Hamilton Standard DIVISION OF UNITED AIRCRAFT CORPORATION

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FEASIBILITY STUDY OF A HUMIDITY CONTROL AND OXYGEN SUPPLY SYSTEM UTILIZING A WATER VAPOR ELECTROLYSIS UNIT

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for

National Aeronautics and Space Administration

Manned Spacecraft Center

Houston, Texas

FOREWORD

This report has been prepared by the Hamilton Standard Division of the United Aircraft Corporation for the National Aeronautics and Space Administration's Manned Spacecraft Center, in accordance with contract NAS 9-10773. This report covers work accomplished during two program phases:

Phase I - (May 1970 - August 1970)

A design study period to derive a water vapor electrolysis (WVE) cell concept which would tolerate a wide range (5% to 90%) of relative humidity and to devise a control system using this WVE design to maintain the spacecraft oxygen partial pressure.

Phase II - (September 1970 - April 1971)

A hardware design, manufacture and test period to verify the design concept which was selected in Phase I.

Personnel responsible for the conduct of this program were Mr. F. H. Greenwood, Program Manager, Mr. J. C. Huddleston, Engineering Project Manager and Dr. J. R Aylward, Technical Consultant from Hamilton Standard; and Mr. D. Price, Phase I Technical Monitor, Mr. A. Behrend, Phase II Technical Monitor and Mr. R. J. Gillen, overall program supervisor for the NASA Manned Spacecraft Center.

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ABSTRACT

This report presents the design and test effort involved in the development of an improved water vapor electrolysis cell for generating and controlling a spacecraft's oxygen and for supplementing a spacecraft's humidity control system. This program was conducted under contract number NAS 9-10773 by the Hamilton Standard Division, United Aircraft Corporation for the National Aeronautics and Space Administration, Manned Spacecraft Center.

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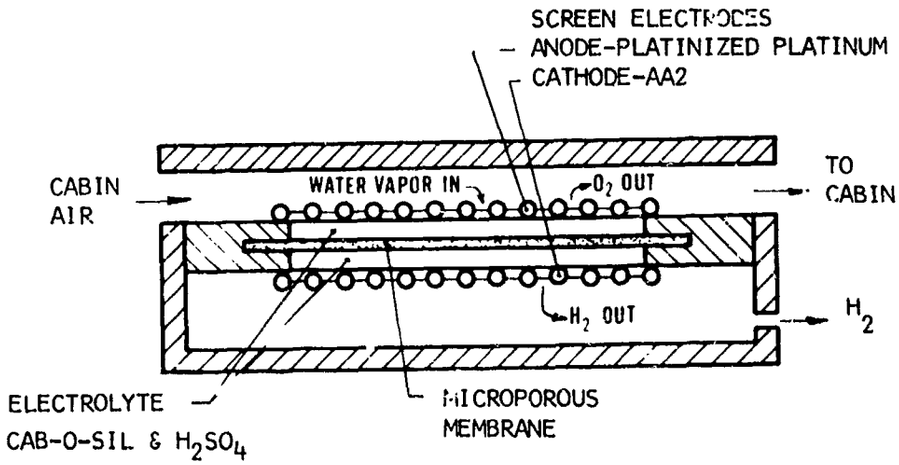
DEFINITIONS

Cell	Electrolysis cell consisting of an anode screen, matrix with electrolyte and cathode screen.
Cell Pair	Two cell packages with back to back cathodes, which share a common hydrogen chamber and housing assembly.
Dry Out	The condition of the cell, when the volume of the electrolyte is insufficient to completely fill the matrix due to loss of water.
Flooding	The condition of the cell when the electrolyte (acid) has absorbed an amount of water which results in an electrolyte volume exceeding the capacity of the cell matrix.
H_2 Crossover	Occurs at dry out of the matrix and allows hydrogen to pass through the matrix to the anode side.
Reservoir	A porous material which absorbs the excess electrolyte during cell flooding and returns it to the matrix during drying conditions.
Steady State Operation	The operating condition when the cell voltage and current do not change significantly with time.
Tafel Curve	A plot of cell voltage or electrode potential versus the logarithm of the current density.
Tafel Slope	The slope of the Tafel curve.
Capacity Factor	The maximum amount of electrolyte wicked from the reservoir by the matrix divided by the saturated capacity of the reservoir.

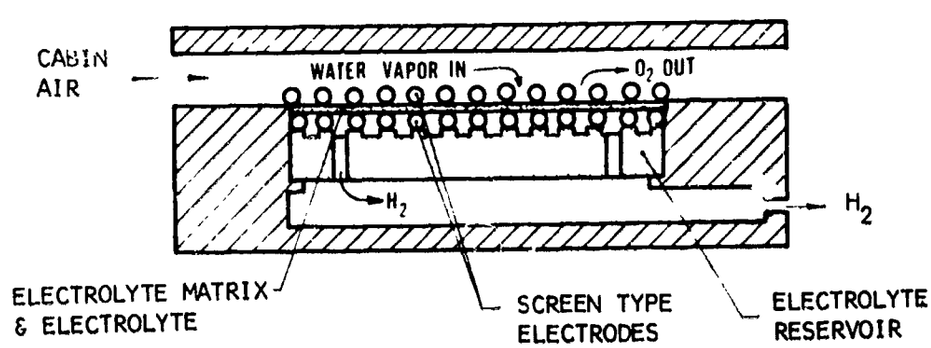
NOTE: Appendix A has a complete list of symbols and abbreviations.

SUMMARY

This program was conducted in two phases. The Phase I study effort was devoted to improvement of the basic WVE cell design of the NASA/Ames Research Center (Contract NAS 2-5699), which was limited to relative humidities of 35% to 65%. This improvement was mainly due to the addition of an electrolyte reservoir, illustrated in Figure 1, which allowed the cell to withstand operating conditions ranging between 5% and 75% R.H. Studies revealed that this improved WVE cell design, when operated under constant voltage conditions, could maintain the spacecraft oxygen partial pressure at 3.1 ± 0.2 psia.



NASA/AMES RESEARCH CENTER
WATER VAPOR ELECTROLYSIS CELL

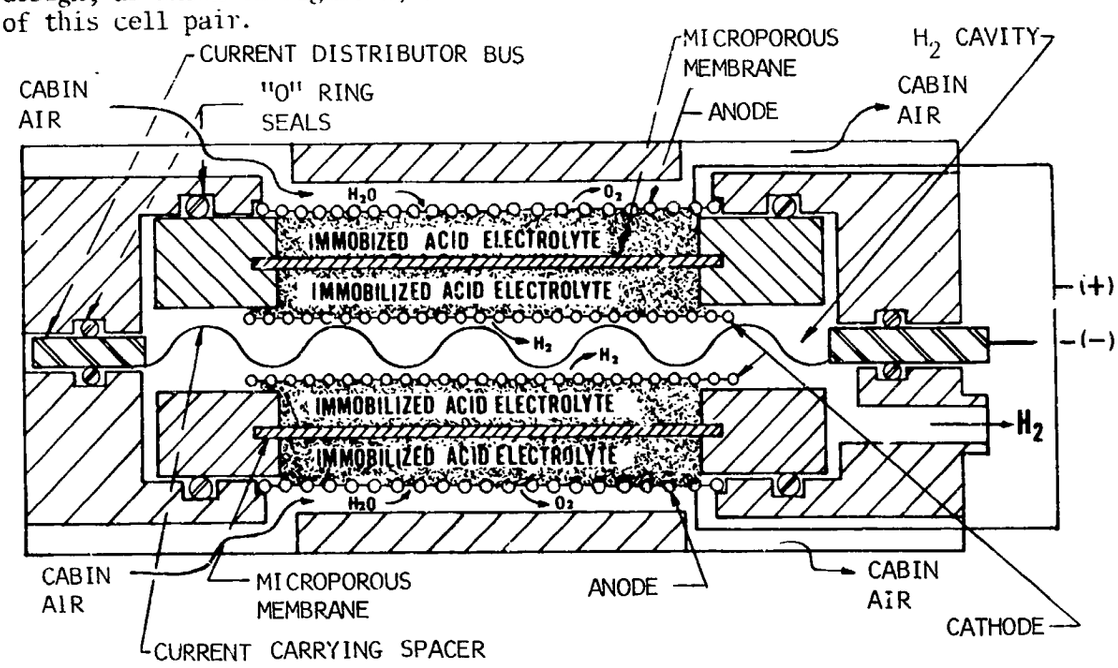


IMPROVED WVE CELL CONCEPT

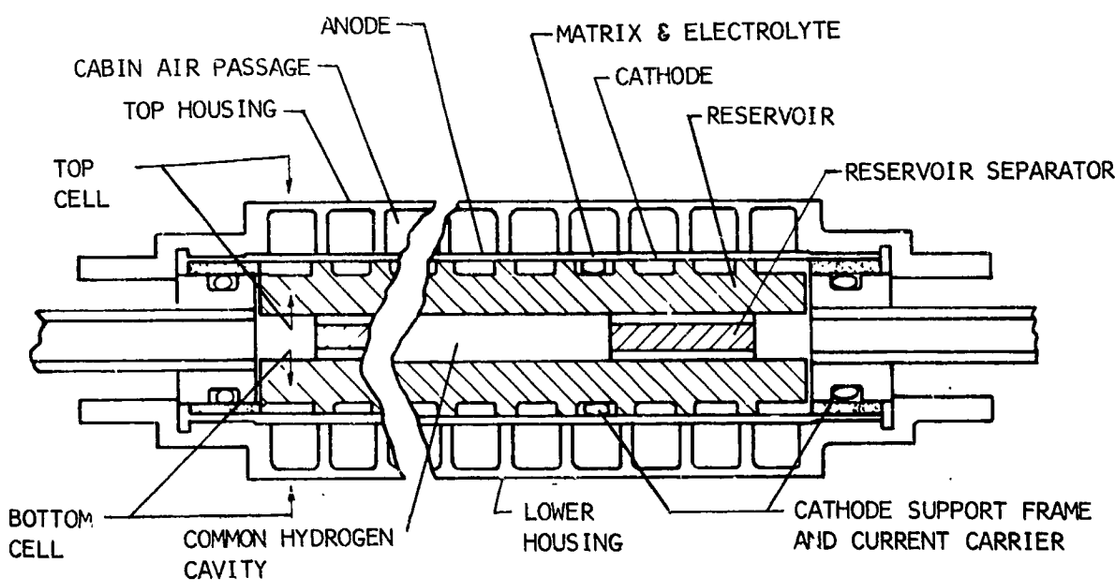
WVE CELL COMPARISON

FIGURE 1

The Phase II portion of this program modified the NASA ARC WVE cell pair design, as shown in Figure 2, and included the fabrication and the testing of this cell pair.



NASA/AMES CELL PAIR ASSEMBLY



WVE CELL PAIR ASSEMBLY

WVE CELL PAIR COMPARISON

FIGURE 2

The modified cell pair design provides a number of improvements over the present NASA/Ames Research Center cell pair. These improvements as defined below include a cell that is tolerable to a wider range in operating humidity, a decrease in cell voltage, an increase in operating current density, improvements in the materials of construction and a larger cell size. The new cell pair design accumulated 428 hours of continuous operation, during this phase.

Increased WVE cell tolerance to relative humidity: It was established that a WVE cell could be designed to operate over a relative humidity range of 5% to 90% R.H. by incorporating an electrolyte reservoir. This desired operational range was not achieved in the Phase II testing because the initial reservoir sizing data was inadequate, resulting in insufficient reservoir capacity. A range in humidity tolerance of 5% to 75% R.H. actually was obtained. Revised reservoir sizing data substantiates the actual test results for the reservoir which was used.

Decreased the cell operating voltage: A new anode catalyst and electrode structure developed by Hamilton Standard reduced the cell pair operating voltage from 2.1 volts at a current density of 30 amps/ft^2 to 1.85 volts at a current density of 37.0 amps/ft^2 (which is equivalent to 1.66 volts at a current density of 30 amps/ft^2).

Increased the cell pair current density: The improved cell pair design can be operated at higher current densities than the NASA/ARC cell because of lower heat generation rates and more efficient heat transfer. A current density of 90 amps/ft^2 at voltage levels below 2.0 volts was achieved.

Improved the cell pair size and materials: The improved cell pair design replace the polyvinylchloride (P.V.C.) material with titanium and increased the cell pair size from an effective area of 1/6 ft^2 to 1 ft^2.

CONCLUSIONS

The results of this program effort to determine the feasibility of a Water Vapor Electrolysis (WVE) System that is capable of providing the required spacecraft oxygen while operating over a wide range of humidity (5% to 90% R.H.) lead to the following conclusions:

The WVE cell design concept established on this program will operate over a wider range of humidity than previous WVE cell designs. Reasons for this improvement are:

a. An electrolyte reservoir which accommodates for the change in electrolyte volume over a wide range in relative humidity.

b. A thinner matrix which reduces the minimum necessary electrolyte volume in the cell.

The WVE cell pair unit which was tested fell short of operating over the desired humidity range of 5% to 90% relative humidity because of reservoir sizing. However, it did demonstrate the feasibility of it's design concept.

A reservoir capacity factor which corrects for the residual electrolyte must be considered for proper sizing. This factor varies with the wetting characteristics, internal geometry and configuration of the reservoir.

A six (6) inch air passage length over the anode is feasible. This conclusion is based on an analysis of the cell mass transfer processes under normal operating conditions and was also confirmed by the subsequent test results.

The new anode catalyst and electrode structure developed by Hamilton Standard for improving the performance of the Phase II cell was successful in reducing the operating voltage from 2.1 volts at a current density of 30 amps/ft^2 to 1.85 volts at a current density of 37. amps/ft^2 (which is equivalent to 1.66 volts at a current density of 30 amps/ft^2).

The cell electrolyte of sulfuric acid (H_2SO_4) provides better performance than phosphoric acid (H_3PO_4) or a mixture of the two acids. However, a cell which uses H_2SO_4 electrolyte must have the hydrogen gas removed from the cathode upon cell shutdown to prevent hydrogen sulfide (H_2S) evolution in the cell.

The WVE system concept of this program will maintain the required spacecraft oxygen partial pressure over a wide range of humidity. This system approach is:

a. Operation of the WVE cells at constant voltage. This utilizes the inherent self compensating nature of the cell and minimizes the effect of changes in humidity.

b. Provision for multiple (three) operating voltage levels. This will allow the cell to provide oxygen at the desired rate as determined by the crew or an automatic control system.

The cell pair performance after 428 hours of continuous operation provided the following steady state values:

Oxygen Production = 0.471 #/day/cell pair at 1.66 volts (112 scc/min)
Hydrogen Production = 0.0591 #/day/cell pair at 1.66 volts (224 scc/min)
Cell Pressure Drop = 1.8 inches of water at 18 SCFM air flow
Operating Voltage = 1.66 volts at 30 amps

RECOMMENDATIONS

The study and test results of this program evolved the following recommendations:

A WVE cell pair configuration per Figure 2 is recommended for use under widely varying humidity conditions. The test results proved the ability of this design concept to operate over a humidity range of 5% to 75% relative humidity. Analysis shows that with proper sizing the unit could operate over a 5% to 90% relative humidity range.

The improved anode which was developed by Hamilton Standard is recommended for use in future cell designs.

Sulfuric acid (H_2SO_4) is recommended as the WVE electrolyte since test results show this acid provides the better performance characteristics.

A nitrogen gas purge system on the cathode side of the cell is recommended to prevent the evolution of H_2S.

Additional study and test effort should be performed in the following areas:

a. Investigation of reservoir materials to achieve a larger void volume (60% min.), increased structural strength and lower weight. Both electrical insulator and non-insulating materials should be considered.

b. Improvement in the cathode current carrier to provide a more uniform current distribution.

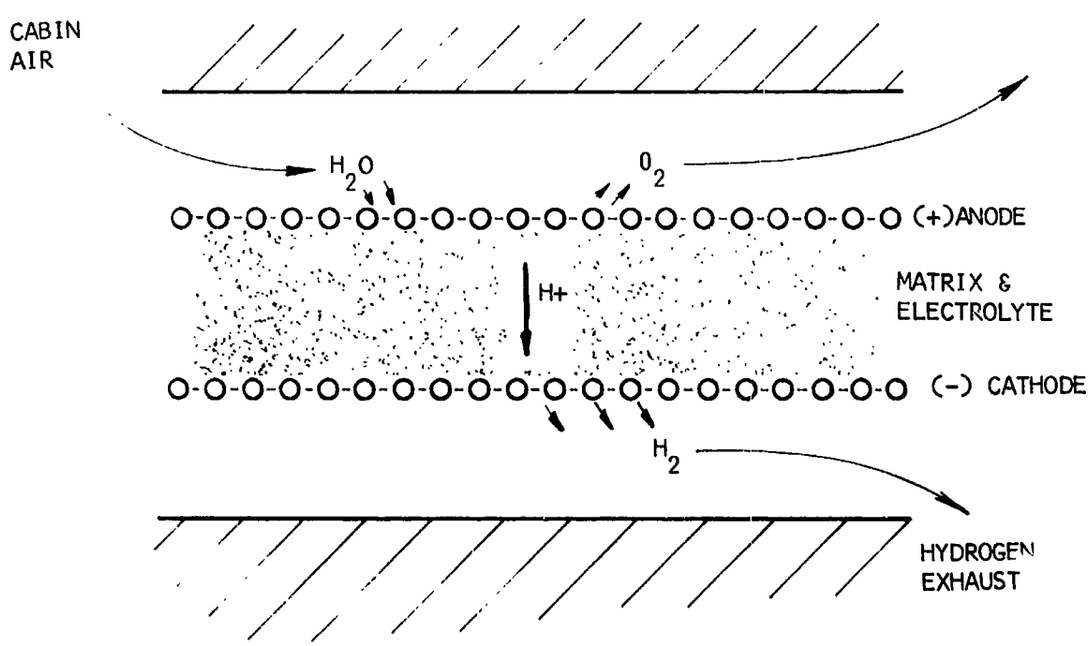
c. Additional cell pair testing to further examine endurance capability, on-off cyclic operation, repeated wetting and drying cycles, and a complete temperature/humidity performance map.

DISCUSSION

The discussion of the results of this program is separated into three sections. The first section, WVE Cell Operation, presents a general description of how the Water Vapor Electrolysis cell operates. The second section, Phase I Studies, discusses feasibility tests results, conclusions and the recommendations that were formed during this program phase, regardless of later Phase II results. The last section, Phase II Test, presents the Phase II cell pair design, various test results and data analysis that were obtained during this period.

WATER VAPOR ELECTROLYSIS CELL OPERATION

The WVE cell, schematically illustrated in Figure 3, operates as described below.



WATER VAPOR ELECTROLYSIS CELL SCHEMATIC

FIGURE 3

When D.C. power is applied to the electrodes a current passes through the cell electrolyzing the water in the electrolyte, liberating oxygen at the anode and hydrogen at the cathode. The electrolyte is contained and immobilized within the matrix.

Make up water is added to the cell by passing moist cabin air over the anode. The water vapor in the incoming air is absorbed by the electrolyte because of the lower vapor pressure within the electrolyte.

As the relative humidity changes a corresponding change also occurs in the electrolyte concentration and volume. On increasing humidity and/or decreasing air temperature the electrolyte becomes less concentrated, increasing its volume causing the cell to flood with excess electrolyte. On decreasing humidity and/or increasing air temperature the electrolyte becomes more concentrated, decreasing in volume causing the matrix to dry out. For this reason the reservoir concept was added to the WVE cell to absorb the excess electrolyte (preventing cell flooding) and to allow the matrix to wick the electrolyte back during drying conditions.

In any given cell, the rate of electrolysis depends solely on the current passed through the cell, illustrated as follows:

For a current of one ampere;

Oxygen produced	= 0.0157 lb/day = 3.74 scc/min.
Hydrogen produced	= 0.00197 lb/day = 7.48 scc/min.
Water vapor consumed	= 0.0177 lb/day

The oxygen required for one man is approximately 2 lbs/day which would require a WVE system to operate at 127 amps. The minimum theoretical voltage required for electrolysis of water is 1.23 volts if the process is conducted at a net rate of zero. Actual voltages for WVE cells, studies in Phase I, ranged from 2.1 to 2.5 volts depending on the electrolyte, matrix, electrodes and electrolysis rate (of current density approximately 30 amps per ft^2). The new anode developed by Hamilton Standard and employed in the Phase II cell design, lowered this voltage to 1.85 volts at a current density of 37.0 amps per ft^2 (which is equivalent to 1.66 volts at 30 amps per ft^2). Differences between actual and theoretical voltages are caused by over voltage primarily at the anode, and by cell resistance effects.

PHASE I STUDIES

The objective of this phase was to investigate a Water Vapor Electrolysis (WVE) unit which would be tolerant to a range of relative humidity from 5% to 90%. A further objective was to determine a control system which would utilize this improved WVE unit and would maintain the space cabin oxygen partial pressure at 3.1 $\pm$ 0.? psia.

A trade-off study was made to establish and select an improved WVE cell design concept and oxygen control system. This trade-off study was derived from data obtained through the definition of operating requirements, research of existing data, feasibility tests and analytical studies. Each of these subtasks is discussed in detail in the following sections.

The results of the trade-off study revealed that a WVE cell containing an electrolyte reservoir could be made operable over a humidity range of 5% to 90% R.H., provide the required oxygen and maintain the oxygen partial pressure at 3.1 $\pm$ 0.2 psia with space cabin air humidities between 35% to 65% R.H. without flooding or drying out. The WVE unit would be operated at a controlled voltage with a manual or automatic voltage level selection of minimum, nominal and maximum.

Requirements

The following tabulation provides a comparison of the performance requirements that have been established for this program as compared to those of the present NASA/Ames Research Center WVE Unit:

	Requirement	Improved WVE Unit	Present NASA/ARC WVE Unit
1.	Gravity - Oper.	0 to 1G	0 to 1G
2.	Ambient Press. - Oper.	7.5-10.0-16.0 psia	7.5-10.0-16.0 psia
3.	Air Temperature	63° to 77°F	65° - 85°F
4.	Relative Humidity - Oper.	Normal 35% to 65%	35% - 65%
5.	Weight Penalties- Power at 28 VDC	591 lb/KW	290 lb/KW
	Power at 115 VAC	710 lb/KW	TBD
	Heat Rejection	.02 lb/BTU/hr.	0.1 lb/BTU/hr.
6.	Oxygen Production (lbs/man/day)	1.18 to 3.125	2
7.	Oper. Life w/o Maintenance	2160 hrs. (min)	2000 demonstrated
8.	Total Life	2 years	6 months
9.	Relative Humidity - Oper. (as a design goal)	5% to 90%	35% to 65% demonstrated
10.	Oper. after Vacuum Exposure	0.1 psia for 4 hrs.	10^{-2} torr-but w/ unit sealed

	Requirement	Improved WVE Unit	Present NASA/ARC WVE Unit
11.	Crew Size	12	1
12.	H_2O Available (lbs/man/day)	1.2 - 3.09 - 9.6	TBD
13.	CO_2 in Air (lbs/man/day)	1.98 - 2.12 - 3.1	TBD
14.	O_2 Partial Pressure	3.1 $\pm$ .2 psi	TBD
15.	Cabin Volume	6000 cu ft	TBD
16.	Air Flow	TBD	60 CPM
17.	Storage Environment	5% to 80% RH	at 100% R.H. with unit sealed
	Ambient Pressure	14.7 psia	14.7 psia
18.	Cell Pressure Drop	TBD	2 inches H_2O max
19.	Hydrogen Production (lb/day/man)	0.14 - 0.39	0.25 (min)

Data Research

A review of the work on water vapor electrolysis (see references, Appendix B) was conducted to define the problems associated with present design. A brief summary of this evaluation is given below.

The water vapor electrolysis cell for oxygen generation in spacecraft was developed in two concurrent efforts. A cell employing phosphoric acid electrolyte in a combination microporous rubber and asbestos matrix was developed by Battelle Memorial Institute (BMI) under contract with NASA/ARC (Contract NAS 2-2156). Platinum screens were used for both electrodes in the phosphoric acid cell. In a parallel effort, NASA/ARC developed a cell utilizing sulfuric acid electrolyte, immobilized with Cab-O-Sil to form a gel, supported by a microporous polyvinyl-chloride (PVC) membrane for gas separation. This cell used a platinum screen for the anode and an American Cyanamid AA-2 fuel cell electrode for the cathode. Both cells utilized rigid (unplasticized) PVC housings. The feasibility of the WVE concept was demonstrated by extended testing of laboratory modules (cell stacks) of both the sulfuric and phosphoric acid cells.

The use of plastic (polyvinylchloride) for the cell housing is disadvantageous because the low strength and thermal conductivity limit the individual cells to a relatively small size, and separate provisions must be made for current collection. The cell also employs "O" ring seals which complicate construction.

The choice of anode catalyst and structure in the NASA/ARC cell gave excessive polarization which limits the cell to low current densities in order to prevent ozone production.

A gel electrolyte may be sensitive to vibration and makes cell assemblage somewhat arduous, while the microporous membrane separator gives a significant increase in cell resistance. The relatively large electrolyte volume in this design would be expected to result in low tolerance to flooding or drying, however, the response time would be small. The asbestos matrix with phosphoric acid does not have these problems but the chemical compatability of these two materials is questionable.

A sulfuric acid electrolyte gives much better electrochemical performance than phosphoric acid, and remains liquid under normal operating conditions in contrast to phosphoric acid which forms a solid phase at concentrations above 85 wt-% near ambient temperature (68°F). Sulfuric acid has the disadvantage that it is thermodynamically unstable toward reduction and under certain operating conditions hydrogen sulfide can be evolved at the cathode. Other chemical properties of these acids are given in Appendix C.

Improvement of the water vapor electrolysis unit required solutions to the above problems. Various possible solutions were investigated and the results are presented in the appropriate sections of this report.

Feasibility Tests

A survey of available test results on the primary electrolyte candidates H_3PO_4 and H_2SO_4 led to the conclusion that sufficient comparative data was not available to adequately relate these electrolytes or to properly design a WVE cell for expanded humidity tolerance. Accordingly, an electrochemical test program was conducted in which cell candidate designs and configurations could be tested on common instrumentation. Test objectives were:

To provide a cell design that would permit operation over a wide range of inlet humidity.

To select a cell matrix.

To select a cell electrolyte.

In order to compare the various cell configurations and electrolytes it was necessary to obtain polarization curves, (cell voltage versus current density) and cell resistance at various electrolyte concentrations, corresponding to electrolyte equilibrium at various inlet humidities (Reference Appendix D).

Initial testing was conducted on one inch square WVE cells having the same electrolyte and matrix as previously tested by Battelle Memorial Institute under contract NAS 2-2156 and NASA/ARC contract NAS 2-5699. A new matrix material, Tissuquartz, was also tested in these small test cells. From the results of this testing, Tissuquartz was selected as the matrix material. Tissuquartz was selected because it provides a thin matrix, reduces the cell sealing problems and has good operational history as a Pratt & Whitney Aircraft fuel cell matrix.

Since it was very time consuming to equilibrate the small WVE cells at various humidities, a "half cell" or bulk electrolyte test program was also conducted. In this testing the electrolyte could be prepared to represent the equilibrated condition in a cell at a specific humidity and tested in a much shorter time.

The electrolyte test results revealed that H_2SO_4 provides excellent electrochemical performance. However, fuel cell test reports by the Pratt & Whitney Aircraft Division of UAC indicated that H_2SO_4 can be reduced to produce H_2S under certain conditions. Because of this one disadvantage a mixed acid (H_2SO_4 + H_3PO_4) electrolyte was tested. It was believed that this mixture would reduce the possibility of H_2S production because of the lower concentration of H_2SO_4.

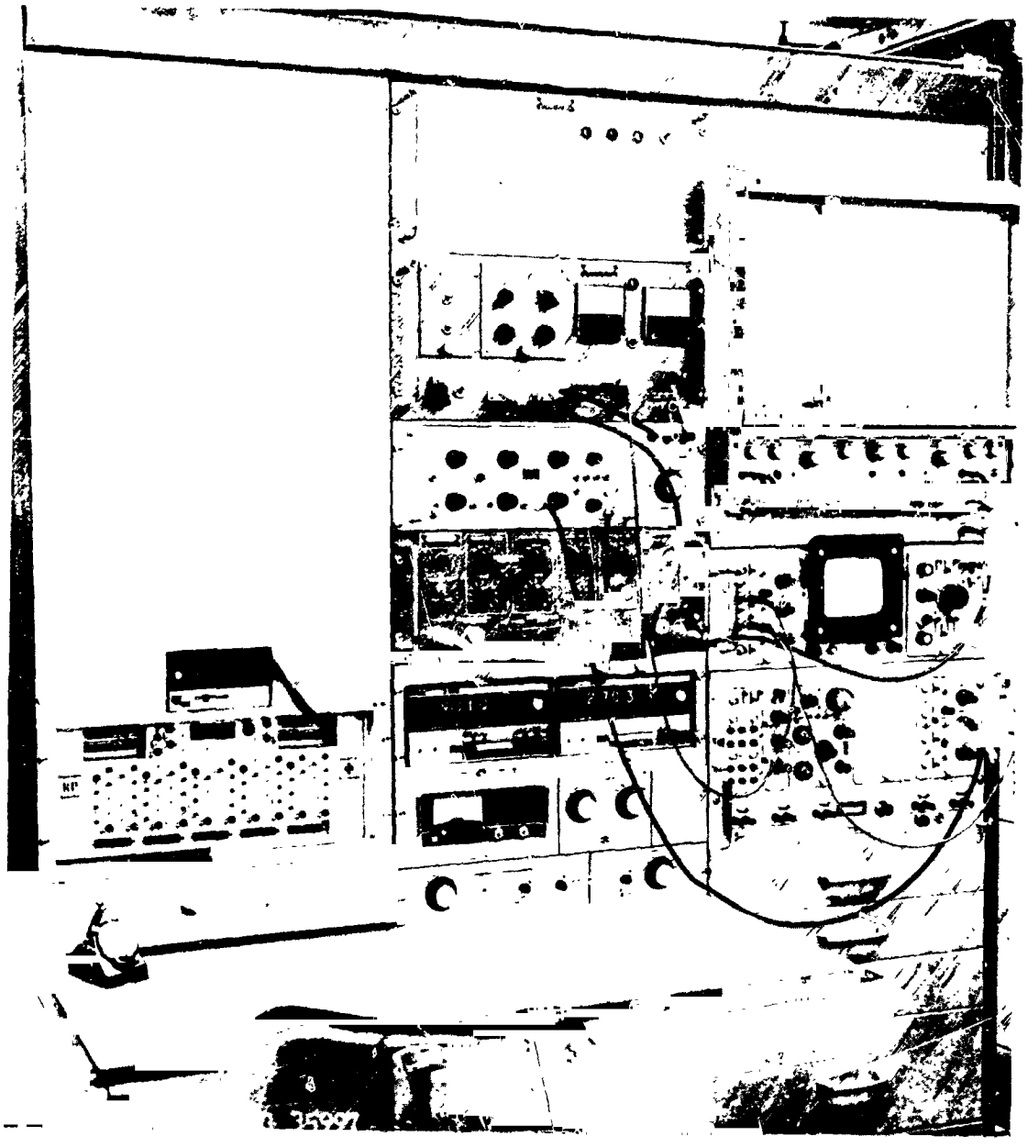
From studying the basic WVE cell performance and the variation in electrolyte volume with ambient humidity, it was established that the WVE cell should incorporate a reservoir for the electrolyte to flow into and out of in order to withstand the specified 5% to 90% range in relative humidity conditions. Testing of the mixed acid in a WVE cell which incorporated a reservoir was performed. Selection of the reservoir material resulted from a "wicking" test program which compared various materials for their wicking action and acid compatibility.

