | 1. Cellulose fiber                | 28           | 216              |
|          | 2. Cellulose mat                  | 33           | 262              |
| Catalyst | 1. 8% NaCl                        | 50           | 162              |
|          | 2. 8% CaCl <sub>2</sub>           | 47           | 178              |
| Aluminum | 1. 8% powder                      | 50           | 216              |
|          | 2. 32% powder                     | 58           | 278              |
|          | 3. 8% foil strips                 | 44           | 190              |
|          | 4. 8% powder and<br>Teflon shreds | 48           | 205              |
| 25% KOH  | 5. 8% powder                      | 53           | 218              |
| 49% KOH  | 6. 8% powder                      | 29           | 235              |

Several runs were made using combinations of sodium hydride and additives. However, cellulose mat added to a test charge with 8 per cent sodium chloride reduced the gas yield. Two runs were then made with a mixture of aluminum powder and sodium chloride. One was a standard run; in the other, the hydride was supported on a screen and the liquid fed into a small open space underneath. This reaction started slowly and then went through a period of rapid reaction and higher temperature (305°F) but at the stoichiometric point yields were the same and remained so on further addition of liquid. The yields from both reactions were no better than would have been obtained with aluminum powder alone.

An earlier test had given promising yields of hydrogen, using sodium borohydride and hydrochloric solution. Several runs were attempted using lithium hydride with 15 per cent hydrochloric solution. Yields were lower than had been obtained with sodium borohydride. The addition of aluminum

powder to the lithium hydride charge as an expander did not improve the yield.

#### 4. Calculations

The following table summarizes the weight requirements of the various fuel systems suggested as possibilities. Column 1 indicates the weight of solid theoretically required to provide the requirements for 24 hours. Column 2 shows the approximate cost per pound of hydrogen, or the cost of 48 hours of operation.

TABLE XV

#### PREFERRED HYDROGEN SOURCES LBS/DAY REQUIREMENT (200W)

| Sources                           | Column 1     | Column 2               |          |
|-----------------------------------|--------------|------------------------|----------|
|                                   | Wt. of Solid | Cost/lb H <sub>2</sub> |          |
|                                   | lbs          | (5/1/60)               | (1/9/63) |
| CaH <sub>2</sub>                  | 5.3          | 11.00                  | ---      |
| NaH                               | 6.0          | 35.00 <sup>(a)</sup>   | ---      |
| LiH                               | 2.0          | 44.00                  | ---      |
| NaBH <sub>4</sub>                 | 2.34         | 187.00                 | 11.75    |
| LiBH <sub>4</sub>                 | 1.36         | Approx. 600.00         | 900.00   |
| NaAlH <sub>4</sub> <sup>(b)</sup> | 3.40         | Unknown                |          |
| LiAlH <sub>4</sub>                | 2.40         | ---                    | 33.00    |

(a) Estimate from price of 25 per cent dispersion in mineral oil.

(b) Availability reported to us after discontinuation of fuel generation work.

Assuming realization of a two-thirds conversion efficiency for this same group of materials, twenty-four hour requirements range from 2.0 to 8.0 pounds of chemical reactants, exclusive of the weight of the generator. Taking an arbitrarily assigned value of 7 lbs for the generator device with valves and regulator, 5 lbs for miscellaneous mechanical components (blowers, etc.) and a weight of 15 lbs for an activated 200-watt battery based on the "fixed-zone" electrodes with water vapor transpiration, the total battery system weight becomes 32 pounds as a lower limit for operation at 35 ASF, and perhaps 35 lbs as a realistic target. Improvements in the

yield of chemically produced hydrogen will permit increase in the recharge interval or reduction in weight for a 24-hour interval.

#### 5. Pressurized Gas Storage

An investigation was made into the weights and volumes of small pressurized bottles obtainable for hydrogen gas. The data obtained is summarized in Table XVI following.