Detailed discussions of the various tests associated with the matrix and electrolyte investigations are presented in the following sections:

Test Apparatus
Small Cell Tests
Half Cell Tests
Cell Tolerance Tests
Mixed Acid Electrolyte
Wicking Tests

Test Apparatus

The electrochemical studies on the 1 x 1 inch cell and half cell were carried out with a fast rise-time Tacussel model PIT 20-2X potentiostat and associated equipment shown in Figure 4. Current-voltage curves were run at a sweep rate of 200 mv/min. The total number of



Operating Console For Water Vapor Electrolysis Tests

Figure 4

coulombs passed in all cases was equivalent to the consumption of less than 5% of the total water contained in the equilibrated electrolyte at various set humidities. The electrolyte voltage drop (IR) was measured by the constant current pulse method with a pulse rise-time of less than 10 micro seconds.

A typical recorder trace showing the voltage/current curve and its correction for IR drop is given in Figure 5 for a 1 x 1 inch cell test.

Small Cell Tests Without a Reservoir

These tests were run to compare the performance of various candidate matrix materials and electrolytes. Small WVE cells were prepared and placed in a humidity control apparatus shown in Figure 6. Electrolyte resistance and Tafel slopes were obtained to determine the electrochemical performance. The following cells were tested at various inlet dew points:

	Acid Concentration (WT-%)	Electrolyte/Matrix
1.	56	H_2SO_4/Cab-O-Sil(1) (NASA/ARC type cell) 10 Wt parts acid/1 Wt Cab-O-Sil
2.	85	H_3PO_4/Asbestos (Battelle type cell)
3.	56	H_2SO_4/Tissuquartz
4.	85	H_3PO_4/Tissuquartz

The cells were equilibrated for at least two hours at each dew point condition. The reproducibility and consistency of results indicated that vapor pressure equilibrium was achieved.

A summary of the test results is presented in Table I. The cell having a gel of Cab-O-Sil/H_2SO_4 and a microporous separator had four times the resistance of the same unit with a matrix of Tissuquartz/H_2SO_4. The cell having a matrix of asbestos/H_3PO_4 had approximately 1.3 times the resistance of the same unit with a matrix of Tissuquartz/H_3PO_4. In addition to having a lower resistance with a given electrolyte, the Tissuquartz has superior physical and chemical properties for this application. It was also confirmed that sulfuric acid gives better electrochemical performance than phosphoric acid in terms of both a larger exchange current and a lower electrolyte resistance. The cell configuration for these tests showed poor tolerance in that it was very sensitive to dry out. Recovery from a dry condition was also difficult.

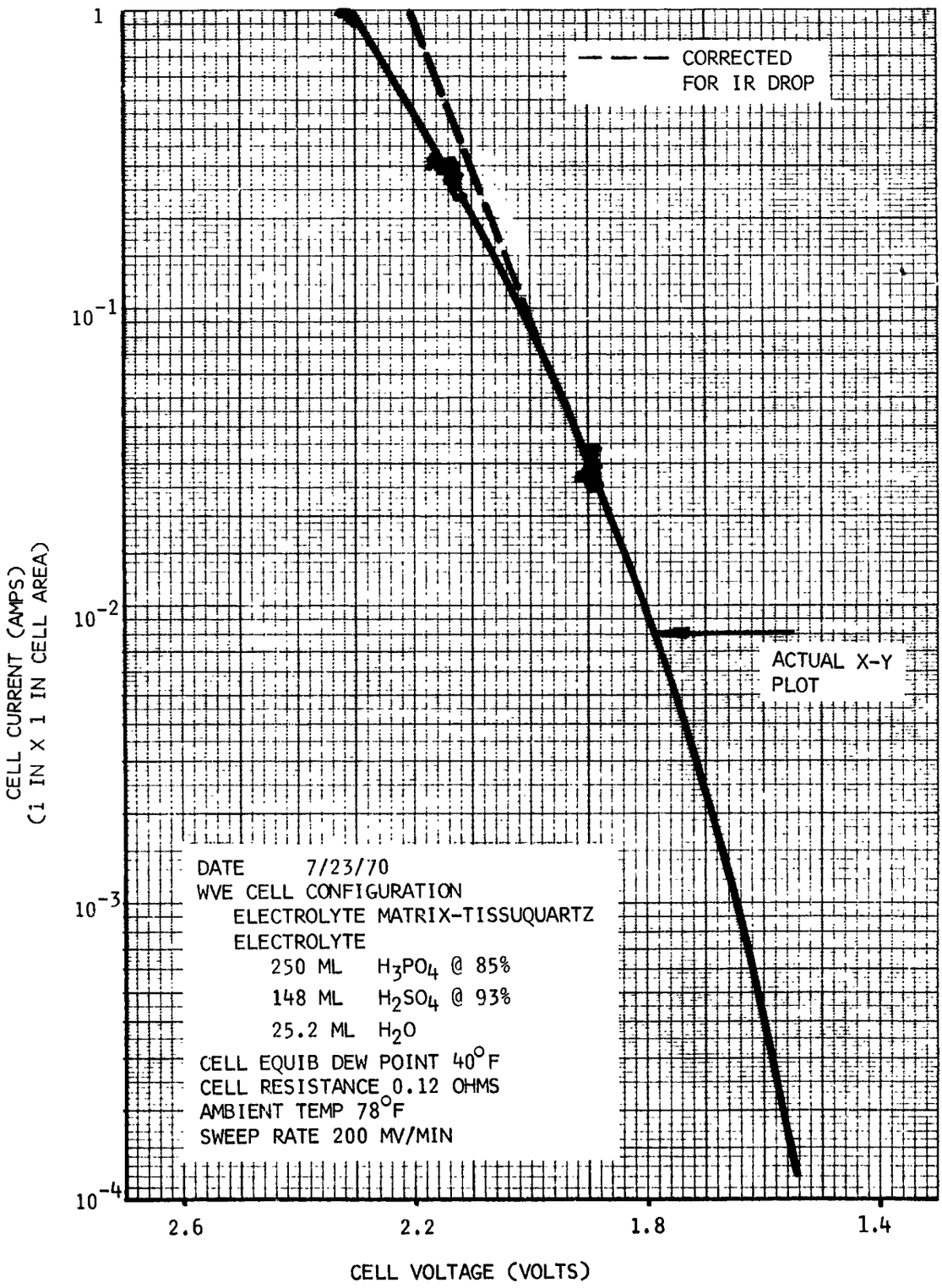

CORRECTED FOR IR DROP
ACTUAL X-Y PLOT
DATE 7/23/70
WVE CELL CONFIGURATION
ELECTROLYTE MATRIX-TISSUQUARTZ
ELECTROLYTE
250 ML H_3PO_4 @ 85%
148 ML H_2SO_4 @ 93%
25.2 ML H_2O
CELL EQUIB DEW POINT 40°F
CELL RESISTANCE 0.12 OHMS
AMBIENT TEMP 78°F
SWEEP RATE 200 MV/MIN
1
10^{-1}
10^{-2}
10^{-3}
10^{-4}
CELL CURRENT (AMPS)
(1 IN X 1 IN CELL AREA)
2.6
2.2
1.8
1.4
CELL VOLTAGE (VOLTS)

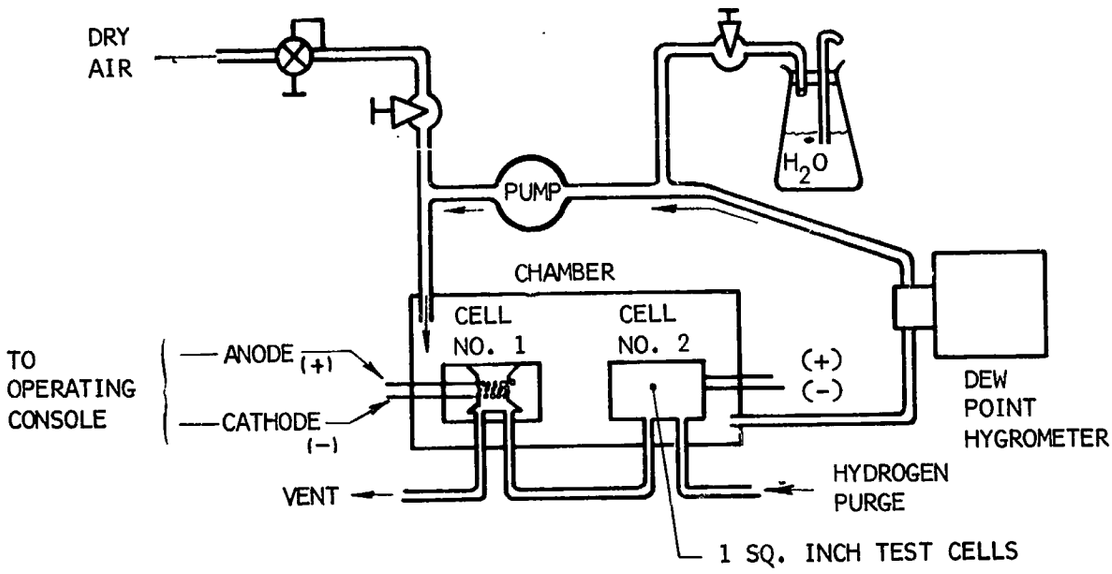
FIGURE 5

TABLE I

WVE CELL TOLERANCE TEST SUMMARY

Cell Configuration Reference Appendix - Test Plan		Electrolyte Vapor Pressure (mm Hg)	Cell Resistance (OHMS)	Tafel Slope (Volts/Decade)	Current Density At 1.8 Volts (Amps/Sq Ft)	Matrix Thickness (Inches)
H_2SO_4/Cabosil		17.0	0.12	0.16	.210	.0625
(NASA/ARC Config.)		9.8	0.23	0.15	.111	.0625
(w/o reservoir)	(3)	5.0				
H_3PO_4/Asbestos		17.0	0.4	0.21	.370	.010
(BMI Config.)		9.8	0.9	0.29	.123	.010
(w/o reservoir)	(3)	5.0				
H_2SO_4/Tissuquartz		9.2	0.03	0.13	.111	.017
(w/o reservoir)		6.0	.041	0.16	.123	.017
	(1)	4.2	0.12	0.32	.160	.017
	(2)	11.5				
H_2PO_4/Tissuquartz		11.5	0.16	0.16	.067	.017
(w/o reservoir)		9.2	0.18	0.14	.062	.017
		6.2	0.20	0.22	.016	.017
	(1)	4.2	0.25	0.34	.0074	.017
H_2SO_4 + H_3PO_4/Tissuquartz plus reservoir		See Figures 9, 10 and 11				

NOTE: (1) Cell indicated drying out.
(2) Cell performance after being dried out. (test data not valid for comparison)
(3) Cell dried out - would not operate.



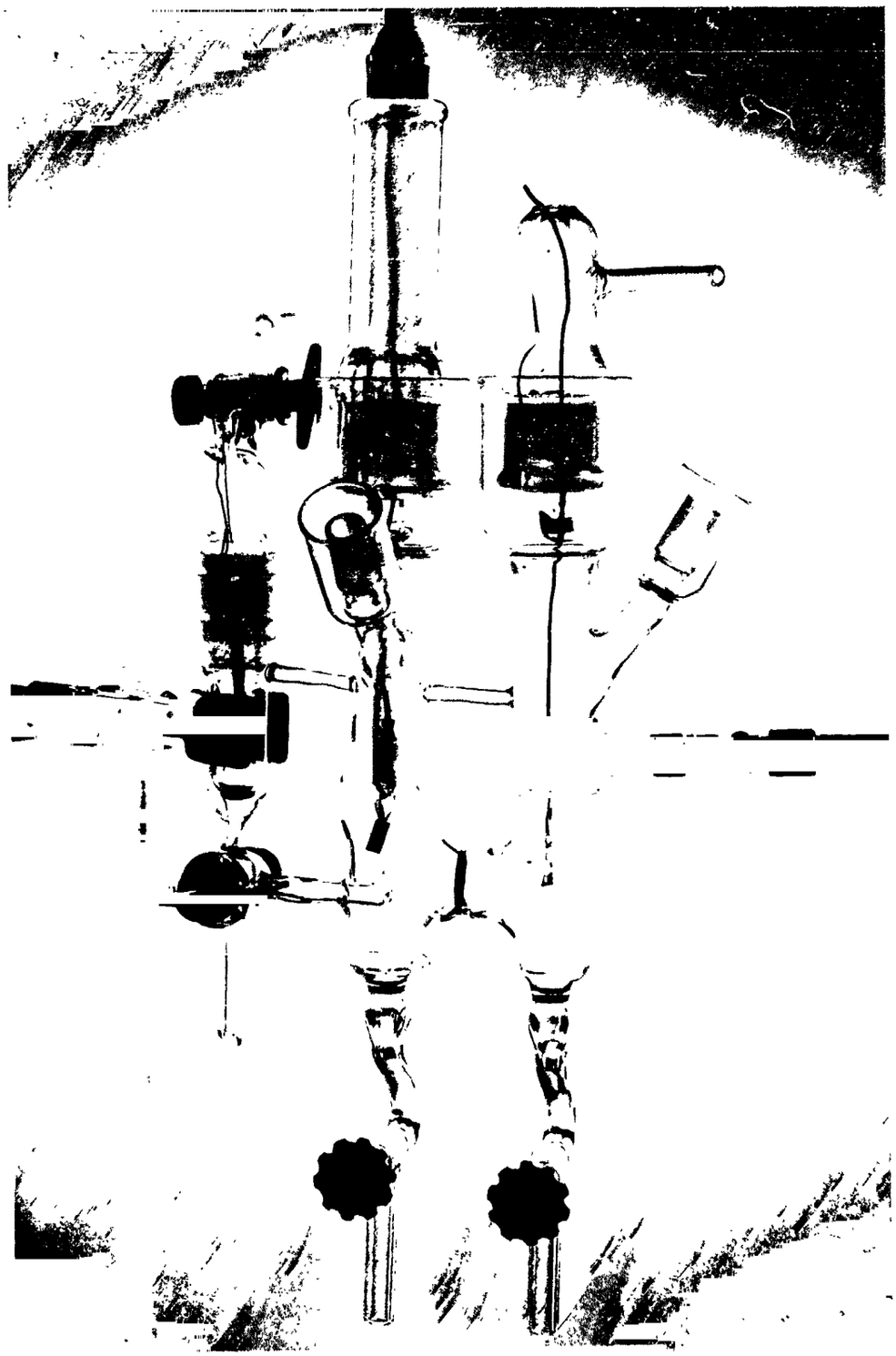
TEST SET-UP FOR 1 INCH TEST WVE CELLS

FIGURE 6

Half Cell Tests Using Bulk Electrolyte

Experiments were conducted in a half cell pictured in Figure 7 to determine the effect of the equilibrium water vapor pressure of the electrolyte (P_V) on the anodic current density at constant potential. Experiments also were directed at comparing the performance of mixed H_2SO_4 + H_3PO_4 electrolytes with individual acids. (The arguments supporting a mixed electrolyte are summarized in section "Mixed Acid Electrolyte".)

The working electrode (anode) was a smooth platinum one cm^2 flag-type strip which was rotated at a speed of approximately 600 rpm to minimize concentration polarization. Potential sweeps were run at 200 mv/min for various acid concentrations. The mixed electrolyte consisted of H_3PO_4 and H_2SO_4 in a 7 to 5 weight ratio with various amounts of water. This choice in mixture ratios was based on an initial mixture of equal volumes of 65 wt-% H_2SO_4 and 85 wt-% H_3PO_4 which have the same vapor pressure. With this mixture a total acid concentration of 95 wt-%



SS 9723-8

Half Cell Test Fixture

Figure 7

(P_V = 0.6 mm Hg) would still contain less than 40% H_2SO_4 so that the possibility of H_2SO_4 being reduced in this mixed electrolyte is very slight (see comments in section Mixed Acid Electrolyte).

Theoretically the anodic current at constant potential (I_V) should be related to the vapor pressure of water in equilibrium with the electrolyte (P_V) by an equation of the form $I_V = k\ P_V^n$ where k is the rate constant whose value depends only on potential, temperature, type of anion and the catalytic activity of the electrode. The exponent n depends only on the reaction mechanism which in turn may be influenced by the nature of the anions present in solution via anion absorbtion.

From the results of the above tests, summarized in Figure 8, it can

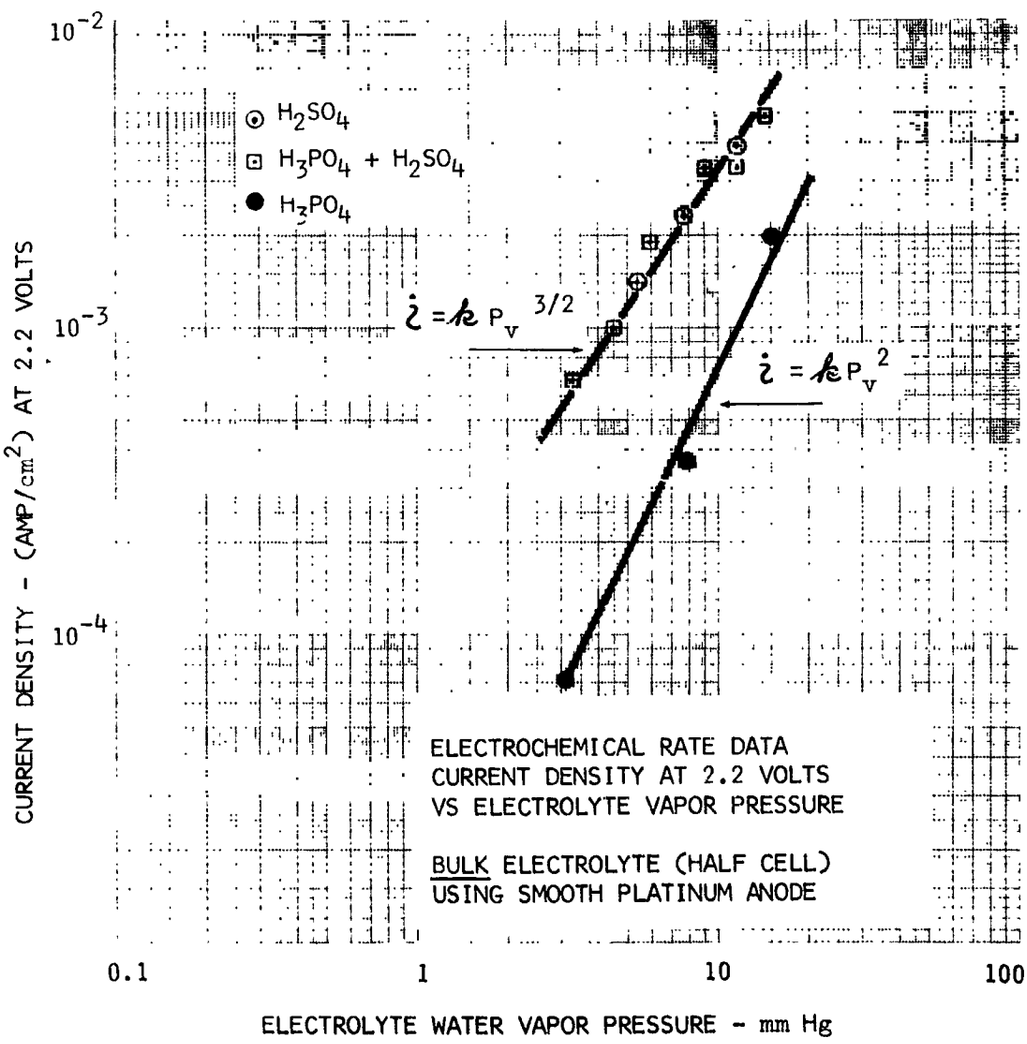


FIGURE 8

be seen that the rate constant k for the sulfuric acid is significantly larger than that for phosphoric acid. This means that at a particular dew point sulfuric acid is more efficient than phosphoric for water electrolysis. The respective exponents for sulfuric and phosphoric acid are 3/2 and 2. The initial Phase I work indicated that the mixed acid had the same electrochemical performance as sulfuric acid. This conclusion was later reversed as a result of Phase II testing.

Cell Tolerance Tests Using The 1 x 1 Inch WVE Test Cell

Additional cell tests with the porous glass reservoir configuration, illustrated in Figure 1, were conducted with the mixed acid(1) in order to determine the overall cell performance. The cell matrix was filled with a slight excess of mixed acid whose total acid concentration was 85 wt-% (P_V = 1.5 mm Hg), to represent a very dry condition of cell operation (low humidity). The assembled cell was then equilibrated with air at various dew points. The increase in electrolyte volume upon equilibration with $P_{H_2O} > 1.5$ mm Hg was taken up by the glass sponge.

The first test with a shiny platinum screen anode gave very high polarization indicative of an extremely dry condition at the anode or very high anode current density. Subsequent examination of the anode showed that it was too dry due to competition from the sponge for electrolyte. This situation was remedied by platinizing the anode screen, the platinum black acting as a sponge with very small pores to hold sufficient electrolyte under all conditions.

With this modification the cell performance and tolerance were improved. Successful performance was achieved over a water vapor pressure range from 2 mm Hg to 19 mm Hg (130 to 70°F electrolyte dew point) with no evidence of performance degradation at the extreme conditions. These test results are summarized in Figures 9, 10, and 11. Figure 9 shows the current density as a function of cell voltage (corrected for IR drop) at various electrolyte water vapor pressures, P_V. These results showed no evidence of concentration polarization up to current densities of 160 amp/ft^2 as would be expected from theoretical considerations. The cell "Tafel Slope" was 0.20 volts/decade. This value for the cell Tafel Slope is difficult to explain since the half cell tests using the anode with free electrolyte gave a more normal Tafel Slope of 0.12 volts. The difference of 0.08 volts would have to be attributed to the cathode polarization, but seems high by a factor of two for this type electrode. The anode polarization for water electrolysis from acid on platinum electrodes seemed excessively high and undoubtedly

(1) This type of cell design is not limited to a mixed acid electrolyte.

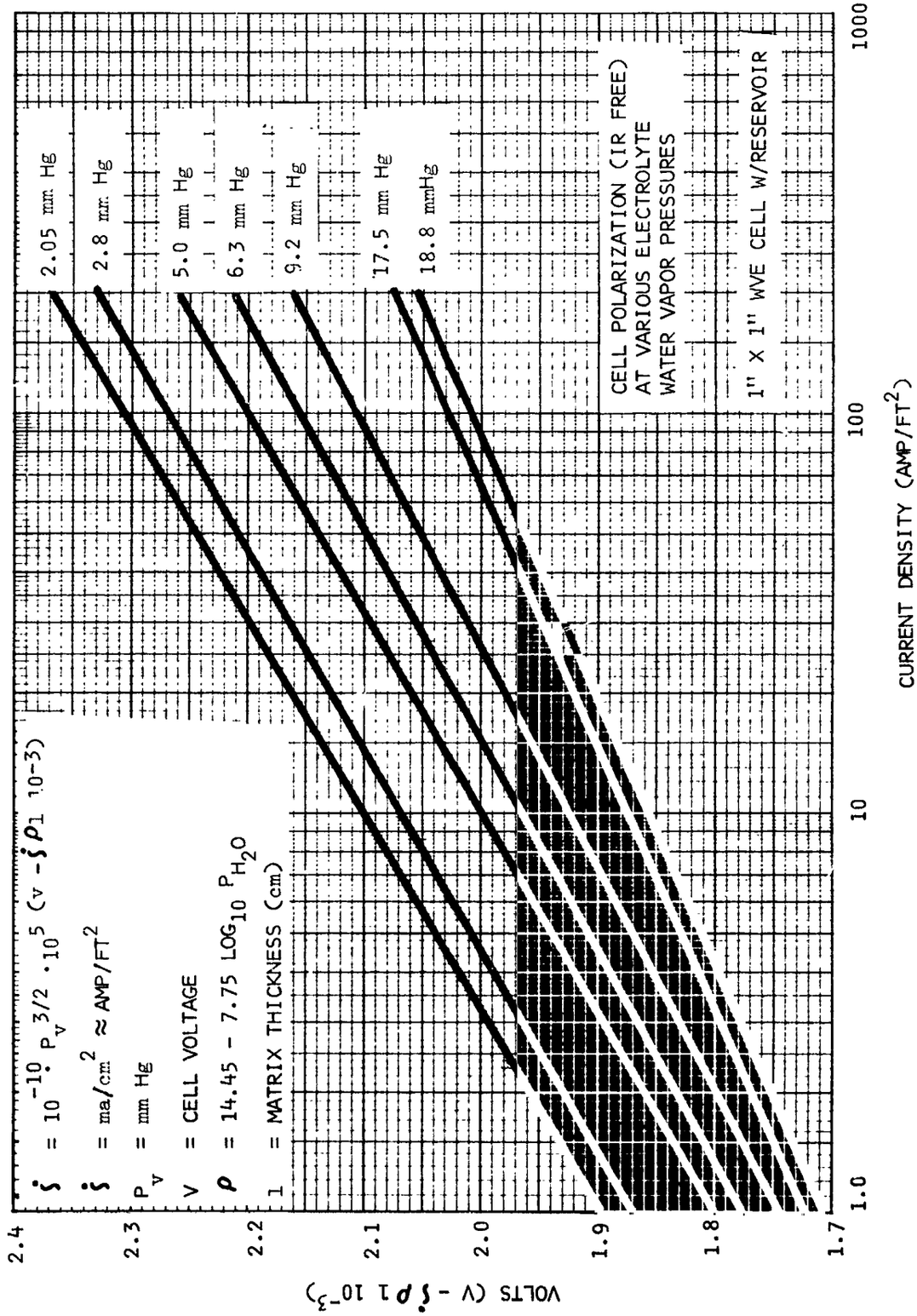

$\dot{\zeta}$ = $10^{-10} \cdot P_v^{3/2} \cdot 10^5 (v - \dot{\zeta}\rho 1\ 10^{-3})$
$\dot{\zeta}$ = ma/cm^2 ≈ AMP/FT^2
P_v = mm Hg
V = CELL VOLTAGE
ρ = 14.45 - 7.75 LOG$_{10}$ P_{H_2O}
1 = MATRIX THICKNESS (cm)
2.05 mm Hg
2.8 mm Hg
5.0 mm Hg
6.3 mm Hg
9.2 mm Hg
17.5 mm Hg
18.8 mmHg
CELL POLARIZATION (IR FREE)
AT VARIOUS ELECTROLYTE
WATER VAPOR PRESSURES
1" X 1" WVE CELL W/RESERVOIR
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.0
10
100
1000
VOLTS (V - $\dot{\zeta}\rho$1 10^{-3})
CURRENT DENSITY (AMP/FT^2)

FIGURE 9

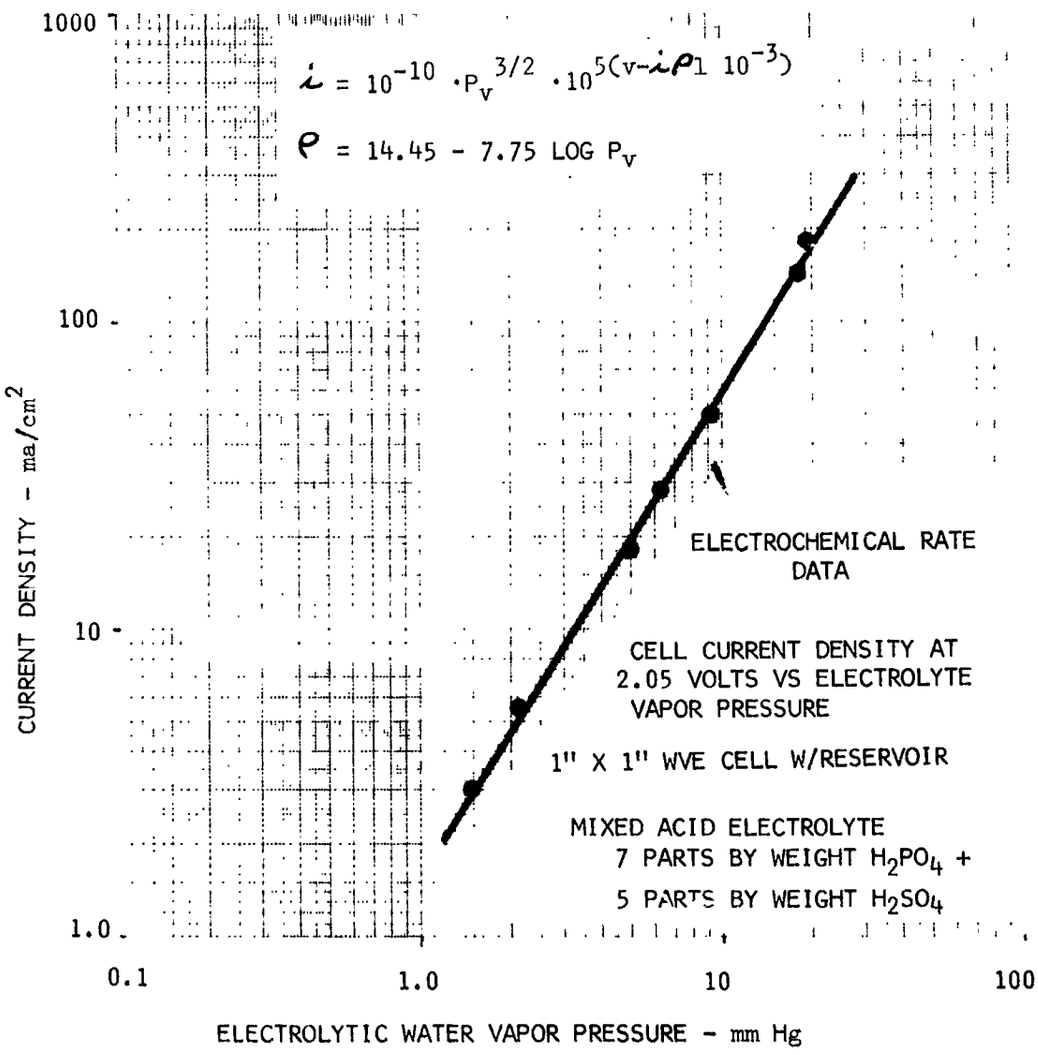


FIGURE 10

could be reduced considerably with a more efficient catalyst. This is important not only from the standpoint of power reduction but also to prevent the formation of ozone which becomes appreciable at potentials greater than 2.1 volts. A better anode catalyst and electrode structure

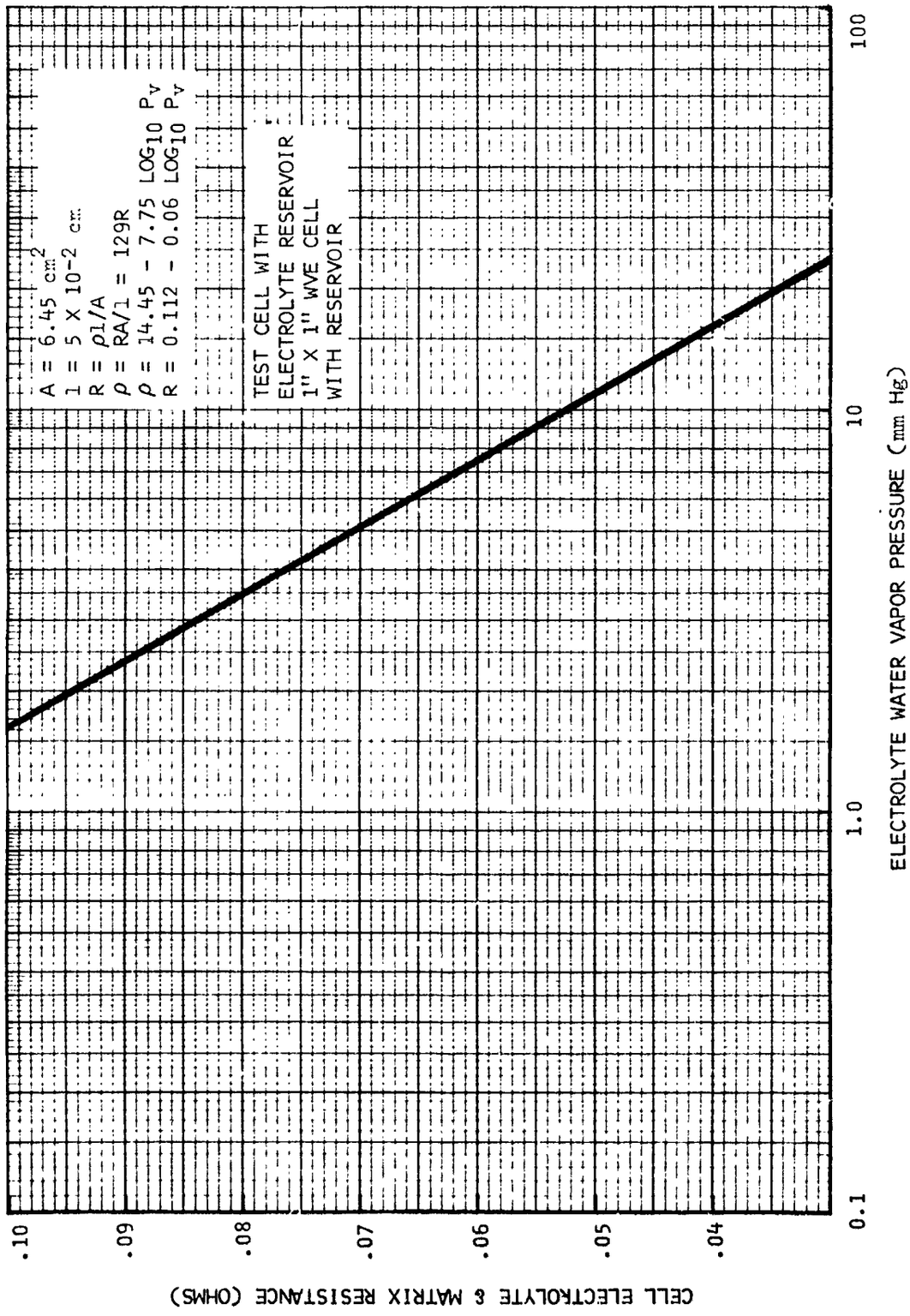

A = 6.45 cm²
l = 5 X 10⁻² cm
R = ρl/A
ρ = RA/l = 129R
ρ = 14.45 - 7.75 LOG10 Pv
R = 0.112 - 0.06 LOG10 Pv
TEST CELL WITH
ELECTROLYTE RESERVOIR
1" X 1" WVE CELL
WITH RESERVOIR
.10
.09
.08
.07
.06
.05
.04
0.1
1.0
10
100
ELECTROLYTE WATER VAPOR PRESSURE (mm Hg)
CELL ELECTROLYTE & MATRIX RESISTANCE (OHMS)

FIGURE 11

was developed by Hamilton Standard and used in the Phase II cell pair hardware.

Observations of the anode during operation showed visible bubble formation resulting in electrolyte spattering only if the electrolyte dewpoint was greater than 50°F (9.2 mm Hg) and the current density greater than 45 amp/ft^2. Figure 10 gives the dependence of the current density (I_V) at constant cell voltage (2.05 volts) on electrolyte water vapor pressure, (P_V) which shows again that $I_V = k\ P_V^{3/2}$. Figure 11 gives the electrolyte resistance as a function of P_V for the Tissuquartz matrix. From this data the cell kinetic equation for the mixed electrolyte is found to be

$$\dot{\zeta} = 10^{-10}\ P_V^{3/2}\ 10^5\ (V - \dot{\zeta}\,\rho\, l\ 10^{-3})$$

where l is the matrix thickness in cm, and the electrolyte resistivity (ρ) in ohm cm, including the matrix factors, is given by:

$$\rho = 14.45 - 7.75 \text{ Log } P_V$$

Where P_V is in mm Hg and $\dot{\zeta}$ is ma/cm^2 ($\approx$ amp/ft^2)

Mixed Acid Electrolyte

The arguments for considering a mixture of H_2SO_4 and H_3PO_4 as a candidate electrolyte were based on potential problems each acid displayed separately. First, examination of the H_3PO_4 phase diagram revealed crystallation would occur at high concentration ($\approx$ 88 wt-%) at room temperature (25°C). Secondly, a review of work performed on the electrochemistry of H_2SO_4 by Pratt and Whitney Division of UAC revealed that H_2S was observed at the H_2 electrode a bulk H_2SO_4 concentrations above 40 wt-% and temperatures greater than 30°C. (1)

In short, the formation of H_2S is thermodynamically possible at these conditions (fuel cell, open circuit or electrolysis mode), and it appears the controlling feature for H_2S formation is the reaction kinetics. Because rate data is not available an electrolyte with a lower H_2SO_4 concentration is recommended.

---

(1) A recent discussion of the experimental details with the principle author confirmed that H_2S was observed experimentally in the H_2 stream sweeping past the H_2 electrode at open circuit and also at fuel cell operating potentials.

It is believed[1] that only non-ionized H_2SO_4 can lead to H_2S formation, so that H_2SO_4 should be diluted with an acid of lower strength to allow further ionization of H_2SO_4. H_3PO_4 is an excellent candidate as its first ionization constant is 5 orders of magnitude less than that for H_2SO_4. This means H_2SO_4 will ionize at the expense of H_3PO_4 thus reducing the possibility of H_2S formation. Also as H_3PO_4 is diluted any crystallization problems are avoided.

A mixture of equal volume 65 wt-% H_2SO_4 and 85 wt-% H_3PO_4[2] was tested in this program and was the recommended cell loading electrolyte at the conclusion of Phase I. It was recommended that H_2S detection tests should be concluded on both H_2SO_4 and mixed acid in Phase II to better select the electrolyte.

Wicking Tests

A materials investigation was made to determine the correct material for the reservoir and electrolyte matrix which would provide the proper wicking relationship and still be compatible with H_2SO_4 and H_3PO_4. The wicking relationship must be such that the matrix material will wick the electrolyte from the reservoir material and the reservoir will wick the excess electrolyte off the matrix. In addition this electrolyte transfer must be made across an electrode.

The AA-2 (Cathode) electrode material, because of the platinized coating, will allow the electrolyte to transfer quite readily.

A data search of acid compatible materials which would wick revealed materials like asbestos (used in the BMI cells), microporous rubber (ACE-SIL" used in batteries), fritted glass and Tissuquartz. Testing of these materials resulted in the following comparisons:

a. Cab-O-Sil matrix (used in NASA/ARC cells) has very poor wicking properties. A material having weaker wicking properties to work with this matrix could not be found.

---

(1) J. Lundquist - Private communication

(2) Nominal electrolyte concentration selection is based on the requirement of a typical mass transfer delta driving force of 4.5 mm Hg. As nominal inlet humidity is 10.5 mm Hg, nominal electrolyte vapor pressure is, therefore, 6.5 mm Hg. It is also noted that the specification requirement to expose cell electrolyte to 0.1 psia (5 mm Hg) for four hours is easily met with selection of nominal vapor pressure of 6.5 mm Hg. This represents only a 5 wt-% change in acid concentration upon 0.1 psia exposure which will not degrade the WVE cell performance.

b. Of the materials tested, the microporous rubber had the best wicking action; however, this material would be a poor matrix candidate since it would have a high IR drop.

c. The Tissuquartz has excellent wicking properties, a large void volume (approximately 98%), low IR drop, and has been used in Pratt and Whitney Aircraft, Division of UAC fuel cells. This would be an excellent matrix, and in addition could act as a wet seal, thereby simplifying cell fabrication.

d. Fritted glass has good wicking properties which would be compatible with the Tissuquartz and would act as a support for the electrode. The only disadvantage is its low percent void volume (approximately 25%).

f. The asbestos has wicking properties equal to Tissuquartz, however, it has limited life with H_2SO_4.

Analytical Studies

A complete analytical model able to describe properly all mass, heat and electrochemical processes occurring within an electrolysis cell is an ideal tool for design. Such a tool requires intimate knowledge of the local processes occurring throughout the cell as well as the ability to provide detail balancing of the extent of these processes during cell operation. As this tool was not available,(1) the analytical effort in the program was channelled to 1) a solution of the mass transport equations for water vapor in the cell, 2) a solution of the equation resulting from coupling the electrochemical rate and mass transport relationships, and 3) use of this coupling device for an oxygen partial pressure control scheme.

Some design parameters were determined by the analytical treatment but other parameters such as the cell flow path length could not be firmly established because local vapor pressures and cell temperature gradients were not known. However, an upper limit for the cell flow path length was obtained.

Mass Transport in Cell

Since the electrolyte vapor pressure is normally not known during cell operation, it is necessary to examine the mass transport of water vapor in the air stream in order to express P_V in terms of known quantities, e.g., water vapor pressure in air stream (P_{H_2O}), air velocity, and cell geometry. Mathematically this is a very difficult problem especially when the rate of water electrolysis has been

(1) A detailed math model of the WVE cell is being developed under the SSP Program

shown to be proportional to $P_V^{3/2}$. However, Levich[1] has developed a solution for the case where the exponent is one and also for the case where $P_V << P_{H_2O}$. The latter case corresponde to a limiting current density where the rate is controlled entirely by mass transport in the gas phase. This maximum average current density is given by:

$$i_{avg.} = \frac{0.67\, n\, FDP_{H_2O}}{RT} \left(\frac{\nu}{D}\right)^{1/3} \sqrt{\frac{U}{\nu h}}$$

where D is the diffusivity for water vapor in air at 25°C (2.75×10^{-4} ft^2/sec), ν the kinematic viscosity (1.5×10^{-4} ft^2/sec), U the bulk air velocity (ft/sec), R is the gas constant (2.2×10^{-3} lb/gm x 555 mm Hg ft^3/lb mole °R), T is the absolute temperature (46° + 77°F = 537°F), n is equivalents per mole (2 eq/mole), F is Faraday's constant (9.65×10^4 amp sec/grams eq), and h is the length of the electrode (ft). This equation reduces to:

$$i_{avg.} = \frac{3.6 \text{ amp sec}^{1/2}}{\text{mm Hg-ft}^2} \left(P_{H_2O}\right) \sqrt{\frac{U}{h}}$$

The lowest P_{H_2O} for cell operation is 5 mm Hg which corresponds to 63°F and 35% RH so that the maximum average current density under these conditions is:

$$i_{avg.} = \frac{19 \text{ amp sec}^{1/2}}{ft^2} \sqrt{\frac{U}{h}}$$

Since the actual average current density for a WVE cell design should be less than one half this ($i_{avg.}$) maximum value to avoid serious mass transport problems, the term U/h must be at least 20 for operation at current densities approaching 45 amp/ft^2 at low dew points. A nominal value for U is 10 ft/sec so that the cell length could be up to 0.5 ft without encountering mass transport limitations as illustrated by Figure 12.

Levich has also shown that the thickness of the diffusion boundary layer is given by:

$$\delta = 3\left(\frac{D}{\nu}\right)^{1/3} \sqrt{\frac{\nu x}{U}} \sqrt{x}$$

at any distance x from the air inlet side of the electrode.

A plot of δ vs. electrode length is shown in Figure 13 for various air flow velocities. For the nominal velocity of 10 ft/sec, δ equals 10^{-2} (or .12") at an electrode length of 0.5 ft (6"). This means

(1) Levich, V.G., Physicochemical Hydrodynamics, Prentice-Hall, due N.J. (1962).

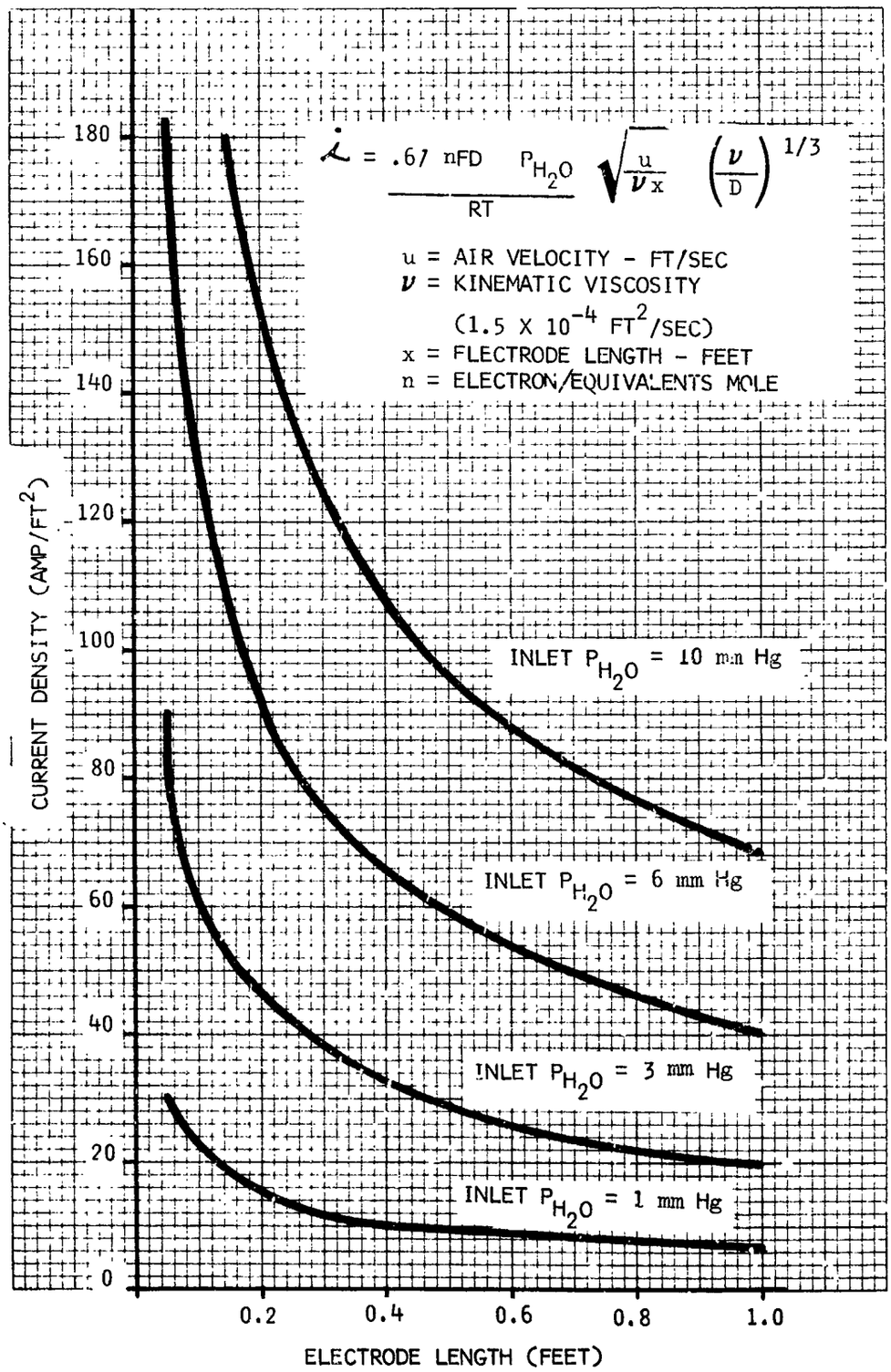

$i = \frac{.67\ nFD\ P_{H_2O}}{RT} \sqrt{\frac{u}{\nu x}} \left(\frac{\nu}{D}\right)^{1/3}$
u = AIR VELOCITY - FT/SEC
ν = KINEMATIC VISCOSITY
(1.5 X 10^-4 FT^2/SEC)
x = ELECTRODE LENGTH - FEET
n = ELECTRON/EQUIVALENTS MOLE
INLET P_H2O = 10 mm Hg
INLET P_H2O = 6 mm Hg
INLET P_H2O = 3 mm Hg
INLET P_H2O = 1 mm Hg
CURRENT DENSITY (AMP/FT^2)
180
160
140
120
100
80
60
40
20
0
0.2
0.4
0.6
0.8
1.0
ELECTRODE LENGTH (FEET)

FIGURE 12

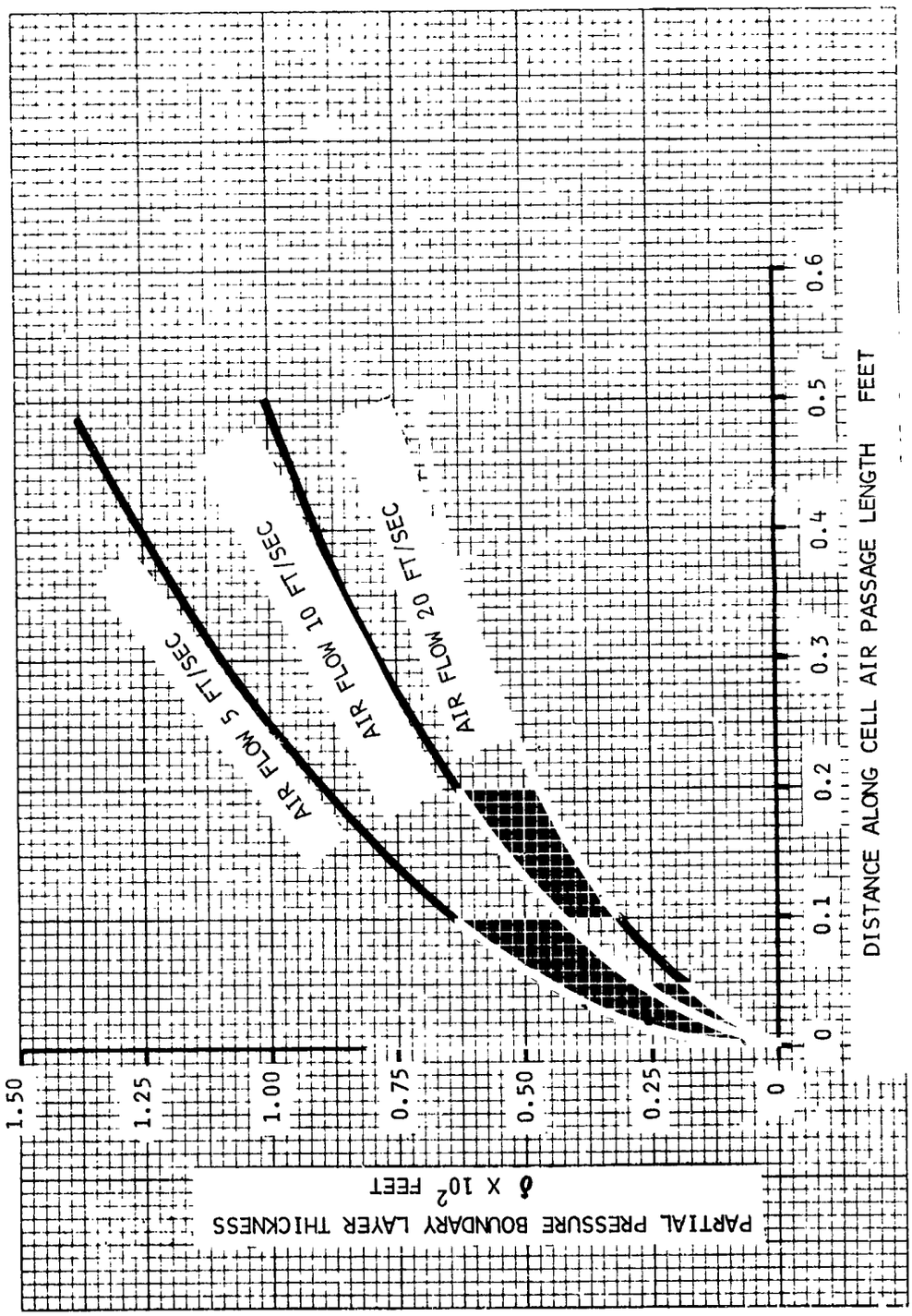

PARTIAL PRESSURE BOUNDARY LAYER THICKNESS
δ X 10² FEET
1.50
1.25
1.00
0.75
0.50
0.25
0
AIR FLOW 5 FT/SEC
AIR FLOW 10 FT/SEC
AIR FLOW 20 FT/SEC
0
0.1
0.2
0.3
0.4
0.5
0.6
DISTANCE ALONG CELL AIR PASSAGE LENGTH FEET

FIGURE 13

if an electrode length of 6" is selected, the air passage height above the anode screen can be no less than .12" to maintain sufficient flow and avoid mass transport problems.

Electrochemical Rate/Mass Transport Relationships

When $P_{V\ avg}$ is significant compared to P_{H_2O} the above equation for δ does not strictly apply. However, it is the best simple approximation available at present. It seems reasonable to assume that the actual δ will be less than that given by the above equation when $P_V \rightarrow P_{H_2O}$. Thus we can write or

$$i_{avg} \approx \frac{3.8\ \text{sec}^{1/2}\ \text{amp}}{\text{mm Hg ft}^2} \left(P_{H_2O} - P_{V\ avg}\right) \sqrt{\frac{U}{h}}$$

Solving the kinetic equation for P_V, neglecting IR drop, and substitution into the above equation gives

$$P_{H_2O} \approx \frac{i}{3.8} \sqrt{\frac{h}{U}} \frac{\text{mm Hg ft}^2}{\text{sec}^{1/2}\ \text{amp}} + 10^{10/3\ (2 - V)}\ i^{2/3} \frac{\text{mm Hg ft}^{4/3}}{\text{amp}^{2/3}}$$

A plot of i versus P_{H_2O} for h = 1/2 ft and U = 10 ft/sec at various cell voltages (uncorrected for electrolyte resistance) is shown in Figure 14. The actual current density should be somewhat higher than shown because of the assumption made for calculating δ. However, this will be counteracted by the IR drop correction which becomes significant at current densities above approximately 30 amp/ft^2, for low values of P_{H_2O}.

Figure 14 summarizes the coupling of the electrochemical reaction rate (current density) and mass transfer (inlet humidity) relationships at various voltages. The figure can be used for devising the O_2 production control scheme as well as relating the nominal, maximum, and minimum operating current densitires. Also included in Figure 14 is a minimum electrolyte vapor pressure line (1 mm Hg). Operation below this vapor pressure is not recommended. This limit serves to select the operation conditions presented below.

Oxygen Partial Pressure Control Scheme

Summarized in Figure 14 is the relationship between cell inlet humidity and current density at various cell voltages (less IR drop). A particular voltage curve represents the current density which the cell will spontaneously try to achieve when exposed to a particular inlet humidity. At constant voltage the cell displays a "self-compensating" principle of producing less O_2 at low inlet humidity and more at high humidity, which is generally the case in spacecraft. This "self-compensating" principle will be employed to design the O_2 control system.

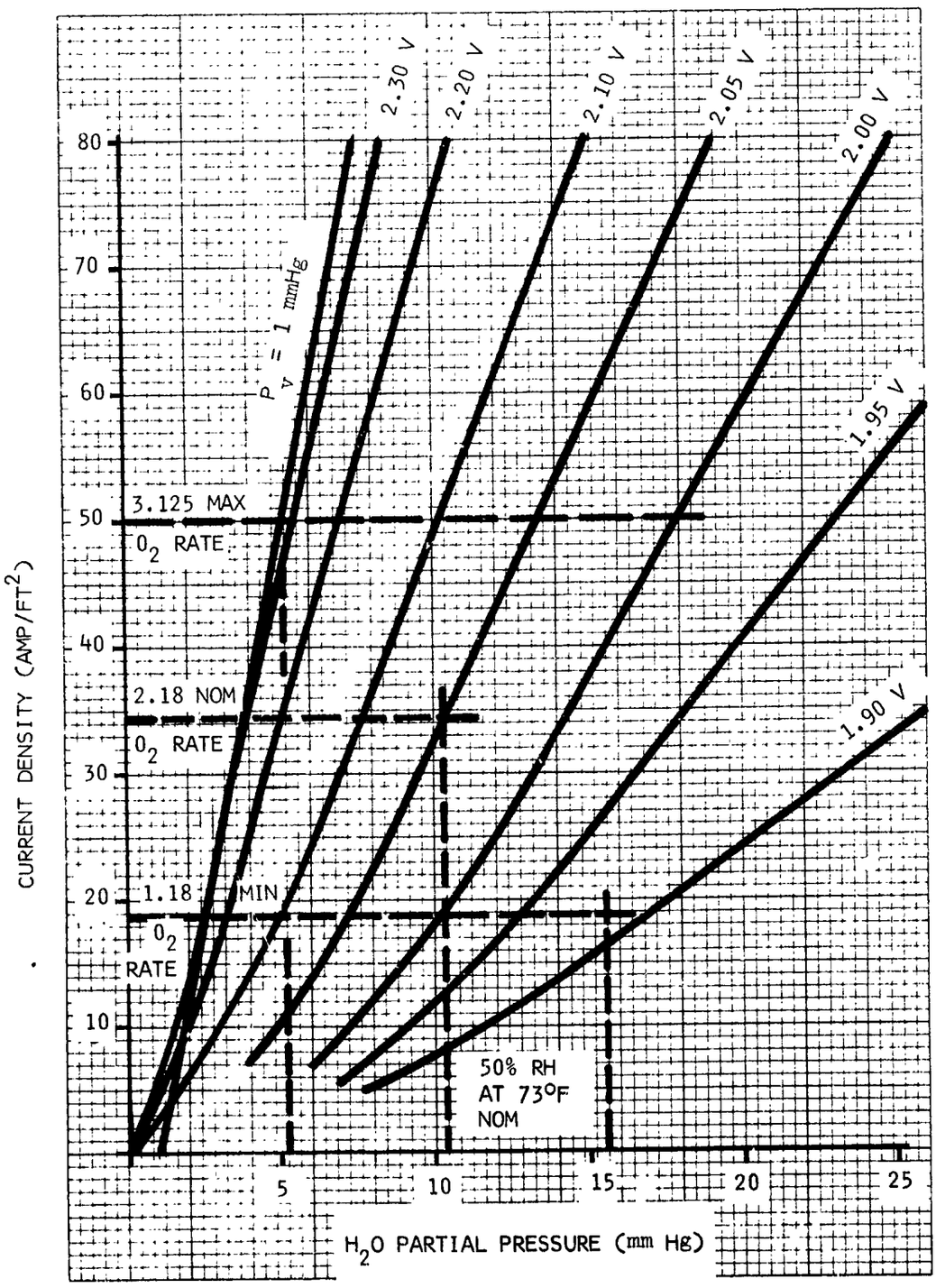

2.30 V
2.20 V
2.10 V
2.05 V
2.00 V
1.95 V
1.90 V
P_v = 1 mmHg
3.125 MAX
O_2 RATE
2.18 NOM
O_2 RATE
1.18 MIN
O_2 RATE
50% RH
AT 73°F
NOM
CURRENT DENSITY (AMP/FT^2)
80
70
60
50
40
30
20
10
5
10
15
20
25
H_2O PARTIAL PRESSURE (mm Hg)

FIGURE 14

The features of this control system are summarized in Figure 15, where inlet humidity is plotted against the O_2 production rate. An operating domain (shaded area) is defined by the maximum/minimum O_2 production rates and maximum/minimum inlet humidities.

Figure 15 was obtained from Figure 14 by setting the maximum O_2 production rate equal to the maximum current density that can be tolerated by the cell when operating at the minimum inlet humidity of 5.2 mm Hg (35% RH at 63°F) without drying out the cell below a safe limit of 1 mm Hg water vapor pressure. The minimum vapor pressure of 1 mm Hg is reached at 50 amp/ft^2 which is, therefore, the maximum O_2 production rate (3.125 lb/man/day). The nominal occurs at 34.8 amp/ft^2 and the minimum (1.18 lb/man/day) at 18.8 amp/ft^2. Now the control box is completely defined.

The nominal cell potential (less [illegible]) of 2.05 volts is obtained from Figure 14 by the intersection of the nominal current density line of 34.8 amp/ft^2 and the nominal inlet humidity line of 10.4 mm Hg (50% RH at 73°F). This will be the normal operating cell voltage. This and other voltage curves are plotted in Figure 15 with the nominal conditions identified. This curve demonstrates the "self-compensating" principle of operating at constant voltage. If the inlet humidity is greater or less the cell will tend to produce more or less O_2 following this curve. It is seen that the minimum O_2 production rate at the minimum humidity of 5.2 mm Hg (35% RH at 65°F) occurs very close to the nominal 2.05 volts. On the other hand, the maximum inlet humidity of 15.6 mm Hg (65% RH at 77°F), at 2.05 volts, leads to greater than required O_2 maximum production rate. Accordingly the voltage should be reduced so as not to exceed 3.125 lb/man/day. If the maximum O_2 rate is demanded at the low inlet humidity level of 5.2 mm Hg, the cell voltage would have to be increased.

Control System

A system review of the imposed requirements to determine the range of control which the WVE unit would have to perform was conducted (reference Figure 15). The minimum oxygen production requirements of 1.18 lb/man/day defined by the review was based on the period when the crew was sleeping and zero cabin leakage (metabolic consumption rate of 300 BTU/hr/man). This minimum oxygen rate which presently is being used on Space Station work is lower than the specified rate of 1.62 lb/man/day. The maximum requirement was established at 3.125 lb/man/day (2.56 lb/man/day metabolic plus 1.565 lb/man/day leakage).

The specified max. rate of 4.21 lb/man/day is in error since it assumes the maximum cabin leakage of 1.65 lb/man/day is pure oxygen instead of a mixed gas having an oxygen partial pressure of 3.1 psi.