TABLE XVI  
SMALL HYDROGEN PRESSURE VESSELS

| No. | Company                                            | Construction                                     | Shape and Size               | Internal vol., in. <sup>3</sup>  | Pressure, psi              | Wgt. lb. | Meets IGC or ASME Code |
|-----|----------------------------------------------------|--------------------------------------------------|------------------------------|----------------------------------|----------------------------|----------|------------------------|
| 1   | Apex Div., White Sewing Machine Co., Cleveland, O. | Fiberglass, reinforced plastic                   | Sphere, 13" O. D.            |                                  | 3000                       | 16       | No                     |
| 2   | Walter Kidde Co., Belleville, N. J.                | Fiberglass wrapped, pre-stressed steel           | Cylinder                     | 850                              |                            | 16.5     | No                     |
|     | "                                                  | Steel wire wrapped, pre-stressed steel*          | Cylinder, 7.4" O. D. x 26.8" | 860                              |                            | 19.5     | No                     |
| 3   | Aluminum Co. of Amer., New Kensington, Pa.         | Fiberglass, wrapped aluminum                     | Cylinder, 9" O. D. x 18"     | 850                              | 3100                       | 13.6     | No                     |
|     | "                                                  | All aluminum                                     | Cylinder                     | 850                              | 3100                       | 16.3     | No                     |
| 4   | Pressed Steel Tank Co. Milwaukee, Wisc.            | Steel                                            | Cylinder 7.2" O. D. x 26.6"  | 850                              | 3100 oprtg. and 4500 burst | 20       | No                     |
|     | "                                                  | Steel                                            | Cylinder                     | 850                              | 3100                       | 35-45    | Yes                    |
| 5   | Linde Company, New York, N. Y.                     | Extra-high strength steel                        | Sphere                       | 850                              | 3100                       | 17.8     | No                     |
| 6   | National Tube Div. U. S. Steel, Pittsburgh, Pa.    | Steel                                            | Cylinder                     | 850                              | 3100                       | 65-75    | Yes                    |
| 7   | Babcock-Wilcox Co., Cleveland, O.                  | Steel                                            | Cylinder                     | 850                              | 3100                       | 60-70    | Yes                    |
| 8   | Combustion Engineering, Cleveland, O.              | Manufacturers pressure vessels only to ASME Code |                              |                                  |                            |          | Yes                    |
| 9   | Linde Company, Tonawanda, N. Y.                    |                                                  | Sphere 15" to 16" O. D.      | (Approx 1/2 lb of liq. hydrogen) |                            | 14       | ---                    |

(\*) These pressure vessels are currently in production or available from stock. All others would have to be custom made.

The hydrogen fuel for 24 hours operation is approximately 0.5 lb or 100 cubic feet at NTP. To contain this volume of gas in 1/2 cubic foot of tank space requires a pressure of approximately 3100 psi. The table shows that the lightest container weights would range from 14 to 20 lbs, but while it

is mechanically possible to build a one-half cubic foot pressure vessel that will withstand 3100 psi internal pressure, these vessels do not meet the standards of the ICC or the ASME. The ICC requires a safety factor of 3 to 1, and ASME 4 to 1. Containers meeting ICC or ASME design specification would be about the same size as those indicated in the table, but would be of heavier wall construction with weights ranging from 40 to 70 lbs.

It is obvious from this data that a 24-hour supply of hydrogen enclosed in a one-half cubic foot certified container would result in a total package weight far in excess of the limits. If it is mandatory to meet the codes and still retain portability then only a few hours of bottled hydrogen gas supply can be carried.

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## **VI. Conclusions**

1. Based on the utilization of ultra thin, porous electrodes, optimization of battery auxiliaries, coupled with vapor phase removal of by-product water through the electrodes, a weight of 32-35 pounds is projected for a complete 200-watt Fuel Cell Battery system.
2. This battery will operate for 24 hours daily at 35 ASF. With anticipated moderate improvement in current density further weight reduction becomes practical.
3. Operating periods up to 500 hours at 50 ASF have been demonstrated to date for cells using such thin air-depolarized cathodes.
4. The 200-watt UNION CARBIDE Fuel Cell Battery system featuring baked-carbon electrodes can be developed to a weight of 35 pounds when operability at 70 ASF is achieved.
5. The UNION CARBIDE Fuel Cell Battery with baked-carbon electrodes withstands severe shock and vibration conditions without physical damage or loss in operability. No difficulty is foreseen in meeting the usual military specifications for environmental conditions.
6. Various metal hydrides and borohydrides show promise as hydrogen sources, subject to the following considerations:
  - a) Weight
  - b) Cost - a negative factor for otherwise good materials.
  - c) Availability - related to cost and potential demand.
  - d) Safety - a factor in otherwise promising materials.
  - e) Freezing point - a limiting factor for reactant liquids.
7. Alkali hydrides react relatively safely with alkaline solutions such as the 12-15 molar alkalies electrolyte used in the UNION CARBIDE Fuel Cell Battery.
8. Yields from the above reaction are low when the reaction products are soluble, but the yield can be improved by addition of aluminum,  $\text{CoCl}_2$  or  $\text{NaCl}$ , or with fibrous extenders as described in this report.