Since a time-line of oxygen consumption and cabin humidity was not available, an assumption was made that any change in the oxygen consumption

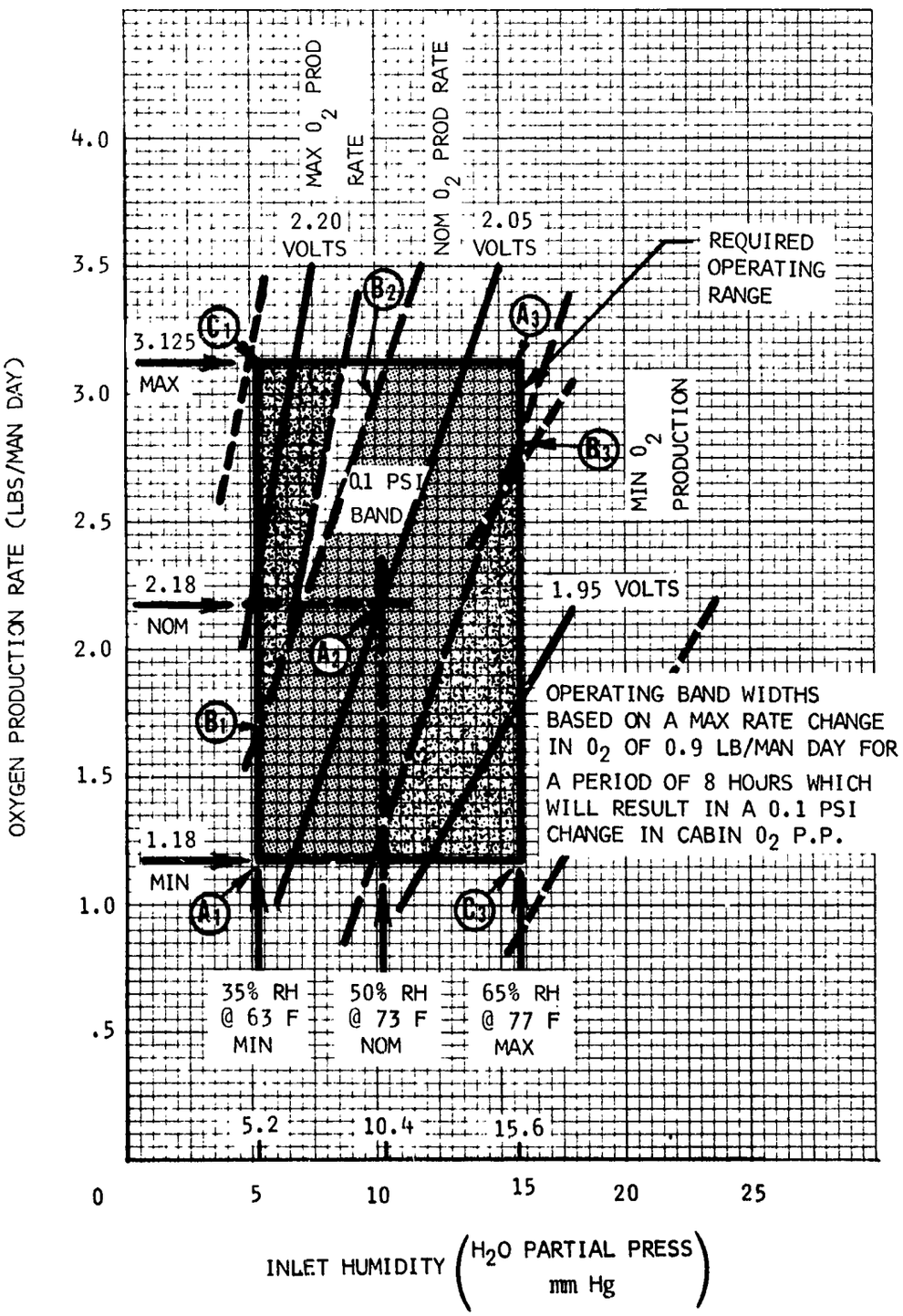

MAX O_2 PROD RATE
NOM O_2 PROD RATE
2.20 VOLTS
2.05 VOLTS
REQUIRED OPERATING RANGE
3.125
MAX
2.18
NOM
1.18
MIN
0.1 PSI BAND
MIN O_2 PRODUCTION
1.95 VOLTS
OPERATING BAND WIDTHS BASED ON A MAX RATE CHANGE IN O_2 OF 0.9 LB/MAN DAY FOR A PERIOD OF 8 HOURS WHICH WILL RESULT IN A 0.1 PSI CHANGE IN CABIN O_2 P.P.
35% RH @ 63 F MIN
50% RH @ 73 F NOM
65% RH @ 77 F MAX
5.2
10.4
15.6
OXYGEN PRODUCTION RATE (LBS/MAN DAY)
INLET HUMIDITY (H_2O PARTIAL PRESS mm Hg)

FIGURE 15

rate for an eight hour period would not exceed 0.9 lb/man/day delta to the WVE production rate. This delta would produce a 0.1 psi change in the cabin oxygen partial pressure, of which the allowable tolerance is ± 0.2 psi.

When the WVE is operated at constant voltage the normal changes in relative humidity and oxygen demand tend to be self compensating; however, with large changes in the oxygen demand as noted above it would be necessary to have three voltage settings (with current limiters) for operating the WVE unit (reference Figure 15, voltage settings of 1.95 - 2.05 - 2.20). The voltage settings would be selected manually or automatically based on the direction of change in the oxygen partial pressure. Under normal operation the unit would maintain the nominal voltage and produce oxygen with respect to the relative humidity as indicated in Figure 15. It should be noted that the 2.20 voltage setting is in the range of WVE operation which produces ozone which must be filtered out. The improved anode used in the Phase II tests reduces the cell voltage substantially below this danger point.

Figure 16 presents two cases where the delta between the WVE oxygen production rate and the crew oxygen demand exceed the 0.9 lb/man/day and one case of a normal change in oxygen rates. In all three of these cases the cabin oxygen partial pressure is held within the tolerance of 3.1 ± 0.2 psi.

Case 1.- This situation could occur when the crew wakes up after sleeping 8 hours (Point A_1, Figure 16, WVE set on 2.05 volts, humidity at 35% RH at 63°F) and all start to work or exercise, along with max. cabin leakage (O_2 demand increases from 1.18 to 3.125 lb/man/day). It is assumed that the increased O_2 demand continues for 8 hours without an increase in cabin humidity (not very likely but possible). Under these conditions the P_{O_2} will drop to 3.0 within 2 hours and the WVE would be switched to 2.20 volts (Point B_1). This increase in O_2 production still cannot meet the crew demand and after 8 hours the P_{O_2} drops to 2.9 psi (Point C_1). At this point it is valid to assume that the cabin humidity has increased and the WVE production rate exceeds the demand and the P_{O_2} comes back to the nominal value of 3.1 psi.

Case 2.- This is a normal situation where the delta in O_2 rates does not exceed 0.9 lbs/man/day at a condition of constant cabin humidity.

Case 3.- This situation could occur when the crew stops exercising or working (Point A_3 demand max. WVE set on 2.05 volts, humidity at 65% RH at 77°F) and they all rest or go to sleep (O_2 demand drops to min. of 1.18 lb/man/day). It is also assumed that the cabin humidity remains constant for 8 hours. Under these conditions the P_{O_2} will rise to 3.2 within 2 hours, and the WVE would be switched to 1.95 volts (Point B_3). This decreases the WVE oxygen production rate but it still produces more O_2 then the crew demands (because the cabin humidity is high) and after 8 hours the cabin P_{O_2} is at 3.3 psi (Point C_3). Again it is assumed that by the time the

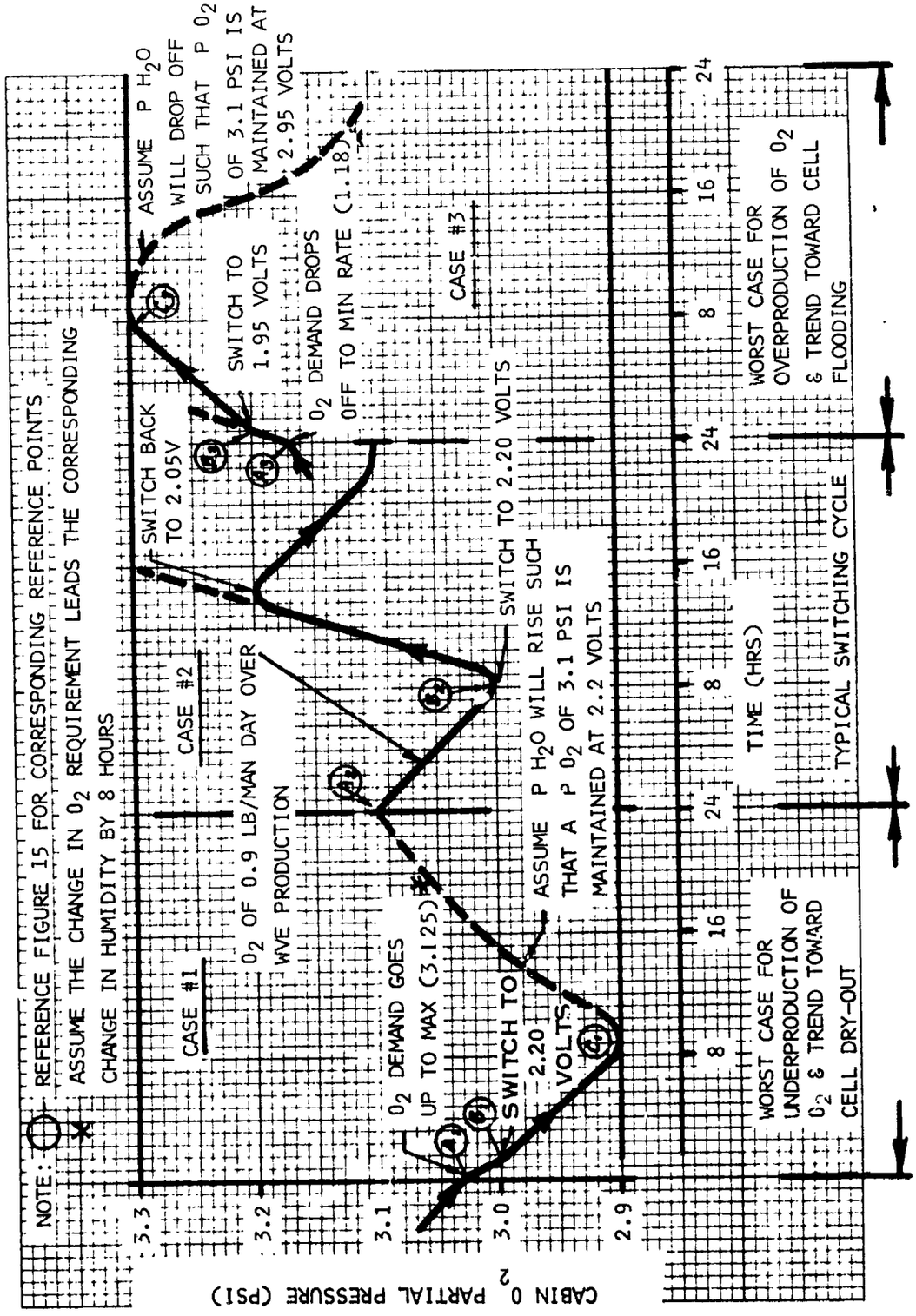

NOTE: ○ REFERENCE FIGURE 15 FOR CORRESPONDING REFERENCE POINTS
✕ ASSUME THE CHANGE IN O_2 REQUIREMENT LEADS THE CORRESPONDING CHANGE IN HUMIDITY BY 8 HOURS
CASE #1
O_2 OF 0.9 LB/MAN DAY OVER
WVE PRODUCTION
O_2 DEMAND GOES UP TO MAX (3.125)
SWITCH TO 2.20 VOLTS
ASSUME P H_2O WILL RISE SUCH THAT A P O_2 OF 3.1 PSI IS MAINTAINED AT 2.2 VOLTS
SWITCH TO 2.20 VOLTS
CASE #2
SWITCH BACK TO 2.05V
SWITCH TO 1.95 VOLTS
O_2 DEMAND DROPS OFF TO MIN RATE (1.18)
CASE #3
ASSUME P H_2O WILL DROP OFF SUCH THAT P O_2 OF 3.1 PSI IS MAINTAINED AT 2.95 VOLTS
WORST CASE FOR UNDERPRODUCTION OF O_2 & TREND TOWARD CELL DRY-OUT
TYPICAL SWITCHING CYCLE
WORST CASE FOR OVERPRODUCTION OF O_2 & TREND TOWARD CELL FLOODING
TIME (HRS)
CABIN O_2 PARTIAL PRESSURE (PSI)
3.3
3.2
3.1
3.0
2.9
8
16
24

FIGURE 16

cabin humidity starts to decrease and the cabin P_{O_2} will return to its normal value of 3.1 psi within the next 16 hours.

Cases 1 and 3 are extreme situations which most likely would not occur for the length of time that is presented.

Final Cell Comparison

Table II presents a comparison of each detail part of a Water Vapor Electrolysis Cell which resulted in the final design configuration. This table includes the data obtained from the Phase II WVE cell pair tests.

Design Limits

Table III presents a tabulated listing of design parameters and their respective limits for WVE design. This table includes the data obtained from the Phase II WVE cell pair tests.

Phase I Conclusions

The results of the Phase I study and feasibility tests indicated that a Water Vapor Electrolysis Cell having a tolerance to a wide range of inlet humidity (5% to 90% RH) can be obtained and operated to maintain the cabin oxygen partial pressure at 3.1 $\pm$ 0.2 psia.

Phase I Recommendations

At the completion of Phase I recommendations were made to the design configuration, of the NASA/ARC cell pair unit which evolved from the Phase I study, as presented on drawing SVSK 81296, Appendix E. This modified design incorporates the design limits which have been established by this report and the following details:

Electrodes - Anode - Platinized platinum, screen
Cathode - P&WA type that replaces the present AA-2
Electrolyte - A mixed acid solution of H_2SO_4 & H_3PO_4
Electrolyte Matrix - Tissuquartz (.017 thickness)
Reservoir - Fritted glass

The Phase II test hardware should be a cell pair of the same basic configuration as above having a 2 inch air passage with modifications for instrumentation and added test flexibility. (i.e. Plexiglas housing, thermocouple ports, inlet duct, etc.).

An investigation should be made to improve the Anode such that the polarization is reduced (increased catalytic activity). The production of H_2S during water vapor electrolysis should be more thoroughly investigated.

TABLE II

FINAL TRADE COMPARISON

Detail cell component	Advantage	Disadvantages
Electrolyte		
H_2SO_4	1. Provides desirable Tafel curves over a wide range of concentrations. 2. Is a more efficient cell operation. 3. Low IR drop.	1. Will produce H_2S if hydrogen gas is not purged from cell at shutdown.
H_3PO_4	1. Apparently is compatible with asbestos matrix.	1. Will precipitate at high concentrations. 2. Has a higher Tafel slope and a lower exchange current. 3. Has higher viscosity. 4. Has high IR drop. 5. More corrosive than H_2SO_4 (stronger complexing agent)
H_2SO_4 — H_3PO_4	This mixed acid electrolyte was initially selected for the Phase II cell design, however, the proposed advantages were disproven and H_2SO_4 was used as the electrolyte.	
Electrolyte Matrix		
Cab-O-Sil	1. Provides good surface contact with the electrode. 2. Has experimental test time on NASA/ARC program. 3. Void volume approximately 97%	1. Requires a relatively larger electrode spacing resulting in high electrolyte IR drop. 2. Has poor wicking properties and is not compatible in a cell which uses an electrolyte reservoir. 3. Requires a microporous membrane which results in a more complex cell design.
ASBESTOS	1. Has a high bubble point. 2. Has average wicking properties. 3. Has a void volume of approximately 80%.	1. Not compatible with H_2SO_4.
TISSUQUARTZ	1. Is compatible with H_2SO_4 and H_3PO_4. 2. Has excellent wicking properties (Can be easily used with a reservoir). 3. Provides accurate cell loading. 4. Provides a very thin matrix (low resistance). 5. Has many hours of operational use (P&WA fuel cell). 6. Reduces the matrix sealing problems. 7. Has a large percentage of void volume (approx. 98%).	1. Has a low bubble point. 2. Requires great care on handling during cell assembly.
Electrolyte Reservoir		
FRITTED GLASS AND/OR POROUS CERAMIC	1. Is compatible with H_2SO_4 and H_3PO_4. 2. Will wick the excess electrolyte and will allow the matrix to wick it back. 3. Is available in several pore sizes which allows a range of wicking forces. 4. Provides a very wide range (dew point) of cell operation. 5. Can be used to support the electrode.	1. Low percentage of void volume (approx. 25%).
MICROPOROUS RUBBER	1. Very good wick properties. 2. Compatible to H_2SO_4 and H_3PO_4.	1. Will not allow the matrix to wick back the electrolyte.
Electrodes		
AX-2	1. It is desirable electrode and provides good liquid transfer.	1. Is no longer being manufactured.
PAN FIBERMAT	1. Is available in various pore sizes. 2. Is consistent to AX-2.	
STAINLESS STEEL SCREEN	1. Improves wetting of the electrolyte into the screen for maximum surface contact.	Note: Additional [illegible] investigation should be conducted to reduce polarization.
NICKEL STEEL SCREEN	1. Cost and time savings by deleting catalyst or platinizing.	1. Results in very low surface tension between the electrolyte and the screen.

TABLE III

DESIGN LIMITS

Design Variable	Limiting Values Max	Nom	Min	Controlling Parameter Max	Min
Electrolyte	-	-	-	H_2SO_4	ACID - Base will be poisoned by cabin CO_2 - Must be electrochemically stable at cell voltages
Electrolyte Concentration	-	-	-	SEE TEXT	
Matrix Material	-	-	-	Should have a large % of void volume (above 50%)- also good wicking characteristics.	
Matrix Thickness (inches)	.017 (Compressed)		.034 (Not Compressed)	Physical handling requires two sheets.	
Reservoir Material	-	-	-	Wicking properties must be less than those of the matrix. Should have large % void volume (above 50%).	
Reservoir Capacity	Dependent on Operating Conditions (relative humidity)			Reservoir capacity factor and electrolyte volume expansion factor.	
Cell Voltage (without IR), Volts	2.0	1.06		Not to exceed 100 amp/sq ft - also ozone production above 2.1 volts.	
Current Density, i, amp/sq ft	100	30	10	Drying of cell	N/A
Electrode Air Flow length in	6	*		Mass transport, delta T, IR drop	N/A
Electrode Length, in		12		Assembly techniques	N/A
Anode		*			
Anode Thickness, (inches) Cathode	.010 Pratt & Whitney Electrode	.005	.003	Cost	Fab. problems - only acid electrode being mfg.
Cathode Thickness, in		.006		Only size available	
Air Passage Opening, in			.12	Pkg. limits	Mass transport
Air Velocity, U, ft/sec**		10	10	Delta P	Mass transport
Cell Temp. Gradient, °F	*	*	*	U., i, Cell/structure conductivity	

* Hamilton Standard developed anode - Note: Should allow free flow of electrolyte.

** iR drop will add ≈ 0.5 volts to cell potential at nominal conditions.

*** Recommended design does not require variable air velocity.

N/A Not Applicable

PHASE II TESTS

The primary objectives of this phase were to design a prototype WVE test unit, based on the concept selected in Phase I, fabricate the unit and perform a series of verification tests that would demonstrate the feasibility of the selected concept. Two secondary test objectives were requested by NASA as a result of the Phase I study data. These were to obtain additional wicking characteristics of the various cell materials and to investigate the generation of H_2S when using H_2SO_4 as a WVE cell electrolyte.

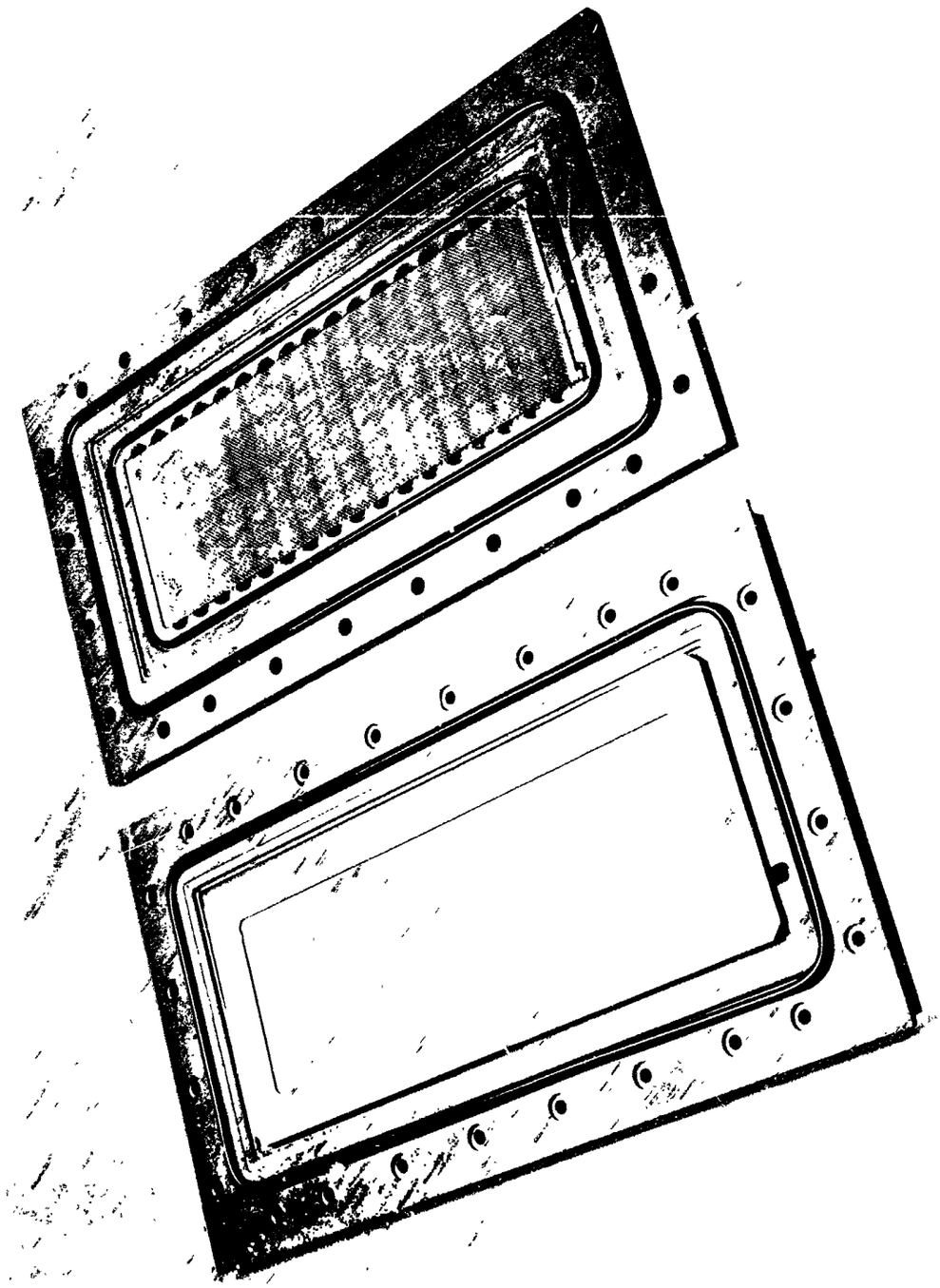
Cell Pair Design

In the initial program plus the Phase II prototype hardware was to be obtained by modifying the NASA/ARC cell pair assembly (reference Figures 17 & 18), for incorporation of the design features set forth in Phase I. This philosophy was changed early in Phase II, by Hamilton Standard and NASA/MSC, because the NASA/ARC cell pair was too small (0.167 ft^2 effective electrolysis area) to be competitive for future spacecraft programs. Instead the cell pair size would be optimized based on weight, power and Phase I limitations and new hardware would be fabricated. Figure 19 shows a comparison of the two cell pair designs. The optimization study was obtained from the NASA Space Station Prototype (SSP) Program and was based on the Hydrogen Depolarized Cell Unit (reference Figure 20), which is very similar to the desired WVE cell pair design. An electrode size of 6 inches (width) by 12 inches (length) was established for this study which corresponds with the Phase I cell limitations.

The cell pair configuration (reference Figure 21) consists of two titanium housings which sandwich the two electrolysis cells and their reservoirs. The anode was a platinum rhodium screen (80 mesh with .003" dia. wire rolled to .005") with catalyst applied to one side. The matrix consisted of two sheets of Tissuquartz (.017" each) saturated with the H_2SO_4 acid electrolyte. The cathode electrode was an expanded tantalum screen (.007") coated on both sides with platinum black catalyst and attached to a silver current carrier frame. The electrolyte reservoirs were made of porous ceramic (pore size of 44 to 55 microns) separated by a 0.060" thick spacer and enclosed by a plexiglas frame.

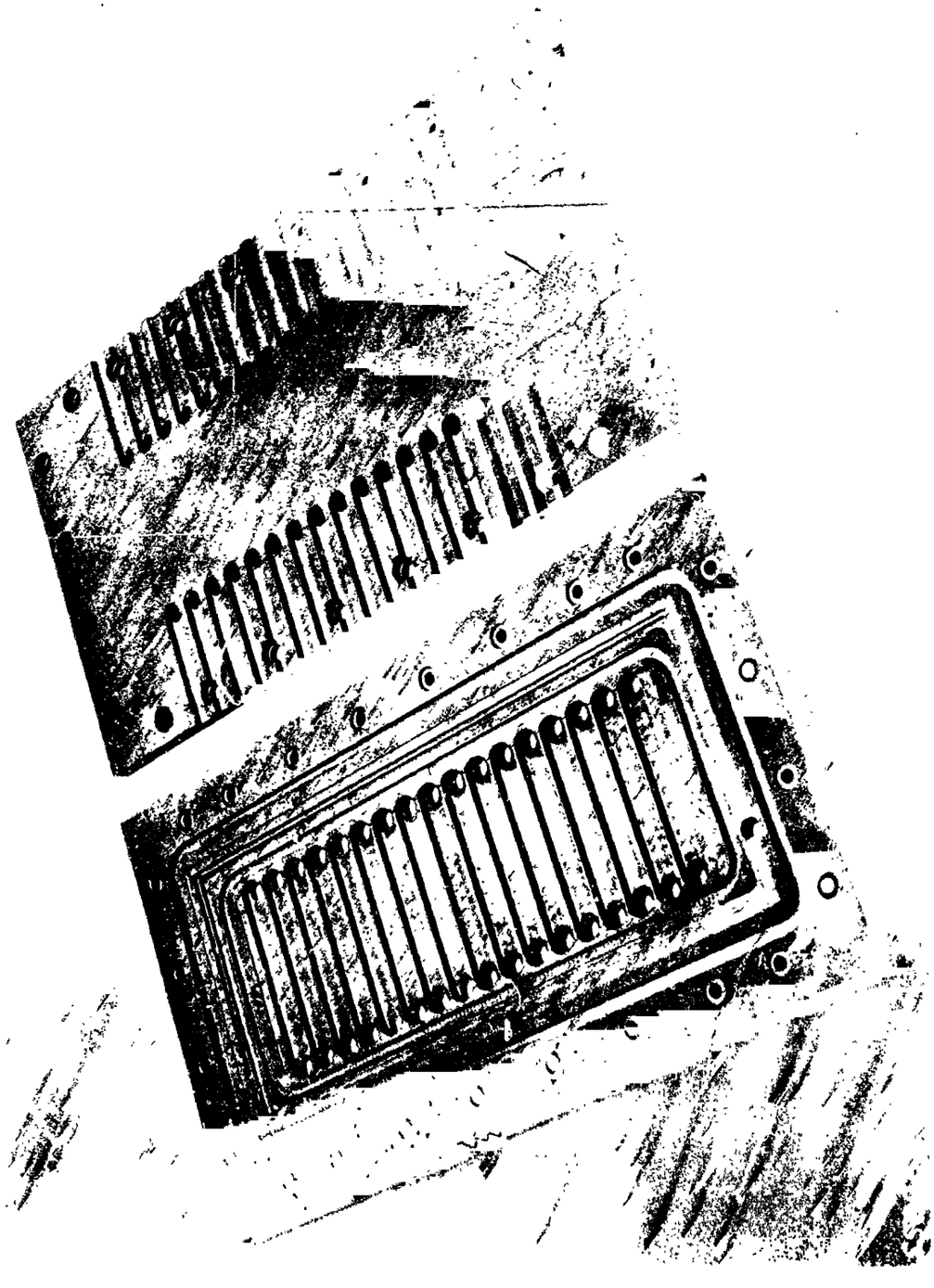
Housings

Titanium was selected for the housings because of its compatibility with H_2SO_4, good electrical conductivity (electrical resistance 50 microhm-cm, good modulus of elasticity (15.0 x 10^6 psi), and very light weight (density 0.164 lb/cu in). The inside face of both housings in the area of electrode contact was platinum plated to maintain electrical contact between the anode and the current carrying housings.



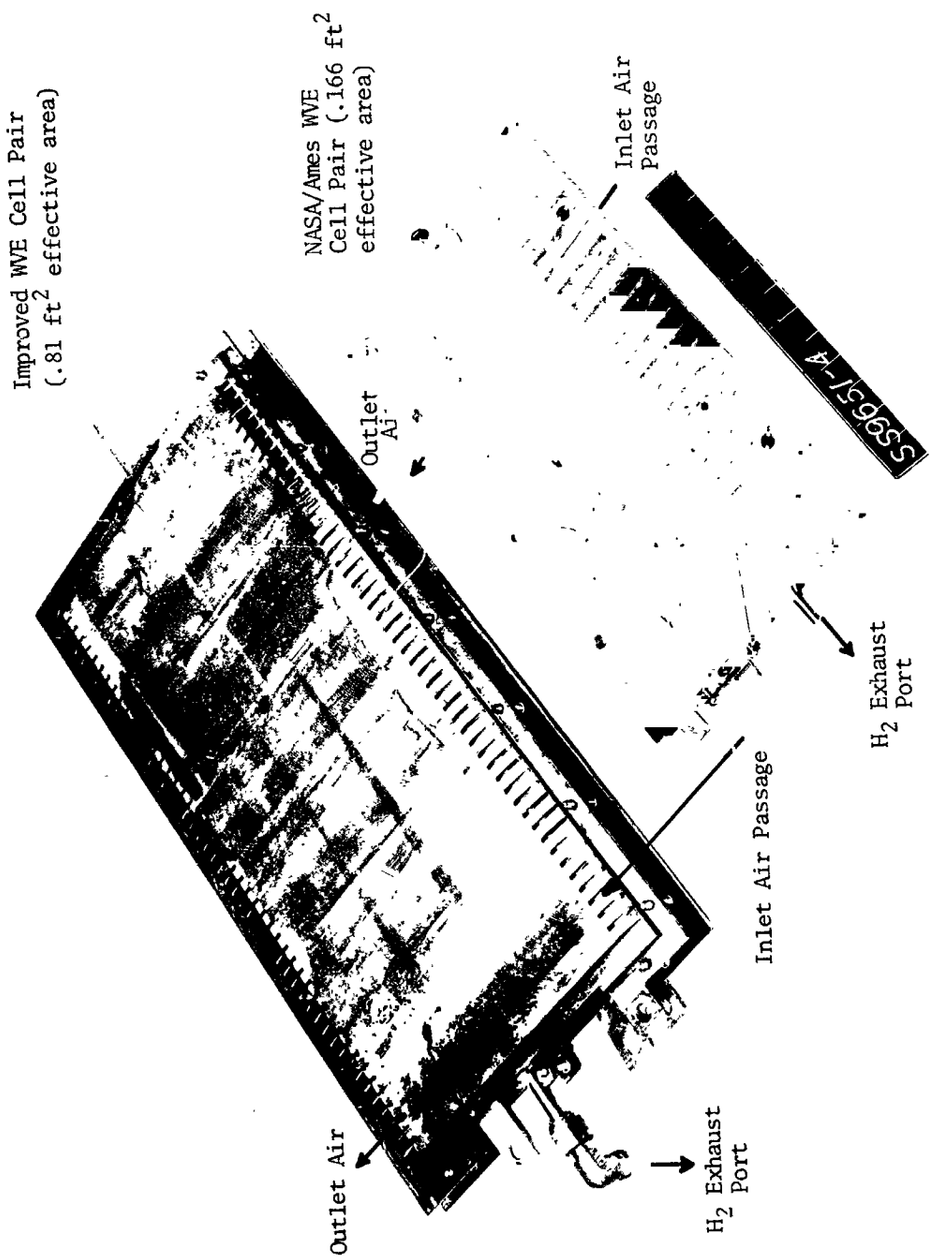
NASA/Ames Cell Pair Build Up

Figure 17



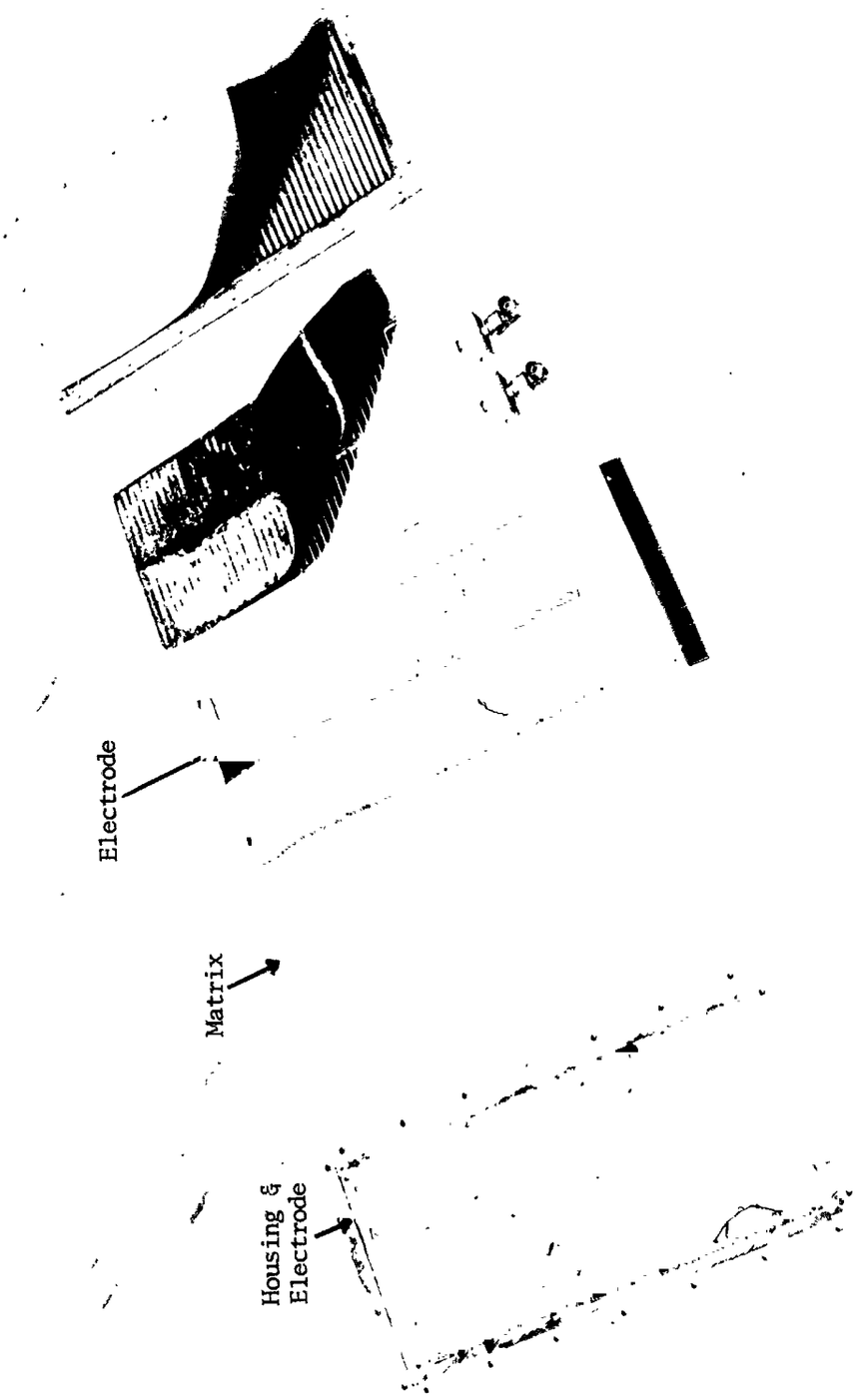
NASA/Ames Cell Pair Housing Set

Figure 18



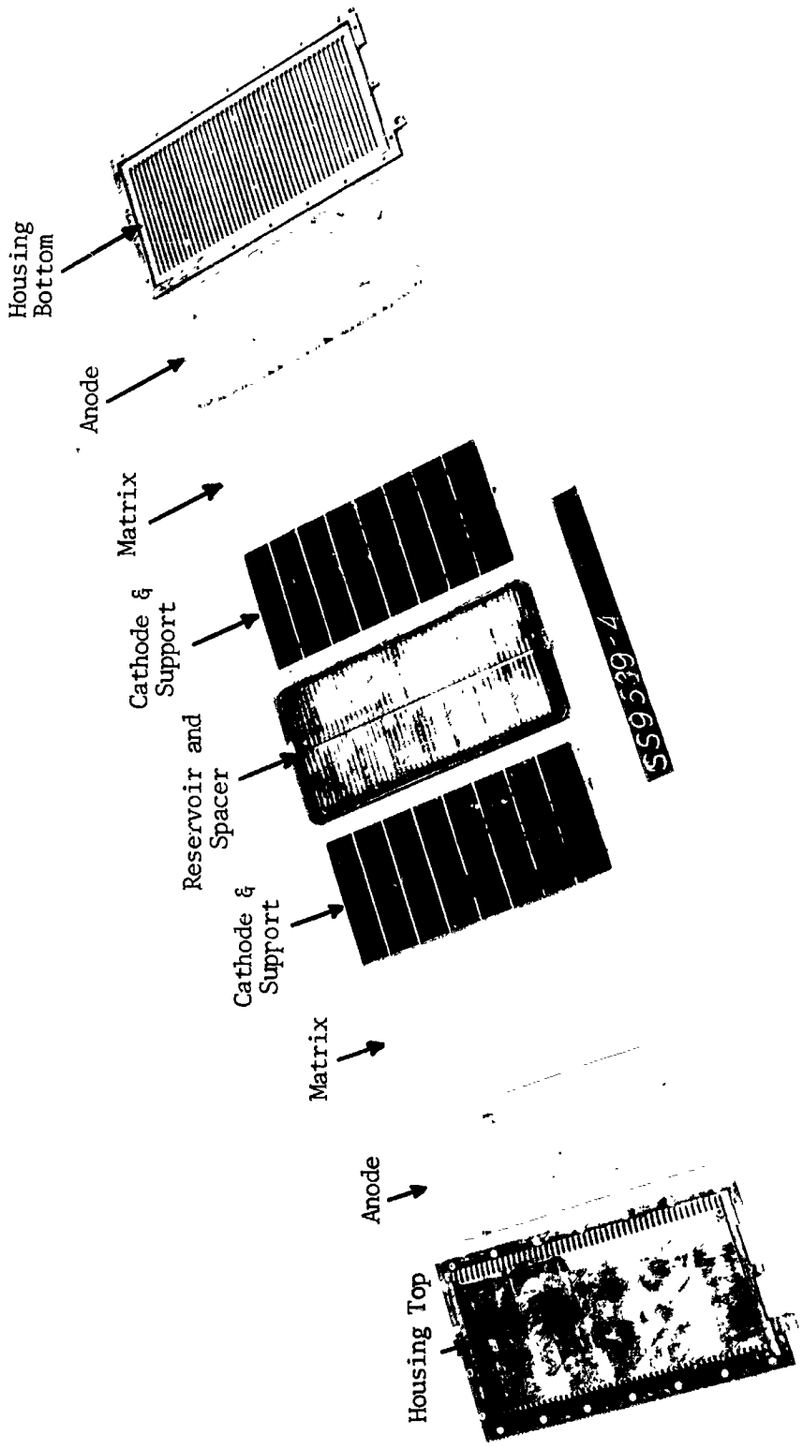
Comparison Of Improved WVE Cell Pair
With NASA/Ames Cell Pair

Figure 19



SSP Hydrogen Depolarized Cell Pair Assembly

Figure 20



Improved WVE Cell Pair Assembly

Figure 21

Anode

The platinum rhodium electrode was selected because of its low electrical resistance and its compatibility with H_2SO_4. The proprietary catalyst that was applied to the matrix side of the screen, was developed at Hamilton Standard to improve cell efficiency.

Matrix

The Phase I study recommended Tissuquartz as the matrix material. Two sheets of Tissuquartz were used for each matrix to improve strength and reduce the likelihood of hydrogen breakthrough. The thicker matrix did increase the stored electrolyte volume causing a slight increase in cell resistance.

Electrolyte

The recommendation of Phase I was to use a mixed acid of H_2SO_4 and H_3PO_4 as the electrolyte. This was based on the possibility of producing H_2S when operating with an H_2SO_4 concentrations greater than 40 wt-%. A Phase II investigation into the production of H_2S in the electrolysis cell revealed that it could be generated upon cell shut down regardless of the H_2SO_4 concentration. For this reason and because of the poorer performance of the mixed acid, it was decided to use H_2SO_4 electrolyte during Phase II.

Cathode

The Phase I study recommended use of the Pratt & Whitney Aircraft acid electrode for the cell because the AA-2 electrode was no longer being manufactured by American Cyanamid Company. Due to the increased cell size the available Pratt & Whitney Aircraft electrodes were not large enough. However, there was sufficient AA-2 of the correct size on hand for the larger cell. As a result of initial Phase II tests, the cathode was modified at the reservoir interface to improve the electrolyte transfer between the matrix and the reservoir. This modification consisted of replacing the AA-2 catalyst on the expended mesh screen with Tissuquartz.

In order to provide good current distribution in the cell the AA-2 electrode was attached to a silver frame.

Reservoir

The wicking test results of Phase I recommended fritted glass for the reservoir. However, the cost and schedule to obtain a fritted glass reservoir of this size (12" x 5 1/2") ruled it out. Porous ceramic manufactured by the Coors, Co., equivalent to the fritted glass, was obtained instead. As a result of initial Phase II tests, the common reservoir was separated horizontally with a 0.060" gap as discussed in greater detail in a subsequent

section. This allows the gas in the reservoir to escape as it is displaced by the electrolyte.

Verification Tests

Test Program

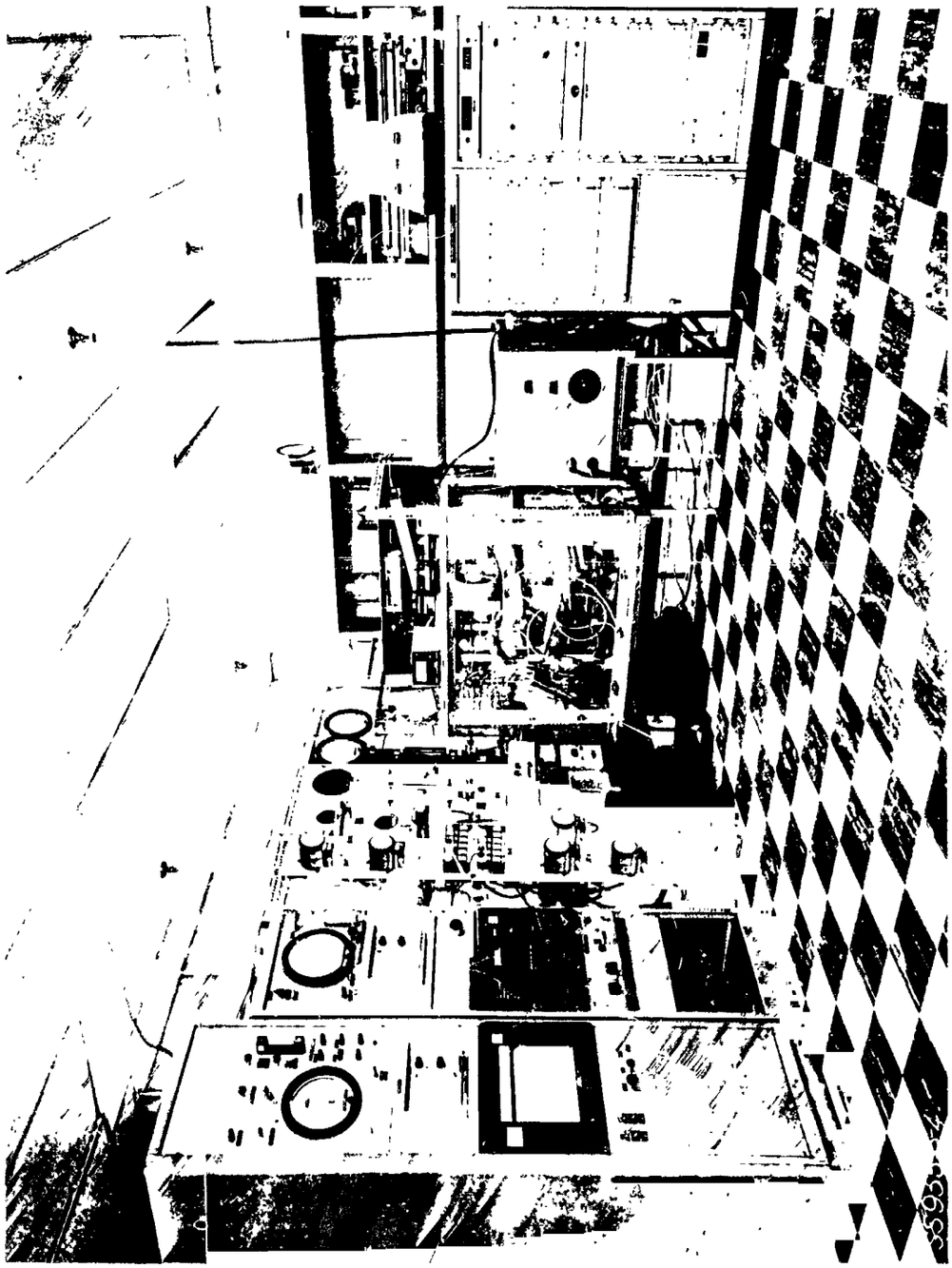
WVE cell pair testing was conducted in accordance with an approved test plan, reference Appendix F, and consisted of a series of steady state tests and a sequence of dry-out and no-wet tests. Testing started in December 1970 but was interrupted for 1 1/2 months to allow the SSP Program to conduct WVE Design Data Tests using the same hardware but without the reservoir. Testing was completed in early March, 1971, accumulating a total of 428 hours of continuous operation.

During this time the cell was operated from an initial cell loading equivalent to 5% relative humidity at 63°F, to a maximum air inlet condition of 75% RH at 65°F and then lowering to a drying condition of 32% RH at 65°F. The cell experienced flooding at the 75% RH inlet condition and hydrogen crossover (matrix dry-out) as the humidity was lowered to 32% RH. This limit in operating conditions was attributed to insufficient capacity of the reservoir. A degradation in the performance was also exhibited over the 428 hours of operation. This is discussed further in the performance degradation section.

A special series of reservoir/matrix tests was conducted as part of the investigation to determine why the unit would not operate over the full, desired range of inlet conditions (5% to 90% RH).

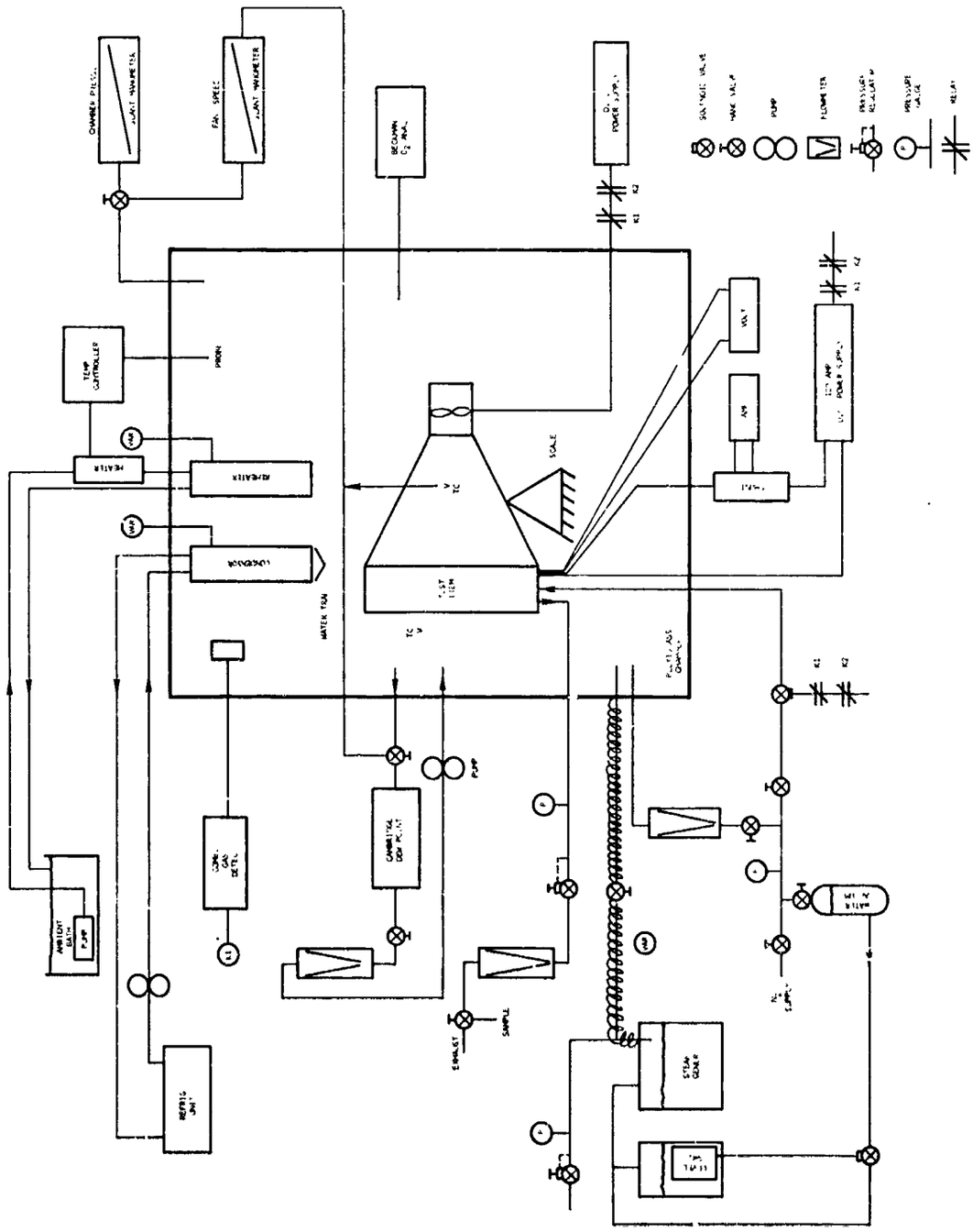
Test Apparatus

The test stand (reference Figure 22 and schematic Figure 23) consisted of a Plexiglas chamber, power supply, data recorder and facility support equipment which maintained the chamber environment. The desired chamber dew point was obtained by a controlled inflow of steam and a condensing heat exchanger within the chamber. The chamber air temperature that was maintained at the test item inlet, was controlled by the combination of the condensing heat exchanger and a preheater. The unit was powered by a Udylite power unit capable of providing 0 to 6 volts at a maximum current of 300 amps. The cell pair was placed in a text fixture (reference Figure 24) that incorporated a Plexiglas support housing, an aluminum exhaust duct and a variable speed DC fan that could provide cell air flows of 0 to 60 CFM. All testing was conducted at an 18 CFM air flow through the cell. This corresponds to the SSP cell air flow. The concentration of oxygen in the chamber was maintained at 20% + 1% by adding nitrogen as the diluent gas. The hydrogen produced by the cell pair was vented out of the chamber through a flow meter and into an exhaust stack. Samples of this gas were taken and checked for H_2S. The cell pair voltage and current were measured with digital meters. The cell pair and the test fixture were placed on a scale, within the chamber,



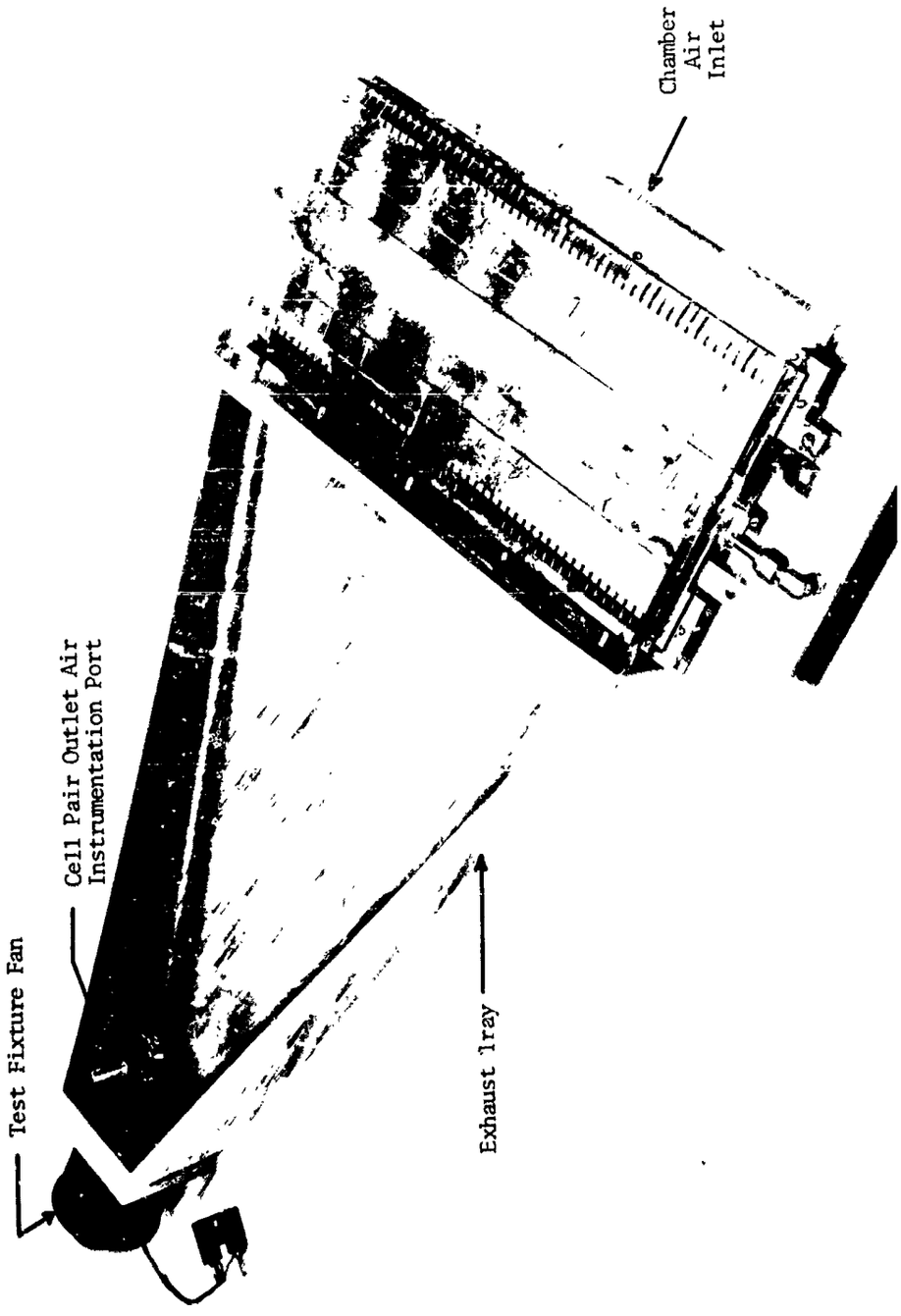
WVE Cell Pair Test Facility

Figure 22



WATER VAPOR ELECTROLYSIS TEST RIG SCHEMATIC

FIGURE 23



WVE Cell Pair and Test Fixture

Figure 24

providing a measurement of any change in the weight of the cell pair.

Test Results

A tabulation of the test conditions and cell pair performance for each cell pair build up is summarized in Table IV. A discussion of each cell pair assembly and its subsequent test is presented in chronological order.

Assembly #1.- The intent of this assembly was to assemble the unit for the wettest condition (90% at 77°F). This required both the cell matrix and reservoir to be saturated with the H_2SO_4 electrolyte at 17 wt-% concentration.

Run #1: The unit was placed in the test chamber, set at 50°F air temperature and 40°F dew point (68% RH), and the cell pair operated at 1.70 volts. At this condition the resultant current was 100.3 amps. However, after one hour of operation hydrogen crossover occurred ending the test. Since the unit was not operated long enough to obtain a steady state conditions this cannot be used as a data point. A quick (approximately 3 min. duration) voltage vs. current sweep was made when the cell was initially powered (reference Figure 25).

Disassembly #1: Disassembly revealed the matrix to be dry and that the reservoir had lost only 19% of its original electrolyte.

Assembly #2.- Since the matrix was limited in the amount of electrolyte it could wick from the reservoir, the unit was assembled such that it would only be operated in a wetting mode. This would require the excess electrolyte to flow from the matrix to the reservoir. The cell pair was assembled using H_2SO_4 at 65 wt-% concentration and saturating only the matrix. This represented the driest condition, of 5% RH at 63°F.

The unit was then placed in the test chamber, at 52°F air temperature and 42°F dew point (71% RH) and a quick voltage vs. current sweep was made (reference Figure 26). Power was then removed and the acid was allowed to equilibrate at this chamber condition for 42 hours with an air flow of 18 CFM through the cell.

Run #1: After conditioning, the unit was operated for three hours at 1.736 volts, producing 39.4 amps. Steady state conditions were not obtained during this time. The unit was shut down and sealed for the weekend, and the chamber was maintained at 50°F T_{in} and 40°F dew point.

Run #2: The unit was operated at 1.810 volts with inlet conditions of 50°F air temperature and 40°F dew point. This condition produced 64.8 amps for 2.5 hours at which time excessive electrolyte was observed in the exhaust duct of the test fixture (flooding). Testing was terminated at this point without obtaining steady state operation.

Disassembly #2: Disassembly revealed the matrix and cell pair housings to be very wet; however, the reservoir had only absorbed a few grams of electrolyte.

TABLE IV

VERIFICATION TEST CONDITIONS AND CELL PAIRS PERFORMANCE

Test Run No.	Chamber Conditions: Temp. In °F	Chamber Conditions: Dew Point In °F	Chamber Conditions: R.H. %	Cell Pair Performance: Voltage Volts	Cell Pair Performance: Current Amps / Current Density ASF	Length of Run Hrs.	Remarks
Assy #1			90				Cell pair build up was made with 17 wt-% H_2SO_4 (equiv. to 90% R.H. & 77°F). Both matrix and reservoir were saturated.
1	50	40	68	1.70	100.3/123.5	1	This was not a steady state operating point. Unit had a H_2 crossover after one hr. Cell had local dry spots.
Assy #2			5				Cell pair build up was made with 65 wt-% H_2SO_4 but only the matrix was saturated.
	52	42	71	w/o power		42	Conditioning Cell
1	52	42	71	1.736	39.4/48.6	3	This was not a steady state operating point. Current still decreasing.
2	50	40	68	1.810	64.8/80.	2.5	This was not a steady state operating point. Current still decreasing. Excessive electrolyte observed in exhaust duct.
Reservoir reworked - cell hardware used on SSP testing (5 assy & 190 hrs of operation w/o reservoir)							
Assy #3			5				The cell pair build up was made with 68.5 wt-% H_2SO_4 (equiv. to 5% R.H. @ 63°F) and only the matrix was loaded.
1	65	35	32.7	1.99	80.0/98.8	44	
2	65	35	32.7	1.88	52.5/65.	53	
3	65	35	32.7	1.75	34.6/42.8	41	
4	65	53	65.5	1.75	42.5/52.5	48	A small amount of electrolyte was observed on exit edge of cell pair housing - flooding.
5	65	53	65.5	1.85	61.6/76.2	31.5	No additional flooding.
6	65	53	65.5	1.97	76.0/94.	43.5	No additional flooding.
7	65	56.5	74.5	1.97	76.0/94.	21.5	Slight increase in flooding.
8	65	56.5	74.5	1.85	50.0/61.6	75.5	Slight increase in flooding.
9	65	45	48.5	1.85	36.4/45.	45.5	Start of drying - no signs of flooding or matrix dry-out conditions.
10	65	35	32.7	1.85	28.6/35.3	72.5	No signs of matrix dry-out.
11	65	30	26.5	1.85		2	Matrix drying out - steady state conditions were not reached.
	65	35	32.7	w/o power			Rewetting conditions
12	65	35	32.7	1.85	30.0/37.1	7	H_2 crossover indicated - current fairly stable.
	65	42	43	w/o power		17	Rewetting
13	65	35	52.7	1.85	28.0/34.6	5	H_2 crossover indicated - current fairly stable.

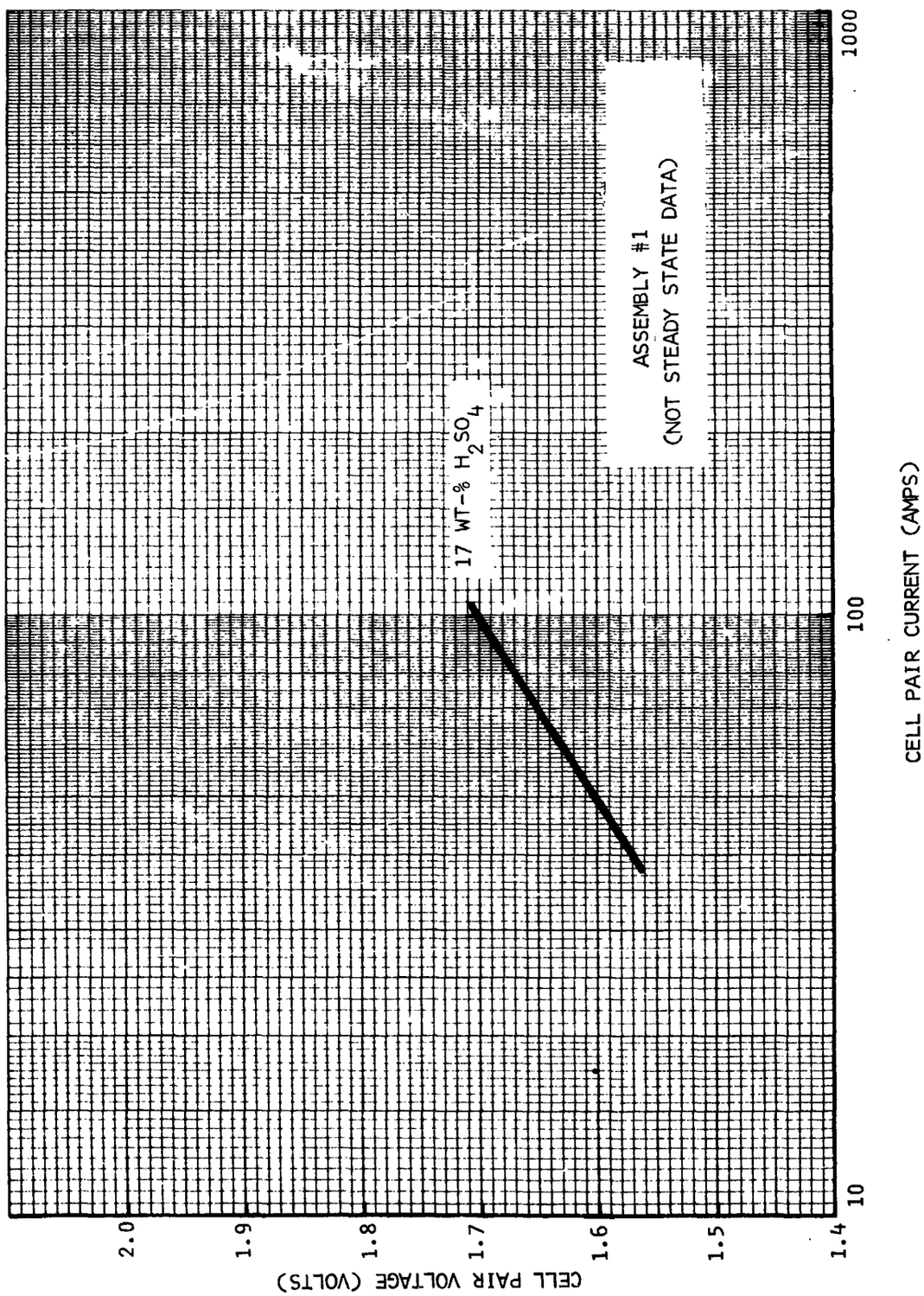

17 WT-% H_2SO_4
ASSEMBLY #1
(NOT STEADY STATE DATA)
CELL PAIR VOLTAGE (VOLTS)
2.0
1.9
1.8
1.7
1.6
1.5
1.4
CELL PAIR CURRENT (AMPS)
10
100
1000

FIGURE 25

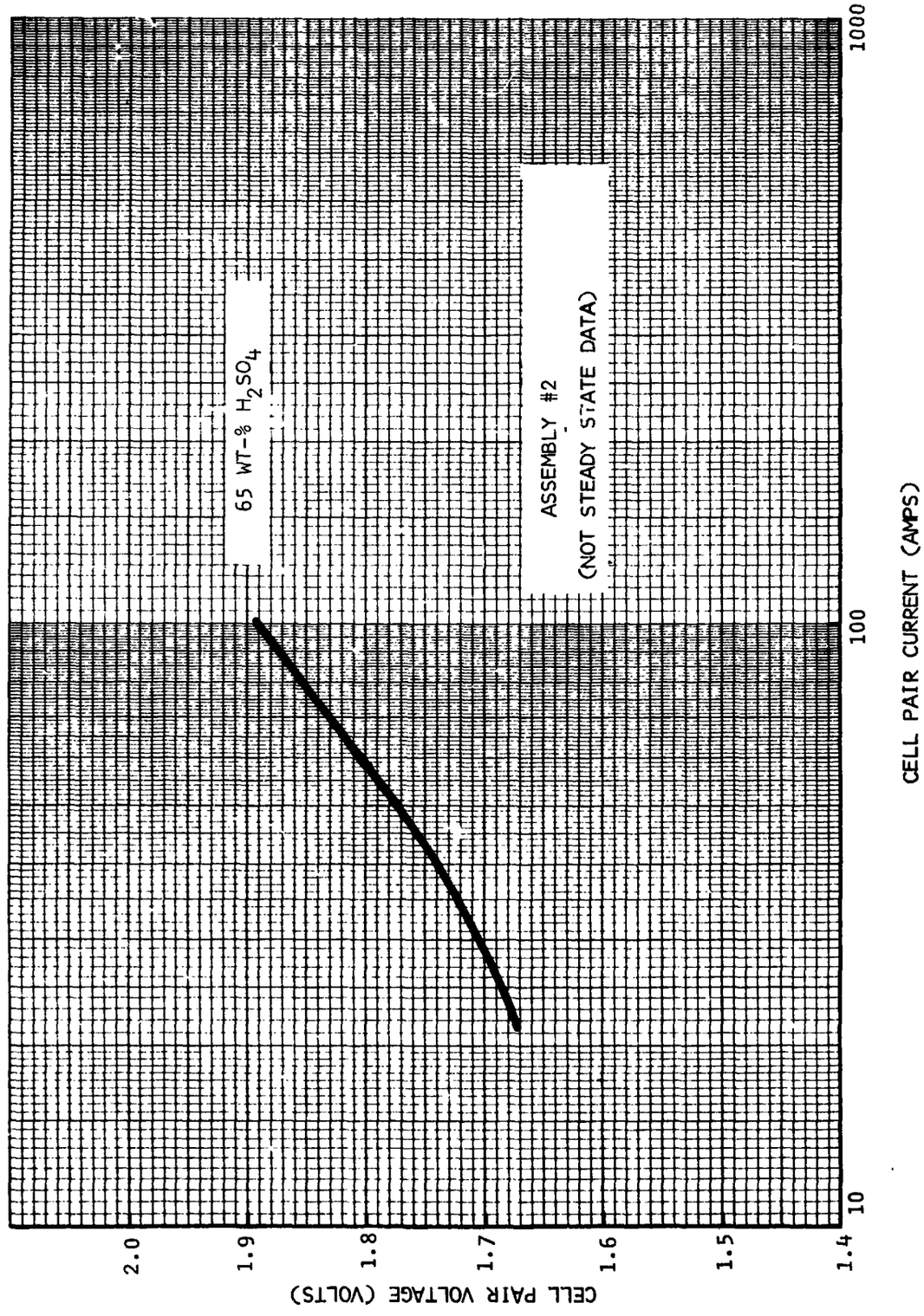

65 WT-% H_2SO_4
ASSEMBLY #2
(NOT STEADY STATE DATA)
CELL PAIR VOLTAGE (VOLTS)
2.0
1.9
1.8
1.7
1.6
1.5
1.4
10
100
1000
CELL PAIR CURRENT (AMPS)

FIGURE 26

SSP design data testing.- At this time the SSP Program required the cell hardware, with exception of the reservoir, to establish WVE design data. This testing consisted of five assemblies/disassemblies of the unit and 190 hours of operation. Data obtained during this testing is presented in Figure 27.