9. Bottled gas supplies appear too heavy unless special designs of containers are used, not presently meeting ICC or ASME safety requirements.

10. Improvements in fuel cell technology justify a review of fuel generation from chemical sources, with emphasis on the gas-solid reactor, and on new compounds not previously available for study.

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## **VII. Recommendations**

Based on the findings outlined in this report which stem from the contract effort supported by independent work of the Consumer Products Company Research Laboratories, it is recommended that:

1. Inasmuch as improvement in electrode technology and by-product water handling makes a 35-pound, 200-watt fuel cell battery plant a very real goal, a new program be undertaken to expedite its development.
2. Effort should be directed to:
  - a) The further development of the "fixed-zone" electrode.
  - b) The adaptation of by-product water removal via vapor phase transpiration through the electrodes, preferably without requirement for electrolyte circulation;
  - c) The problem of effective air circulation in the cathode gas space;
  - d) Solution of the problem of parasitic currents for a field operated battery.
3. The feasibility of chemical sources of hydrogen should be reviewed. A program covering techniques, materials and hardware design is recommended.

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#### VIII. Key Technical Personnel

R. J. Bennett, Project Director. B. S. Ch. E. 1943. Fourteen years experience in engineering and systems development with Union Carbide Corporation, Research Laboratories, especially with proximity fuze battery design and pilot plant operation.

G. T. Jobe, Admn. Project Director. B. S. Electrochemistry 1943. Eleven years experience in battery product and process development, project management, and special assignments.

L. J. Uline, Group Leader and Designer. Mechanical Engineer. Thirty-three years experience in general plant engineering, engineering (proximity fuze), and project engineering (sodium-amalgam oxygen fuel cell).

B. P. Schofield, Development Engineering. B. S. Ch. E. 1943. Seventeen years experience chemical and metallurgical processing.

L. F. Urry, Acting Group Leader of Systems Research, B. A. Sc. degree 1950. Eleven years of experience with all types of batteries, both in production and research. During the last six years, his research work covered primary batteries, secondary batteries, and fuel cells.

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| <p>the current density demand imposed by the 35-pound battery weight stipulation. These determinations were conveyed to the U. S. Army Electronics Materiel Agency and agreement reached that since the prime objective of the contract was not within the state-of-the-art at that time, contract termination was in order to conserve residual funds.</p> <p>Concurrent work by the Research Laboratory of the Union Carbide Consumer Products Company during the later stage of the contract period was concerned with new electrode fabrication techniques which offered large reduction in weight and volume as well as the possibility of easier operation through simplification of by-product water removal. This work was called to the attention of the U. S. Army Electronics Materiel Agency and subsequently incorporated in a new proposal for the development of a lightweight, thin electrode making the 35-pound battery weight a sound technical goal.</p> | <p>the current density demand imposed by the 35-pound battery weight stipulation. These determinations were conveyed to the U. S. Army Electronics Materiel Agency and agreement reached that since the prime objective of the contract was not within the state-of-the-art at that time, contract termination was in order to conserve residual funds.</p> <p>Concurrent work by the Research Laboratory of the Union Carbide Consumer Products Company during the later stage of the contract period was concerned with new electrode fabrication techniques which offered large reduction in weight and volume as well as the possibility of easier operation through simplification of by-product water removal. This work was called to the attention of the U. S. Army Electronics Materiel Agency and subsequently incorporated in a new proposal for the development of a lightweight, thin electrode making the 35-pound battery weight a sound technical goal.</p> |
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