The limited effectiveness of the reservoir experienced in the initial testing was investigated. It was concluded that the hydrogen being generated at the cathode caused a gas barrier at the wicking interface of the reservoir and matrix. During the time the SSP Program Design Data Tests were in progress, the reservoir and the cathode were modified to correct this problem. This is further discussed in the section on the analysis of test results.

Assembly #3.- This assembly incorporated the separated reservoir and the modified cathode. It also used the original anodes which had experienced seven previous assemblies and over 190 hours of testing, which caused removal of some catalyst from the electrode screen. New anodes were not fabricated because of program cost and schedule limitations. The electrolyte loading of the cell pair consisted of saturating only the matrix with H_2SO_4 at 68.5 wt-% concentration. This was equivalent to a non-operating condition of 63°F air temperature and -5°F dew point (5% RH).

Run #1: The unit was placed in the chamber at 65°F air temperature and 35°F dew point (65°F/35°F DP or 32.5% RH) and powered to maintain 80 amps. The unit was held at this condition for 44 hours which provided a steady state point of 1.99 volts and 80.0 amps.

Run #2: The chamber conditions were held at 65°F/35°F DP and the voltage was changed to 1.88 volts and held constant for 53 hours. The steady state performance was established at 1.88 volts and 52.5 amps.

Run #3: The chamber conditions were held at 65°F/35°F DP and the voltage was lowered to 1.75 volts and held constant for 41 hours. This resulted in a steady state performance of 1.75 volts and 34.6 amps. With this data point a performance curve for 65°F/35°F DP could be established as shown in Figure 28.

Run #4: The cell pair voltage was held constant at 1.75 volts and the chamber dew point was increased to 53°F at an air temperature of 65°F (65.5% RH). After 48 hours a small amount of electrolyte was observed on the exhaust duct of the test fixture. A steady state point of 1.75 volts and 42.5 amps was obtained.

Run #5: Chamber conditions of 65°F/53°F DP were held and the voltage was increased to 1.85 volts for 31.5 hours. The steady state point for this condition was 1.85 volts and 61.6 amps. No additional splattering of electrolyte was observed in the exhaust duct.

Run #6: The chamber conditions were held at 65°F/53°F DP and the voltage was increased to 1.97 volts for 43.5 hours. The steady state point for this condition was 1.97 volts and 76 amps. With this data point a performance curve for 65°F/53°F DP could be established as shown in Figure 29 which shows some degradation in cell performance because the current level for 1.97 volts is not higher.

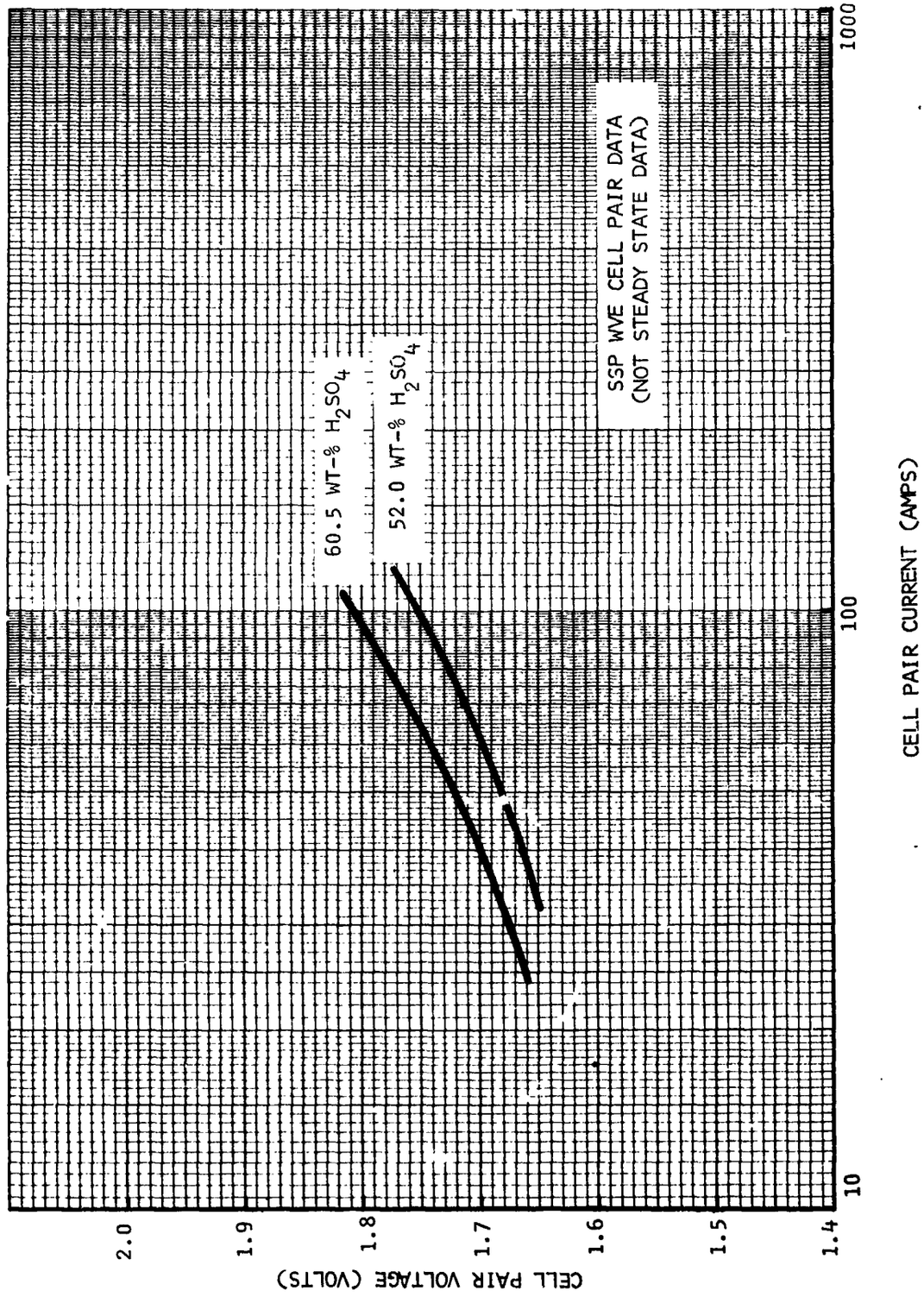

60.5 WT-% H_2SO_4
52.0 WT-% H_2SO_4
SSP WVE CELL PAIR DATA
(NOT STEADY STATE DATA)
2.0
1.9
1.8
1.7
1.6
1.5
1.4
CELL PAIR VOLTAGE (VOLTS)
10
100
1000
CELL PAIR CURRENT (AMPS)

FIGURE 27

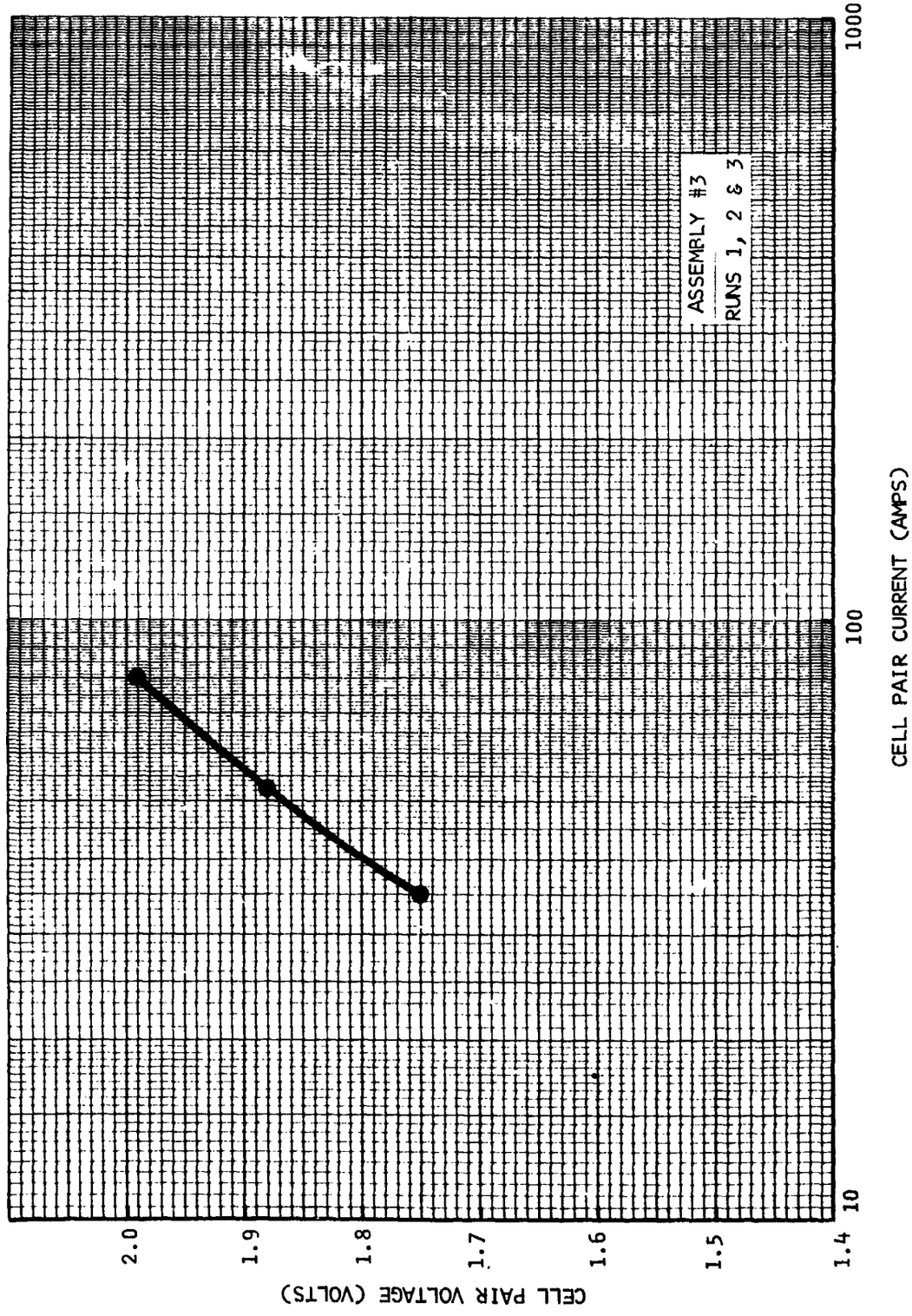

ASSEMBLY #3
RUNS 1, 2 & 3
2.0
1.9
1.8
1.7
1.6
1.5
1.4
CELL PAIR VOLTAGE (VOLTS)
10
100
1000
CELL PAIR CURRENT (AMPS)

FIGURE 28

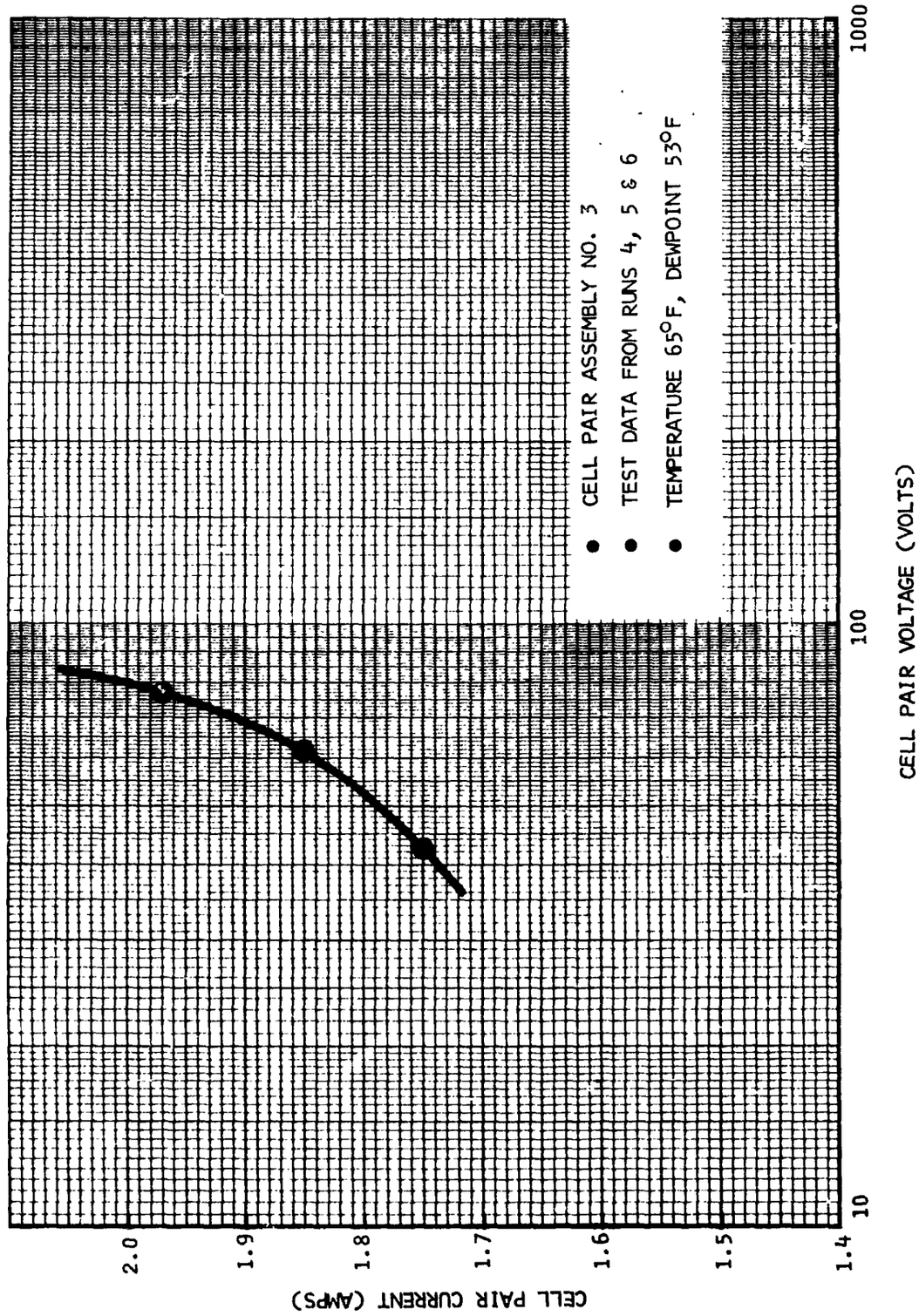

CELL PAIR ASSEMBLY NO. 3
TEST DATA FROM RUNS 4, 5 & 6
TEMPERATURE 65°F, DEWPOINT 53°F
CELL PAIR VOLTAGE (VOLTS)
10
100
1000
CELL PAIR CURRENT (AMPS)
2.0
1.9
1.8
1.7
1.6
1.5
1.4

FIGURE 29

Run #7: The cell pair voltage was held constant at 1.97 volts while the chamber dew point was increased to 56.5°F at 65°F air temperature (75% RH). This condition was held for 21.5 hours at which time cell flooding was observed. The steady state point for this condition was 1.97 volts and 76.0 amps. This test showed additional degradation in cell performance since the current didn't increase with the rise in dew point.

Run #8: Even though flooding was observed at 1.97 volts, an additional data point was required to determine the performance curve at 65°F/56.6°F DP. The chamber conditions were held constant and the voltage was decreased to 1.85 volts for 25.5 hours. Additional flooding was observed during this time; however, a steady state point was obtained at 1.85 volts and 50.0 amps. Significant performance degradation was observed here since the performance is the same as at 35°F DP, as shown in Figure 30.

Run #9: This was the start of the cell pair drying. The voltage was held constant at 1.85 volts and the chamber dew point was lowered to 45°F at an air temperature of 65°F (48% RH). This condition was held for 45.5 hours until a steady state condition was reached. Figure 31 presents a plot of the changing inlet dew point and current vs. time.

Run #10: Cell drying was continued. The voltage remained constant at 1.85 volts and the chamber dew point was lowered to 35°F at an air temperature of 65°F (32% RH). This condition was held for 72.5 hours until a steady state condition was reached. Refer to Figure 31 for a plot of the changing inlet dew point and current vs. time.

Run #11: The power to the unit was held at 1.85 volts and the chamber dew point was lowered to 30°F (26.5% RH). At this condition, the unit operated two hours before hydrogen crossover (cell dry-out) occurred. Steady state operation of the unit was not obtained during this test run.

When dry-out was observed in Run #11 the cell power was disconnected and the chamber air temperature increased to 65°F T_{in} & 35°F dew point. This conditioning was continued for 20 hours to try and rewet the cell matrix. The unit showed a relative weight increase, indicating the cell had become wetter.

Run #12: Power was again applied to the unit with the chamber at 65°F/35°F dew point. After seven hours of operation at this condition, hydrogen crossover occurred, stopping the test.

Power was removed from the unit after dry-out occurred in Run #12 and the chamber dew point was increased to 42°F while maintaining the air temperature at 65°F. This condition was held for 17 hours to try and rewet the cell matrix. The unit showed a relative weight increase indicating the cell had become wetter.

Run #13: Power was applied to the unit and the chamber dew point was decreased to 35°F while maintaining air temperature at 65°F for five hours before the unit dried-out. This concluded the planned test program.

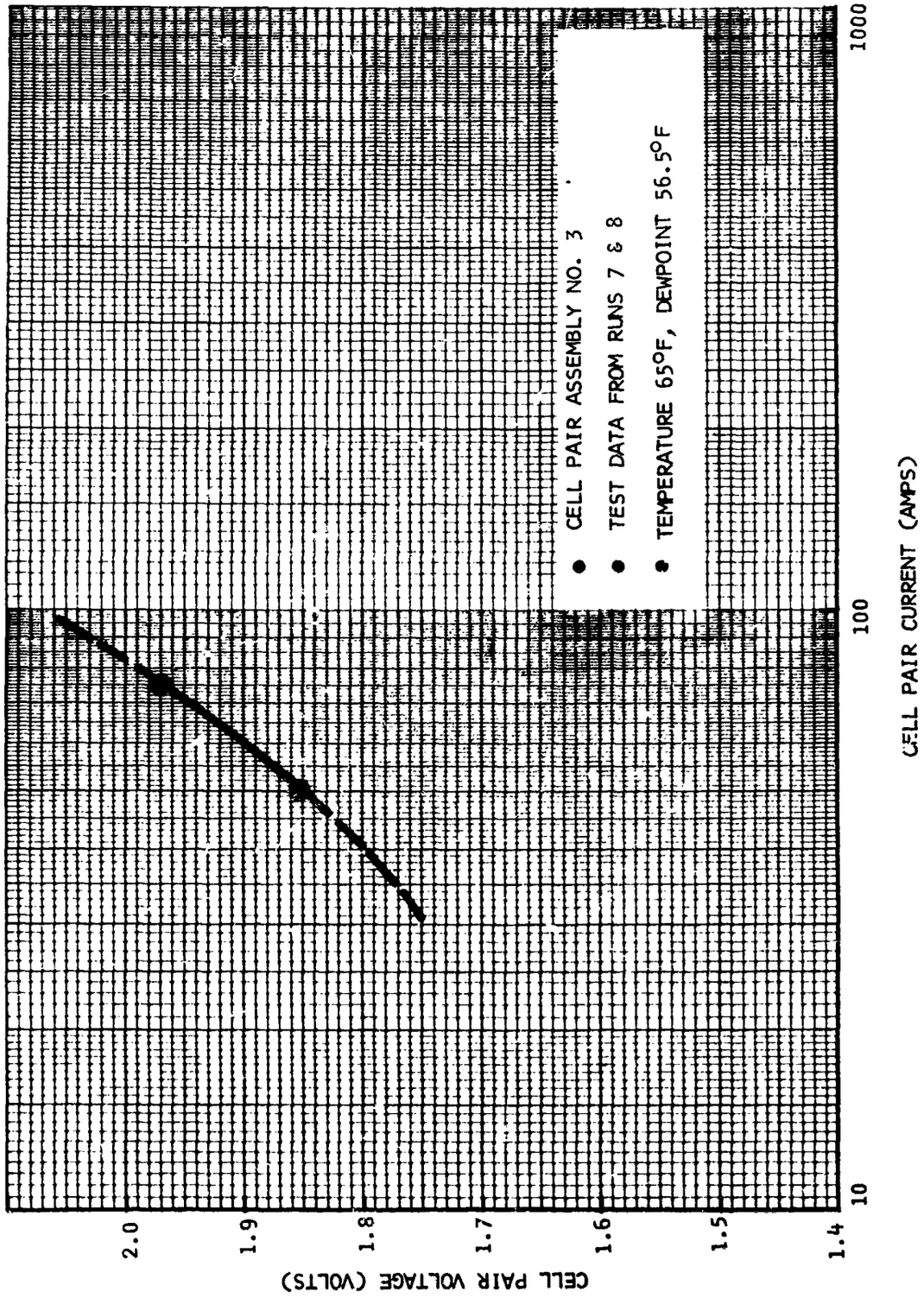

CELL PAIR ASSEMBLY NO. 3
TEST DATA FROM RUNS 7 & 8
TEMPERATURE 65°F, DEWPOINT 56.5°F
CELL PAIR VOLTAGE (VOLTS)
2.0
1.9
1.8
1.7
1.6
1.5
1.4
10
100
1000
CELL PAIR CURRENT (AMPS)

FIGURE 30

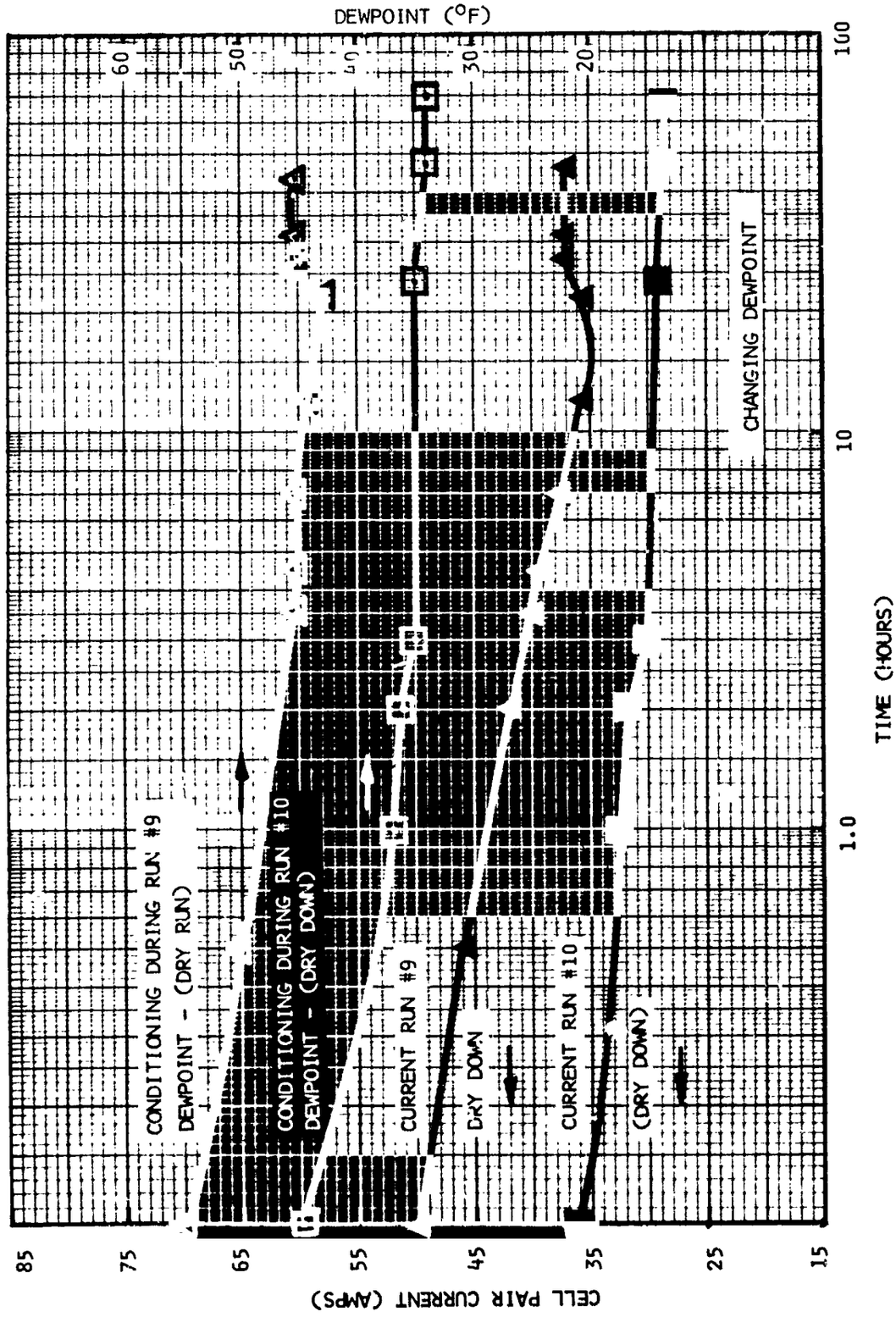

DEWPOINT (°F)
60
50
40
30
20
CHANGING DEWPOINT
CONDITIONING DURING RUN #9
DEWPOINT - (DRY RUN)
CONDITIONING DURING RUN #10
DEWPOINT - (DRY DOWN)
CURRENT RUN #9
DRY DOWN
CURRENT RUN #10
(DRY DOWN)
85
75
65
55
45
35
25
15
CELL PAIR CURRENT (AMPS)
1.0
10
100
TIME (HOURS)

FIGURE 31

During the Phase II testing there were no indications of H_2S or ozone present in the test chamber or hydrogen exhaust.

Post Test Inspection

The cell pair unit was disassembled after Run #13 and inspected. The unit was not turned over during the disassembly. The following detail observations were made:

Both anodes were very wet on the air side. The electrodes were placed, air side up, on a dry sheet of Tissuquartz, but the electrolyte droplets remained on top of the electrodes.

A few droplets of electrolyte were noted in the air passages of both housings.

Matrix from top cell was very wet.

Matrix from bottom cell was very dry (approximately 50% of its area).

Both matrices were black on the anode side indicating anode catalyst deposit.

Both cathodes were stuck to the reservoir and upon separation the catalyst from the cathode stuck to the reservoir.

Reservoir weights:

	At Disassembly (grams)	Dry Weight At Assembly #3 (grams)
Top	535.3	464.0
Bottom	518.6	481.0

There was no evidence of H_2S production.

There was no evidence of metal discoloration or metal corrosion.

Performance Curves

Specific performance curves are discussed in the following paragraphs.

Initial cell performance.- Figure 32 presents the cell pair voltage/current relationship for various electrolyte concentrations at a constant temperature. These curves were generated from data obtained by operating the cell pair through a short (approximately 2-3 minutes total time) voltage sweep immediately after the cell pair was assembled. Because of the short duration of operation the electrolyte did not have time to change temperature or concentration as it does under steady state operation. The electrolyte is, therefore, not conditioned to the chamber conditions or to the temperature change caused by the cell potential.

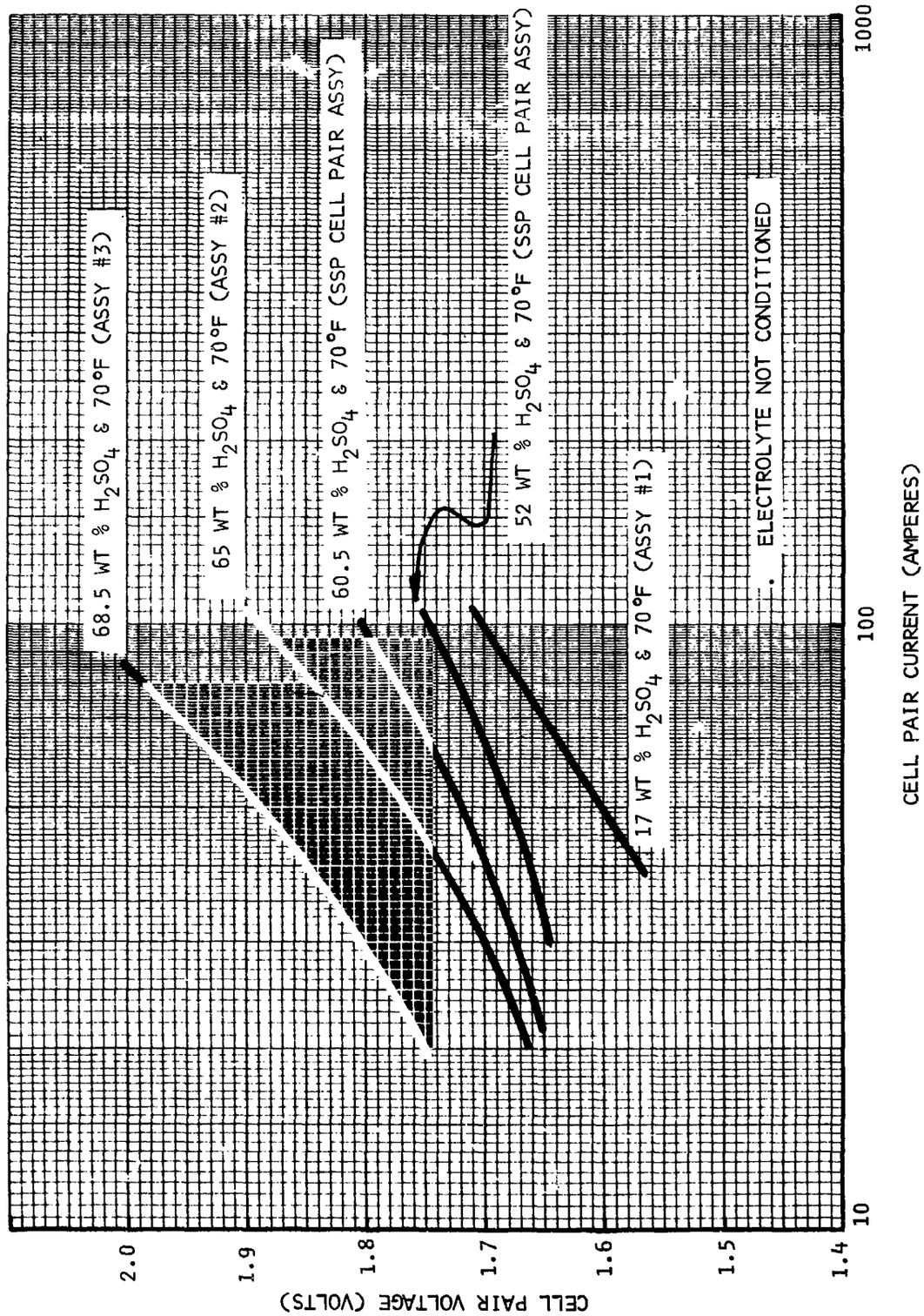

68.5 WT % H_2SO_4 & 70°F (ASSY #3)
65 WT % H_2SO_4 & 70°F (ASSY #2)
60.5 WT % H_2SO_4 & 70°F (SSP CELL PAIR ASSY)
52 WT % H_2SO_4 & 70°F (SSP CELL PAIR ASSY)
17 WT % H_2SO_4 & 70°F (ASSY #1)
ELECTROLYTE NOT CONDITIONED
CELL PAIR VOLTAGE (VOLTS)
2.0
1.9
1.8
1.7
1.6
1.5
1.4
CELL PAIR CURRENT (AMPERES)
10
100
1000

FIGURE 32

Steady state performance.- Figure 33 presents the cell pair voltage/current relationships for various inlet humidities. For a constant inlet temperature and dew point condition the cell pair will have a repeatable, steady state voltage/current relationship as indicated by these curves. However, during the 428 hours of testing a degradation of cell performance was noted. This is shown by the data point from the tenth run which does not fall on the previous performance curve for the respective inlet conditions. The performance curve for the highest humidity (65°/56.6°F DP) should fall to the right of the lower humidity curves. This also indicates a degradation in the cell's performance.

Current vs. time due to step change in voltage.- Figure 34 presents the change in cell pair current with respect to time as a result of a step change in operating voltage while holding the chamber conditions constant. These curves show that it takes 30 to 40 hours of operating at a set condition before steady state performance is obtained.

Current vs. time due to a change in dew point.- Figure 31 presents the change in cell pair current with respect to time as a result of lowering the dew point while operating the unit at constant voltage and inlet temperature. These curves also show that it takes 30 to 40 hours of operating at a set condition before steady state performance is obtained.

Current vs. inlet dew point.- Figure 35 presents the cell pair current inlet air dew point relationship at various voltages. Because of the degradation in the cell's performance after run number 5 of assembly number 3, the desired plots of a maximum, nominal and minimum voltage setting were not obtained. However, the dashed lines show where the maximum and nominal voltage performance curves should lie based on one or two data points.

Post Test Flooding and Dry-Out Investigation

An investigation was made to determine the cause of the cell pair flooding at humidities below 90% RH (maximum design point) and drying out of the matrix at humidities above 5% RH (minimum design point). Three major areas were investigated:

Determination of the specific volume changes in the electrolyte for a wide range in humidities and for various temperatures (reference Figure 36).

Determination of the percentage of the reservoir capacity which was used by the electrolyte for the actual and theoretical cases (reference Appendix G).

Determination of wicking characteristics between the matrix and the reservoir against one g condition and the effectiveness of the reservoir (reference Appendix G).

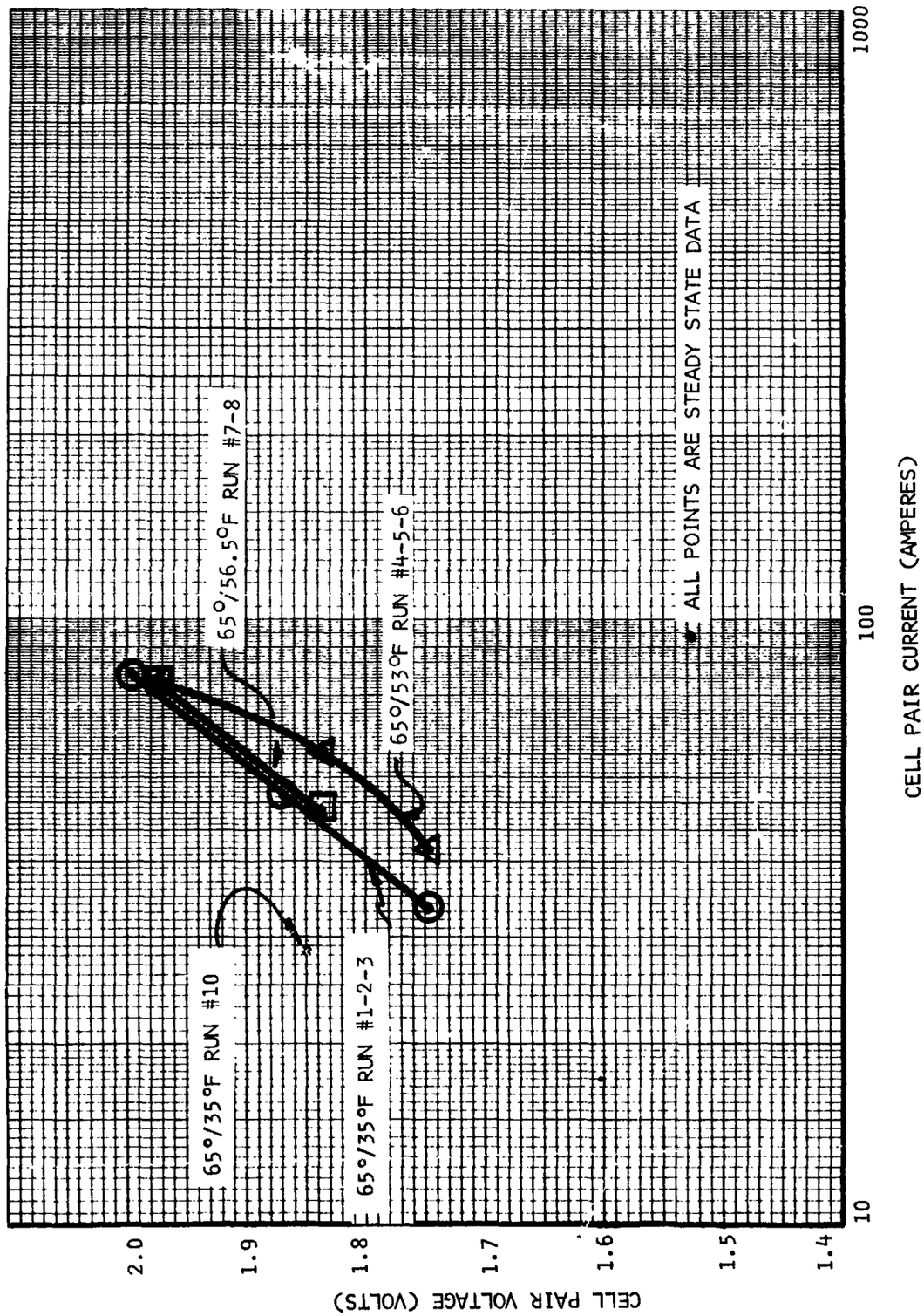

65°/35°F RUN #10
65°/35°F RUN #1-2-3
65°/53°F RUN #4-5-6
65°/56.5°F RUN #7-8
ALL POINTS ARE STEADY STATE DATA
CELL PAIR VOLTAGE (VOLTS)
2.0
1.9
1.8
1.7
1.6
1.5
1.4
CELL PAIR CURRENT (AMPERES)
10
100
1000

FIGURE 33

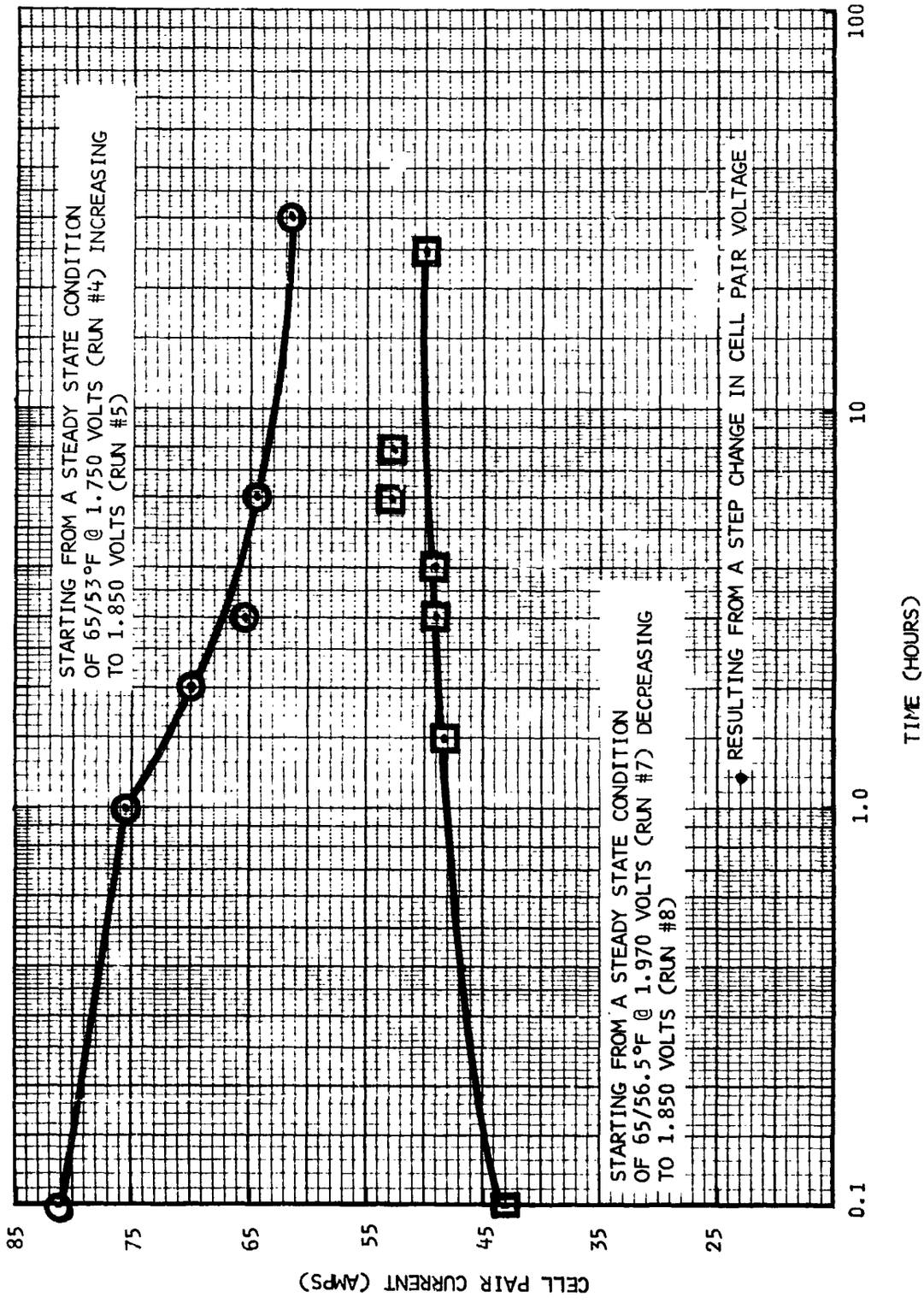

STARTING FROM A STEADY STATE CONDITION
OF 65/53°F @ 1.750 VOLTS (RUN #4) INCREASING
TO 1.850 VOLTS (RUN #5)
STARTING FROM A STEADY STATE CONDITION
OF 65/56.5°F @ 1.970 VOLTS (RUN #7) DECREASING
TO 1.850 VOLTS (RUN #8)
RESULTING FROM A STEP CHANGE IN CELL PAIR VOLTAGE
CELL PAIR CURRENT (AMPS)
85
75
65
55
45
35
25
TIME (HOURS)
0.1
1.0
10
100

FIGURE 34

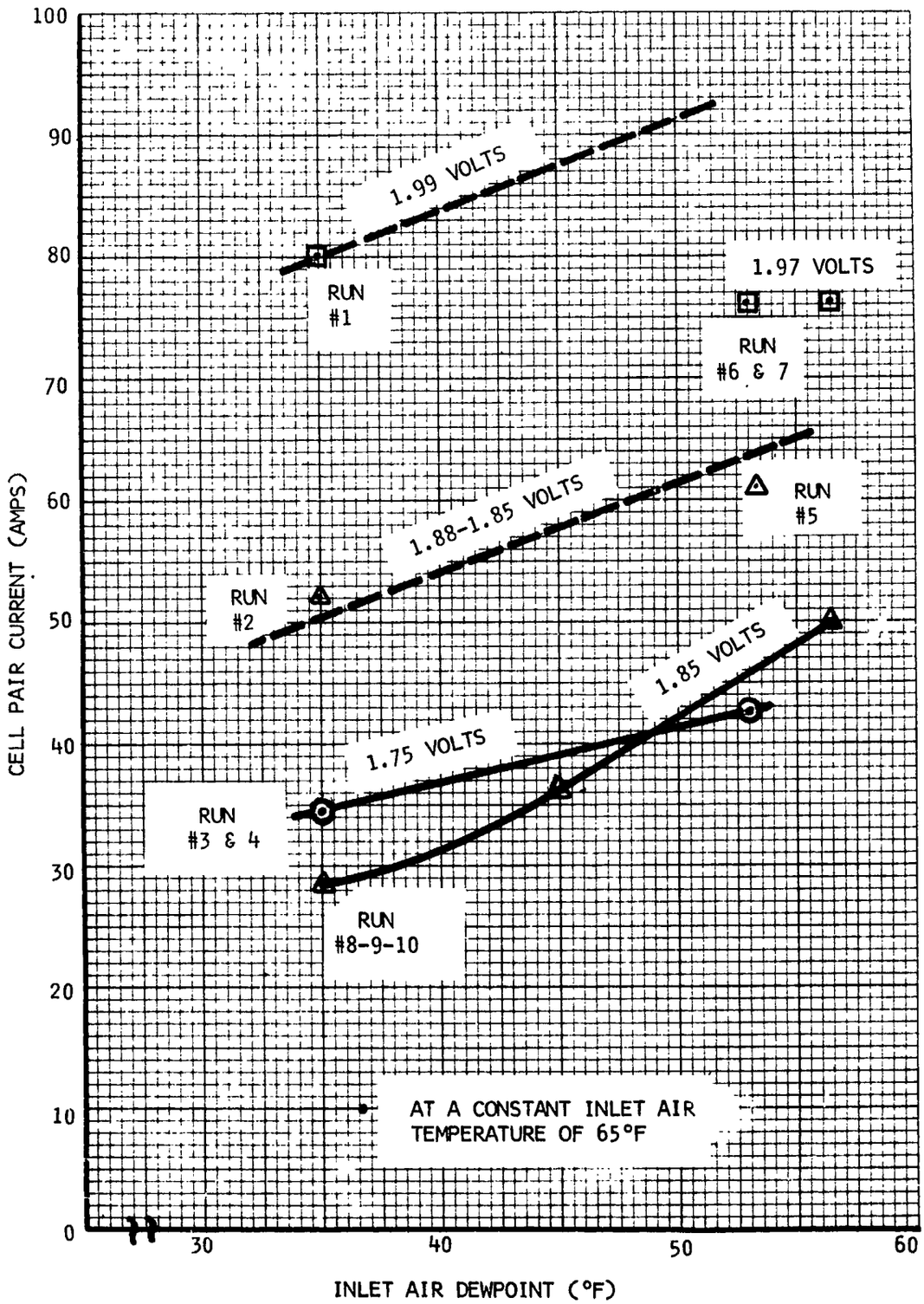
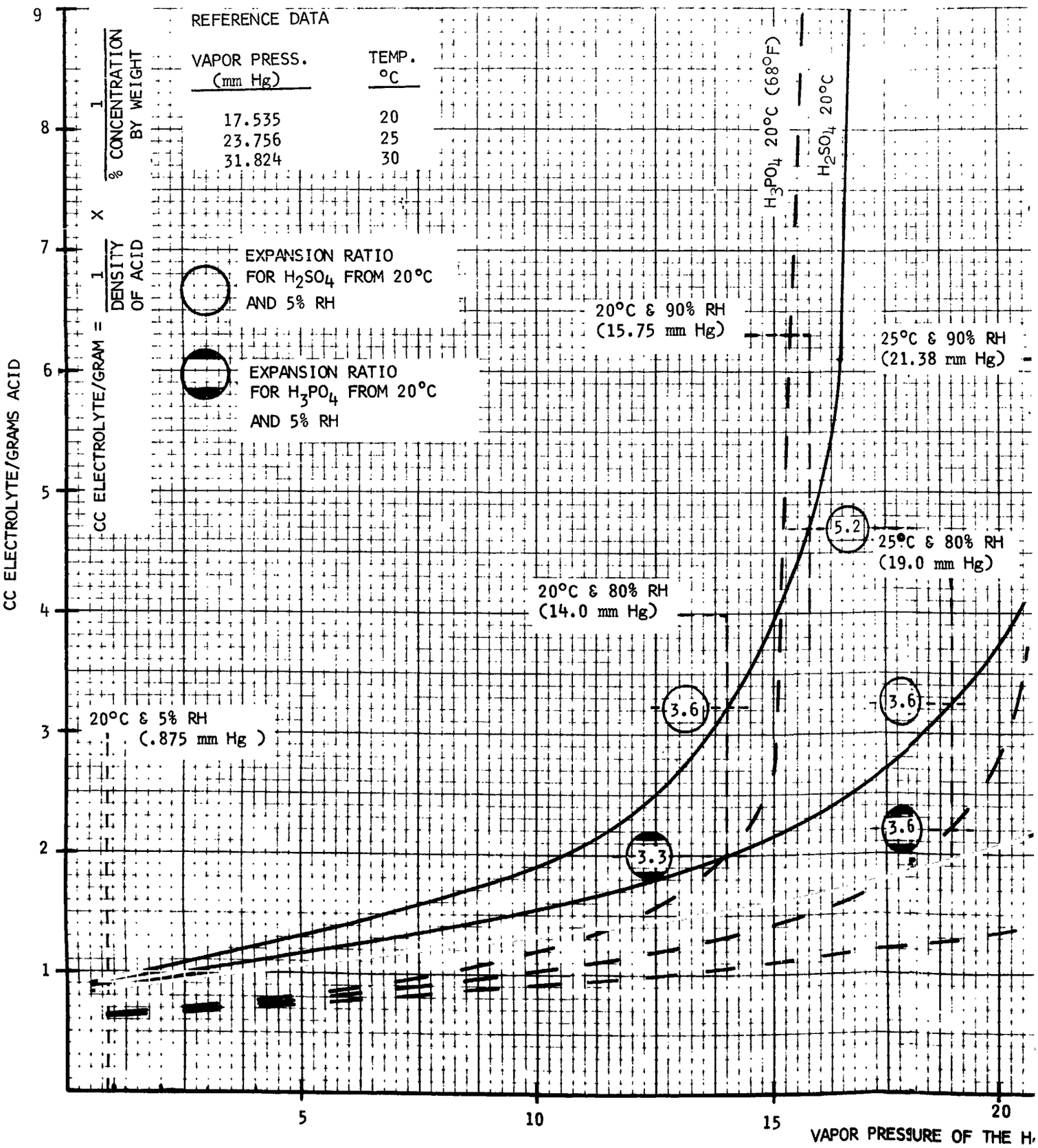

100
90
80
70
60
50
40
30
20
10
0
CELL PAIR CURRENT (AMPS)
30
40
50
60
INLET AIR DEWPOINT (°F)
1.99 VOLTS
RUN #1
1.97 VOLTS
RUN #6 & 7
1.88-1.85 VOLTS
RUN #5
RUN #2
1.85 VOLTS
1.75 VOLTS
RUN #3 & 4
RUN #8-9-10
AT A CONSTANT INLET AIR TEMPERATURE OF 65°F

FIGURE 35

Hamilton Standard DIVISION OF UNITED AIRCRAFT CORPORATION

FOLDOUT FRAME 1



FOLDOUT FRAME 2

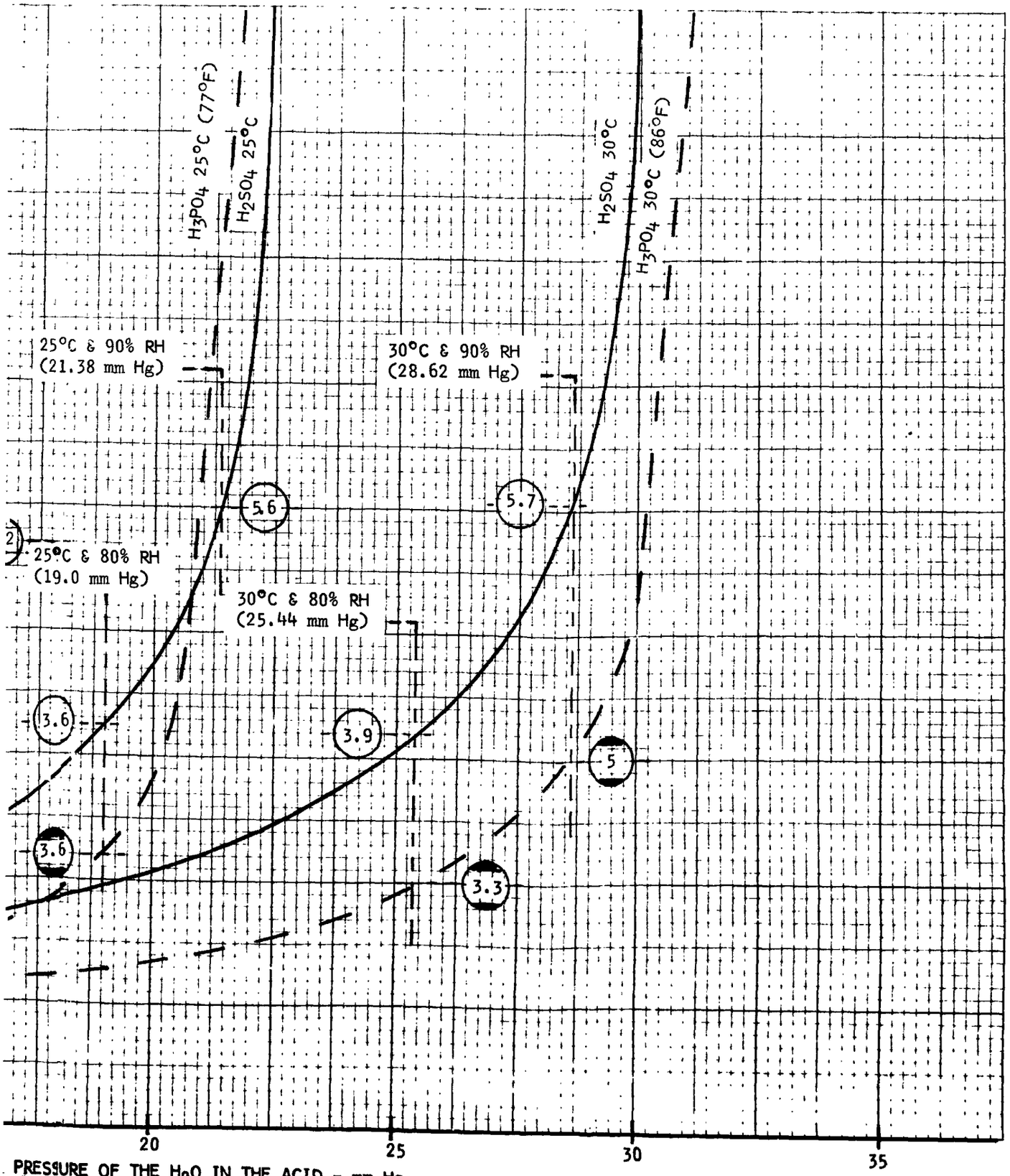


FIGURE 36

The original sizing of the reservoir was based on using a mixed acid electrolyte and a single sheet of Tissuquartz for a matrix. The volumetric change of the electrolyte over a relative humidity range of 90% to 5% was calculated to be 3.5:1. This calculation was based on extrapolated data available at the time, which did not cover this full range in relative humidity. The amount of electrolyte required to fill the matrix was determined to be 12.5 cc, which required a reservoir having a capacity of approximately 44 cc/cell using a 3.5:1 ratio.

The investigation revealed that the original reservoir sizing for the cell pair tested using H_2SO_4 should have been 100 cc capacity/cell for a range of 5% to 90% relative humidity (approximated 6:1 ratio). This new sizing is based on additional electrolyte data (Figure 36), a double Tissuquartz sheet for a matrix, and an 80% reservoir capacity factor (reference Appendix G).

Material Wicking Tests

As a result of the Phase I testing and cell concept, NASA requested additional information on the wicking characteristics of various materials. A test program was established and completed revealing the wicking characteristics presented in Figure 37.

Test Program

The test program selected six materials, which had possible wicking interface relationships, for the wicking tests. Testing was performed per an approved test plan (reference Appendix H) where the wicking rate and percent absorption were determined for each material. The percent absorption is defined as:

$$\% \text{ Absorption} = \frac{\text{Saturated Weight } (H_2O) - \text{Dry Weight}}{\text{Dry Weight}} \times 100$$

Test Results

The wicking rates of the selected materials, except for the Pratt & Whitney Aircraft SKN 42901 electrode, are presented in Figure 37. The Pratt & Whitney Aircraft electrode material could not be wetted and, therefore, a wicking rate was not obtained.

The following percent absorption was obtained for each material:

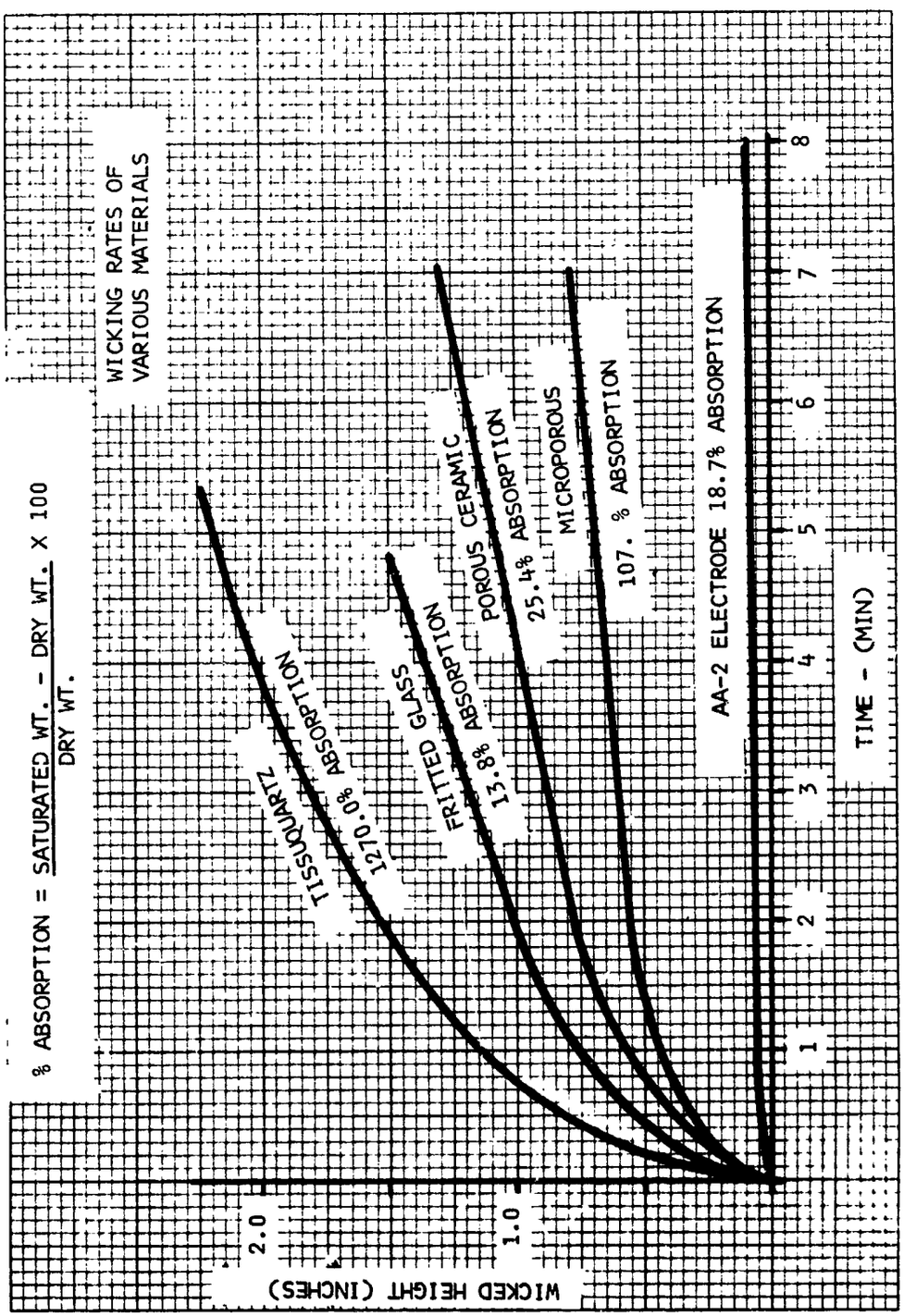

WICKING RATES OF VARIOUS MATERIALS
% ABSORPTION = SATURATED WT. - DRY WT. X 100 / DRY WT.
TISSUQUARTZ
1270.0% ABSORPTION
FRITTED GLASS
13.8% ABSORPTION
POROUS CERAMIC
25.4% ABSORPTION
MICROPOROUS
107. % ABSORPTION
AA-2 ELECTRODE 18.7% ABSORPTION
TIME - (MIN)
1
2
3
4
5
6
7
8
WICKED HEIGHT (INCHES)
1.0
2.0

FIGURE 37

Material	Pore Size (microns)	Absorption %
Tissuquartz	1-6	1270.0
Fritted Glass	30-40	13.8
Porous Ceramic	31-41	25.4
Microporous Rubber	1-6	107.0
AA-2 Electrode	Not Available	18.7
P&WA SKN 42901 Electrode	Not Available	Could not wet

Hydrogen Sulfide Investigation

In the Phase I study is was pointed out that H_2S had been generated and detected during electrolysis tests conducted by Pratt & Whitney Aircraft, which used H_2SO_4 as the electrolyte. The generation was noted in concentrations of H_2SO_4 above 45 wt-%. In order to confirm this production of H_2S with H_2SO_4 as the electrolyte and the lack of H_2S production when a mixed acid electrolyte was used a special test program was conducted (reference test plan, Appendix J).

According to thermodynamic considerations sulfuric acid can be reduced by hydrogen to sulfur and hydrogen sulfide (H_2S). With the proper catalyst the reduction rate becomes appreciable, especially as the temperature and acid concentration are increased. Previous work at Pratt & Whitney Aircraft on the reduction of sulfuric acid at hydrogen electrodes indicated that at temperatures below 80°F and acid concentrations less than 45 wt-% the reduction rate was negligible. Based on this information a mixture of sulfuric and phosphoric acids (5 to 7 weight ratio), in which the sulfuric acid concentration is less than 45 wt-%, was proposed as the electrolyte in Phase I for the WVE cell design. The mixed acid has the superior electrochemical performance of sulfuric acid and prevents the possibility of solids formation characteristic of phosphoric acid at high concentrations.

Subsequently, small cell testing at Hamilton Standard with both 70 wt-% sulfuric acid and 85 wt-% mixed acid revealed no detectable H_2S in the hydrogen gas during cell operation; however, when the cells were allowed to stand at open circuit for a few hours with a slow hydrogen gas purge over the cathode, H_2S was formed. In similar tests with a relatively large volume of free electrolyte (500 ml for a 1 inch^2 cathode) H_2S could not be detected under any conditions over a test period of 100 hours (reference paragraph 3.1 of Test Plan).

From these results it was concluded that H_2S has a high solubility in the electrolyte such that $P_{H_2S}/C_{H_2S} << 1$, where P_{H_2S} is the partial pressure of H_2S (mm Hg) and C_{H_2S} is the concentration (moles/liter), so that during cell operation the H_2S that is produced at the cathode diffuses to the anode where it is oxidized back to H_2SO_4 via.

$$H_2SO_4 + 8\,H + 8\,e \underset{\text{anodic}}{\overset{\text{cathodic}}{\rightleftharpoons}} 4\,H_2O + H_2S.$$

Thus the concentration of H_2S in the electrolyte during cell operation remains at a constant steady state level which is low enough to prevent a detectable H_2S partial pressure in the hydrogen gas. The fraction of the current going to H_2SO_4 reduction and H_2S oxidation is small since typical current efficiencies for hydrogen production are generally about 98% and some of this loss could be due to hydrogen leakage.

It was determined experimentally that H_2S oxidation to H_2SO_4 on a platinum electrode does not occur to any extent below potentials of 1.2 volts. Therefore, a cell on open circuit ($\approx$0.95 volts) cannot oxidize H_2S, and as long as hydrogen gas is supplied the H_2S concentration will continue to increase via the reaction

$$H_2SO_4 + 4\,H_2 \longrightarrow H_2S + 4\,H_2O.$$

The time required to build up the H_2S partial pressure to noticeable levels will depend on the catalyst area and the electrolyte volume.

Tests were run to determine the extent to which H_2S or sulfur would poison the cathode catalyst (reference paragraph 3.2 of test plan). A platinized platinum electrode (1 in^2 geometrical area) with a true surface area of $\approx$1200 in^2, determined by double layer capacitance and hydrogen absorption, was held at a constant current of 50 ma (hydrogen evolution) for 100 hours in 65 wt-% H_2SO_4 at room temperature. Periodically the fraction of the surface covered with atomic hydrogen was measured coulometrically via potential time sweeps at a constant anodic current of 10 ma. Typically, the duration of the hydrogen adsorption measurements was less than 15 sec., and in no case was the potential allowed to exceed 1.0 volt so that the sulfur species on the electrode would not be removed by oxidation. No significant change in catalytic activity or hydrogen adsorption occurred in the first eight hours, but after 100 hours over 90% of the surface had been covered with sulfur species and at this time the cathode potential at 50 ma had decayed only 20 mv from its initial value.

A final potential sweep was made to 1.4 volts to verify that the observed cathode poisoning was due to sulfur species (oxidation peak at 1.3 volts) rather than some other impurity. Also, after holding the electrode at 1.4 volts for five minutes all traces of reduced sulfur species had been removed and the electrode exhibited its initial catalytic activity and hydrogen adsorption capacity. From these tests it was concluded that cathode poisoning due to H_2S or sulfur is not serious.

Analyses of Testing Results

The following analyses of the test data were made in order to resolve cell design problems and to provide better understanding of the WVE cell operation:

- Initial Reservoir/Matrix Wicking Barrier
- Cell Pair Efficiency
- Performance Degradation

Initial Reservoir/Matrix Wicking Barrier

Tests results of cell pair assemblies one and two showed insufficient wicking between the matrix and the reservoir caused by a gas barrier at the wicking interface. This gas barrier was formed as follows:

Hydrogen gas was generated at the same interface where the transfer of electrolyte was required. This was not noted in the Phase I test cell because the humidity changes were made without the cell being powered.

The cell pair common reservoir allowed wicking action to occur at both faces. The Phase I cell had hydrogen gas on the reservoir back side. As the common reservoir absorbed the excess electrolyte, it would trap the hydrogen gas in the inner pores which reduced the effective volume capacity of the unit. As electrolyte was removed from the saturated reservoir it was replaced with hydrogen gas at the surface of the reservoir which in time formed a gas barrier around the reservoir.

To eliminate this wicking problem the reservoir was cut horizontally in half and the two halves separated with a 0.060" spacer. In addition, the cathode was reworked at the reservoir interface by removing the catalyst on the screen and filling in the entire void with Tissuquartz.

Cell Pair Efficiency

The cell pair efficiency was calculated on the basis of the assembly number three steady state test data (reference Table V). Calculations for the efficiency were based on the actual production rate of hydrogen compared to the theoretical hydrogen production for the given current, temperature and pressure conditions of the cell pair. A computer program which was established on the SSP Program, was used for calculating the average electrolyte temperature, the outlet air temperature and the hydrogen production.

$$\text{Cell Pair Current Efficiency} = \frac{\text{Hydrogen Flow Rate} \times .9754^{(1)}}{\text{Calculated Hydrogen Flow Rate}} \times 100$$

The inefficiencies (1.1 to 4.8%) are attributed to the following:

The current loss caused by the formation of H_2S at the cathode which diffuses to the anode to become oxidized (reference H_2S Investigation).

(1) A correction factor (0.9754 must be applied to the measured hydrogen exhaust flow to account for the partial pressure of water of the gas.

TABLE V

CELL PAIR STEADY STATE TEST DATA
(ASSEMBLY NO. 3)

Total Cell Area = 0.81 sq. ft.
Air Flow through Cell = 18 SCFM

ΔP Across Cell Pair @
18 CFM = 1.8 inches of H_2O

	CHAMBER CONDITIONS		CELL PERFORMANCE TEST DATA					CALCULATED VALUES		
Run No.	Inlet Air Temp. (°F)	Inlet Air Dew Point (°F)	Voltage (Volts)	Current (Amps)	H_2 Prod. (cc/min)	Outlet Air Temp. (°F)	Outlet Air Temp. (°F)	Average Electrolyte Temp. (°F)	H_2 Production (cc/min)	Current Efficiency %
1	65	35	1.99	80.0	600	72.5	75	87	602	98.2
2	65	35	1.88	52.5	391	69	70.6	77.3	395	96.6
3	65	35	1.75	34.6	258	67	67.9	71.4	260.4	96.1
4	65	53	1.75	42.5	318	67	68.6	72.8	319.8	97.0
5	65	53	1.85	61.6	470	68.5	71.3	78.7	463.6	98.9
6	65	53	1.97	76.0	580	72.5	74.3	85.3	572	98.9
7	65	56.5	1.97	76.0	580	72.5	74.3	85.3	572	98.9
8	65	56.5	1.85	50.0	378	69.5	70.1	76.1	376.3	98.0
9	65	45	1.85	36.4	270	68.5	68.7	73.1	274	96.1
10	65	35	1.85	28.6	210	67.0	67.9	71.4	215.2	95.2

The current loss from the hydrogen and oxygen diffusion across the matrix.

Hydrogen leakage from the cell pair and the hydrogen exhaust lines.

The low efficiency of run number 10 (95.2%) would indicate that a small amount of hydrogen crossover was occurring. This hydrogen would not be detected in the chamber air because it would be burned up as it passed through the cell at the anode.

Performance Degradation

The degradation of cell performance with time can be traced to a loss in anode performance from the fact that both the cell open circuit potential and the operating voltage at constant current increased with time. It can be shown from electrokinetic considerations that a loss in cathode performance would give no change in the open circuit potential unless $H_2SO_4 \rightleftharpoons H_2S$ reaction was interfering, in which case the cell open circuit would have decreased with time. On the other hand the anode open circuit potential is a mixed potential in that the anodic part involves water oxidation and the cathodic part catalyst-oxide reduction, and the value of this potential would depend on the relative rates of these two reactions. It can be shown that the cell open circuit potential and the voltage at constant current would both increase with time only if the rate of water oxidation at the anode decreased.

There are two possibilities that would cause this effect: 1) adsorption of impurities, and 2) electrode structure changes. The first alternative is difficult to accept since the high operating potentials ($\approx$ 1.8 volts) of the anode would certainly oxidize any impurities present on the anode surface. It is more reasonable to believe that structural changes either in the catalyst surface or the electrode micro structure resulted in a loss in effective surface area for water oxidation. Indications of this were found when the cell was disassembled and it was noticed that a considerable amount of catalyst washed off the anode surface with water. It is conceivable that the Teflon binder (which binds the catalyst to the electrode) could be slowly oxidized at the high anode potentials, resulting in a gradual degradation of the electrode structure. This is supported by the test results. After run 5, where the cell was operating at 1.97 volts, an appreciable degradation of performance was observed. Since a relatively small amount of Teflon binder was used in these anodes it is also possible that oxygen gas evolution may have been sufficient to have mechanically disrupted the catalyst particles.

Anodes with a higher Teflon content are currently being evaluated on the SSP Program. (1) There is also the possibility of eliminating the Teflon altogether by developing a different electrode/catalyst bonding technique.

---

(1) No performance degradation or catalyst loss could be detected after 600 hours of operation on the latest Hamilton Standard fabricated anode.

APPENDIX A

LIST OF SYMBOLS AND ABBREVIATIONS

ABBREVIATIONS

AA-2	Electrode manufactured by American Cyanamid Company
ASF	Amps/Sq. Ft.
Avg.	Average
BMI	Battelle Memorial Institute
BTU	British Thermal Units
°C	Degrees Celsium (Centigrade)
cc	Cubic Centimeter
cfm	Cubic Feet Per Minute
cm^2	Centimeter Squared
D	Diffusivity for Water Vapor in Air
dc	Direct Current
DP	Dew Point
F	Faraday's Constant
°F	Degrees Fahrenheit
Ft.	Foot
h	Length of Electrode
H_2	Hydrogen
Hg	Mercury
hr.	Hour
H_2O	Water
H_2S	Hydrogen Sulfide
H_2SO_4	Sulfuric Acid
H_3PO_4	Phospheric Acid
i	Current
in.	Inches

ABBREVIATIONS (Continued)

I_v	Cell Electrical Current at Constant Potential
IR	Voltage
k	Rate Constant
KW	Kilowatt
l	Matrix Thickness
lb.	Pound
ma	Milliampere
mv	Millivolt
min.	Minute
mm	Millimeter
n	Anions Present in Solution
O_2	Oxygen
psi	Pounds Per Square Inch
psia	Pounds Per Square Inch Absolute
PVC	Polyvinylchloride
P_{H_2O}	Partial Pressure of Water Vapor
P_{O_2}	Partial Pressure of Oxygen
P_v	Vapor Pressure
P&WA	Pratt & Whitney Aircraft, Division of United Aircraft Corp.
rpm	Revolutions Per Minute
R	Gas Constant
RH	Relative Humidity
SSP	Space Station Prototype
sec.	Second
scc	Standard Cubic Centimeter

ABBREVIATIONS (Concluded)

T	Absolute Temperature
T_{in}	Inlet Temperature
TBD	To Be Determined
U	Bulk Air Velocity
UAC	United Aircraft Corporation
ν	Kinematic Viscosity
WVE	Water Vapor Electrolysis
wt	Weight
wt-%	Weight Percent
x	Distance From Air Inlet to Electrode
μ	Micro (10^{-6})
ρ	Electrolyter Resistivity
δ	Thickness of the Boundary Layer
%	Percent
$\approx$	Approximately Equals
=	Equals
$\ll$	Much Less Than
e	Electron

APPENDIX B

REFERENCES

REFERENCES

1. "Research on Electrolysis of Water Under Weightless Conditions", by J. E. Clifford, J. Gates, J. McCallum and L. Faust, Report No. MRL-TDR-62-44, Aerospace Medical Research Laboratories, Wright Patterson Air Force Base, Ohio, May, 1962.

2. "Water Vapor Electrolysis Cell With Phosphoric Acid Electrolyte", by J. E. Clifford, Paper No. 670851, Society of Automotive Engineers, October, 1967.

3. "Water Vapor Electrolysis", by T. Wydeven and E. Smith, Aerospace Medicine, Vol. 38, No. 10, pp 1045-1048, October 1967.

4. "Water Electrolysis: Prospect For Future", by T. Wydeven and R. W. Johnson, Transactions of the ASME: Journal of Engineering for Industry, Vol. 90, pp 531-540, November, 1968.

5. "Prototype Electrolyzer For Oxygen Generation From Water Vapor", by E. L. Smith and T. Wydeven, Journal of Spacecraft and Rocket, Vol. 7, No. 8, pp 1004-1006, August 1970.

6. "Study of Water Vapor Electrolysis Unit" by J. E. Clifford, B. C. Kim, and E. S. Kolic, NASA CR-1531, March, 1970.

APPENDIX C

CHEMICAL PROPERTIES OF H_2SO_4 AND H_3PO_4

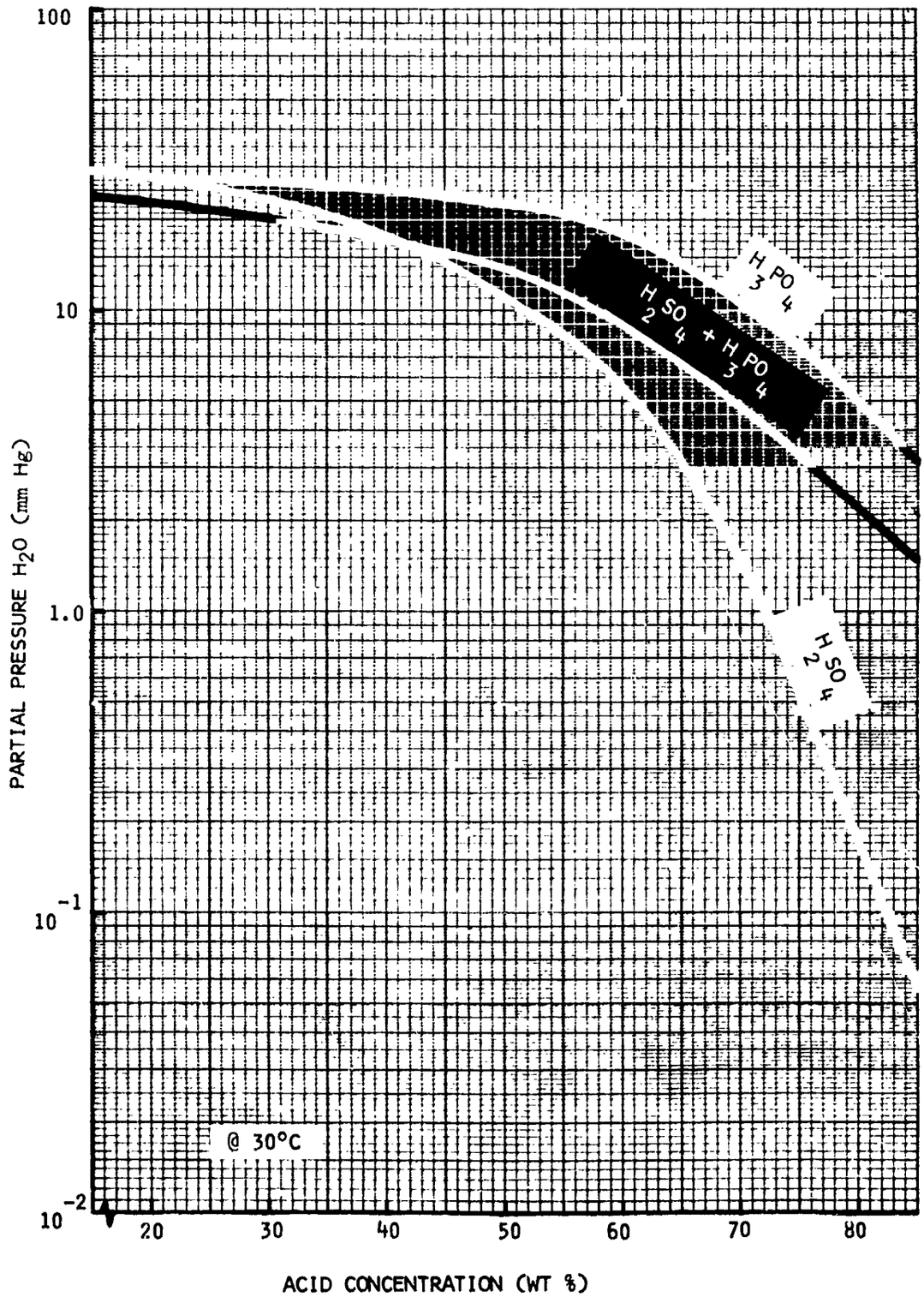

100
10
1.0
10^{-1}
10^{-2}
PARTIAL PRESSURE H_2O (mm Hg)
20
30
40
50
60
70
80
ACID CONCENTRATION (WT %)
H_3PO_4
$H_2SO_4 + H_3PO_4$
H_2SO_4
@ 30°C

FIGURE C-1

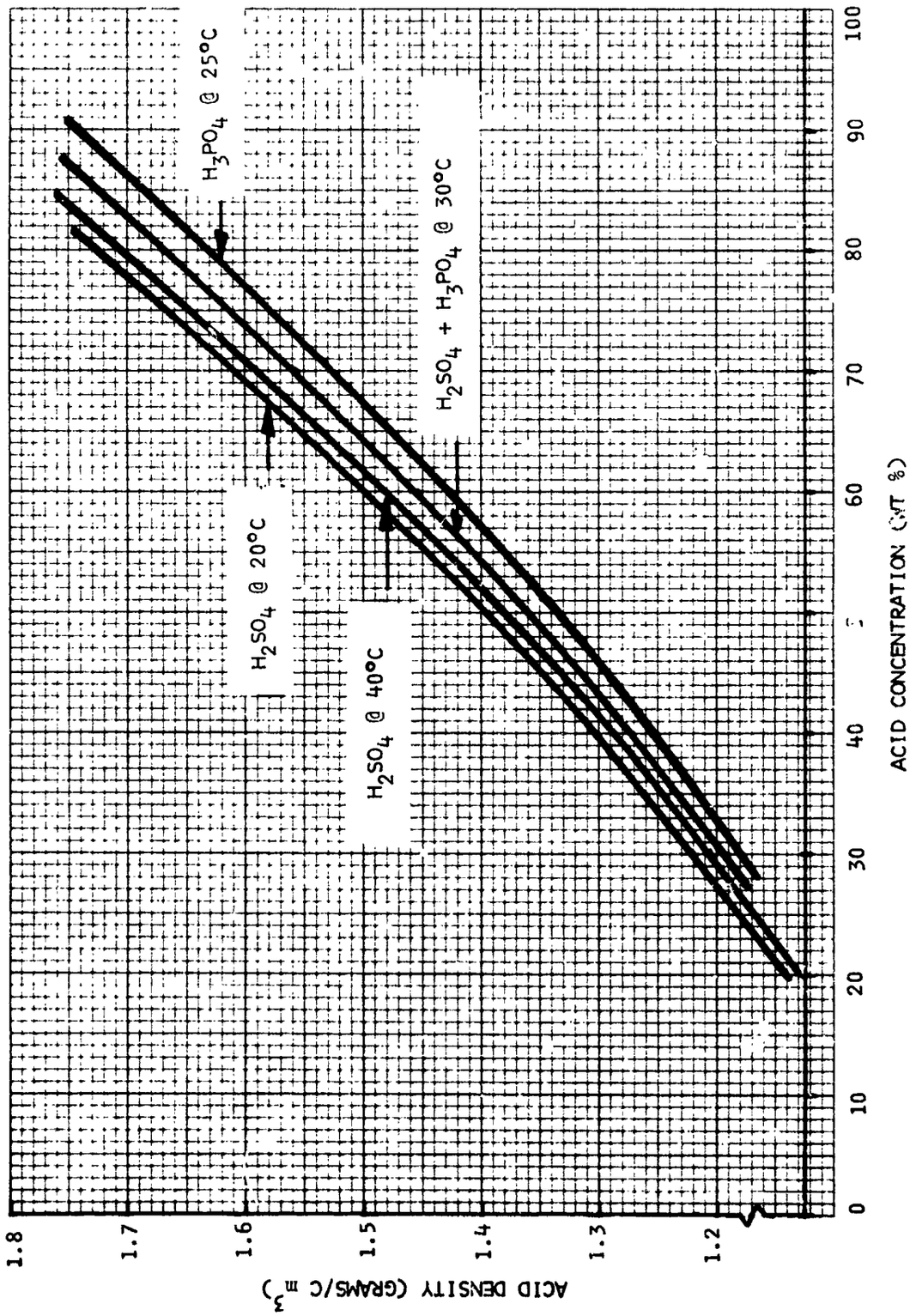

H_3PO_4 @ 25°C
$H_2SO_4 + H_3PO_4$ @ 30°C
H_2SO_4 @ 20°C
H_2SO_4 @ 40°C
ACID CONCENTRATION (WT %)
ACID DENSITY (GRAMS/C m^3)
0
10
20
30
40
60
70
80
90
100
1.8
1.7
1.6
1.5
1.4
1.3
1.2

FIGURE C-2

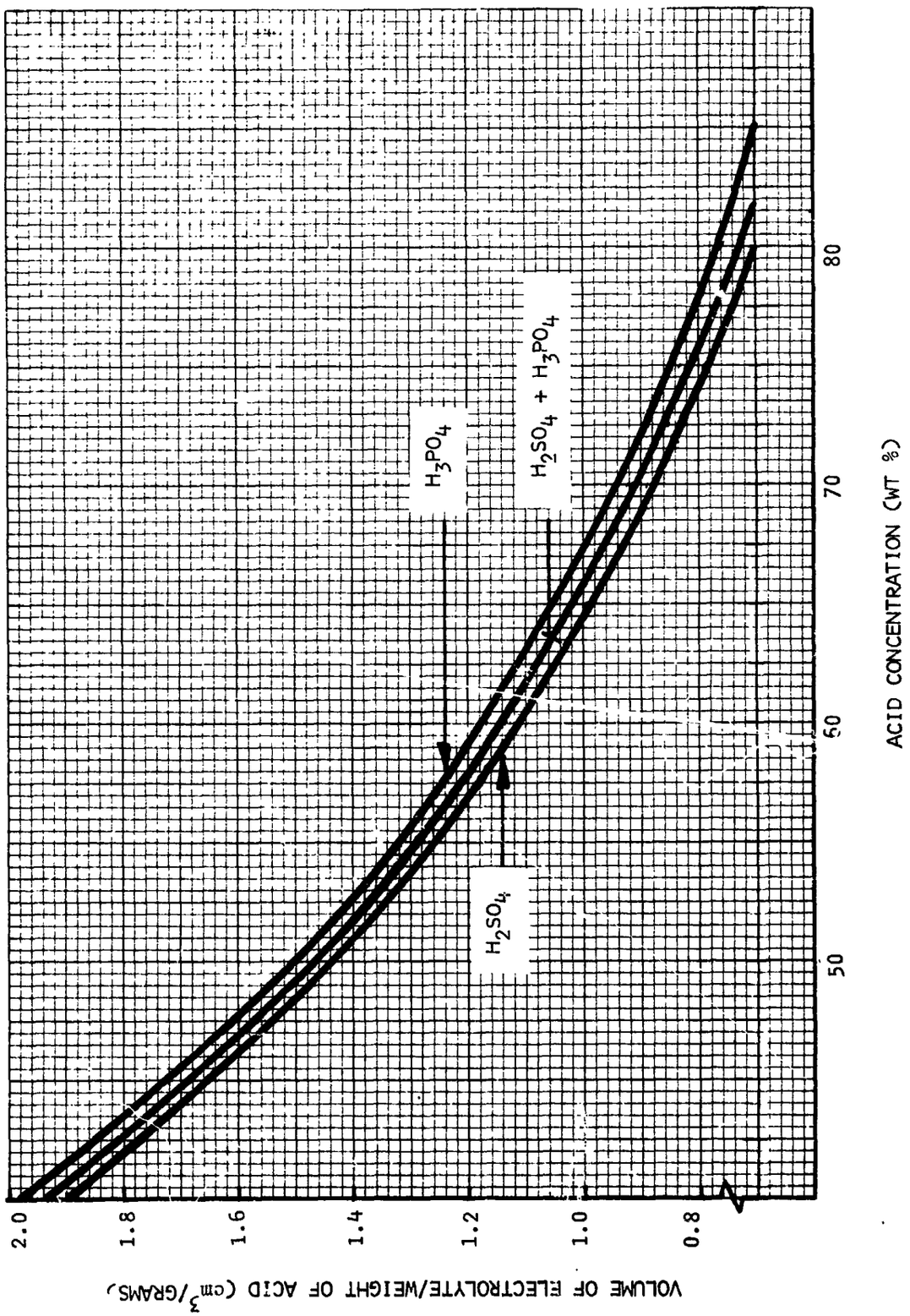

H_3PO_4
$H_2SO_4 + H_3PO_4$
H_2SO_4
2.0
1.8
1.6
1.4
1.2
1.0
0.8
50
60
70
80
ACID CONCENTRATION (WT %)
VOLUME OF ELECTROLYTE/WEIGHT OF ACID (cm^3/GRAMS)

FIGURE C-3

APPENDIX D

TEST PLAN FOR DETERMINATION OF POLARIZATION CURVES FOR VARIOUS ELECTROLYSIS TEST CELLS

Hamilton Standard DIVISION OF UNITED AIRCRAFT CORPORATION

SVHSER 5901
CR-115070

HS F-927 6/66

TEST NO. [illegible]

HAMILTON STANDARD

PLAN OF TEST

PAGE 1 OF [illegible]

JOB: Determine Polarization Curves for Various Electrolysis Test Cells

PREPARED BY: [illegible]

PROJECT & ORDER: B62-100-000A

APPROVED BY: [illegible]

INSTRUCTION:

TEST ENGINEER: [illegible]

TIME PERIOD: TO

(2) - Rev A - 7/20/70 [illegible].

1. WHAT IS ITEM BEING TESTED?

2. WHY IS TEST BEING RUN? WHAT WILL RESULTS SHOW OR BE USED FOR?

3. DESCRIBE TEST SET UP INCLUDING INSTRUMENTATION. ATTACH SKETCH OF INSTALLATION.

4. ITEMIZE RUNS TO BE MADE GIVING LENGTH OF EACH AND READINGS TO BE TAKEN.

5. SPECIAL INSTRUCTIONS: SAFETY PRECAUTIONS FOR OPERATORS AND HANDLING EQUIPMENT. OBSERVATIONS BY SIGHT, FEEL, OR HEARING. LIST POINTS OF OBSERVATION WHICH MIGHT CONTRIBUTE TO ANALYSIS OF (A) PERFORMANCE OF UNITS, (B) INCIPIENT TROUBLE BEFORE IT OCCURS, AND (C) CAUSE OF FAILURE.

6. HOW WILL DATA BE USED OR FINALLY PRESENTED? GIVE SAMPLE PLOT, CURVE, OR TABULATION AS IT WILL BE FINALLY PRESENTED.

NUMBER ENTRY AS LISTED ABOVE AND DESCRIBE BELOW

1. Item Being Tested - Feasibility electrolysis cells having one (1) square inch area, an anode of platinized platinum screen, a cathode AA2 screen and the following electrolyte configurations: (Reference figure I)

   a. H_2SO_4 (at 56% concentration by weight) with cabo-sil mixture by weight of 10 parts H_2SO_4 and 1 part cabo-sil

   b. H_2SO_4 (at 56% concentration by weight) with tissuquartz

   c. H_3PO_4 (at 85% concentration by weight) with asbestos

   d. H_3PO_4 (at 85% concentration by weight) with tissuquartz

   (2) e. H_2SO_4 + H_3PO_4 with tissuquartz and fritted glass reservoir.

2. Reason for Test - To determine the E_{IR} (ohmic polarization) drop and cell current density vs. voltage (polarization curves) of each cell configuratio at various levels of water in the electrolyte (provided by changing the ambient dew point). This cell evaluation is part of Phase I test effort under NASA contract "Humidity Control and Oxygen Supply System Study" (# NAS-9-10773).

2.1 Use of Test Results - The test results will be used to support the analytic model of the various cell designs and to aide in the selection of final cel configuration for the trade study.

3. Test Set-Up - Reference Figures II & III for test set-up.

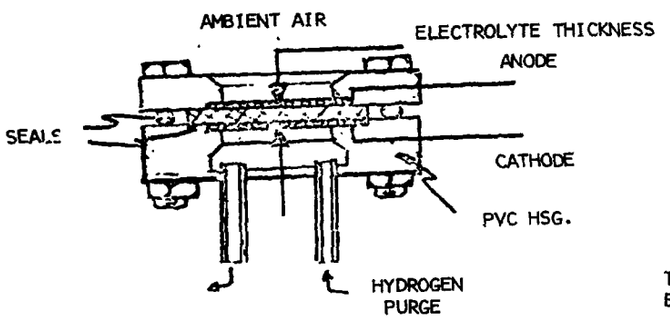
4. Tests

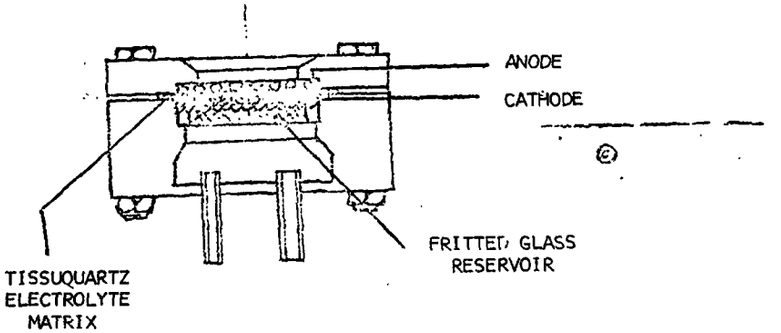
a. - Determine E_{IR} drop on each cell. Photograph voltage and current pulses and determine the resistance. (Approximately 100 micro second pulses).

b. - Trace the voltage vs. current density characteristics as the voltage is increased at a constant rate from zero to approximately 2.5 volts and back to zero. (Voltage change at 1,000 mil volts/min.)

c. - Perform the above tests at chamber dew points of (°F) 70; 50; 40; 30; 20 or until the cell dries out. Each chamber setting will be held until the electrolyte has reached equilibrium. (Approximately 8 hours)

d. - Perform a electrochemical kinetic study of H_2SO_4, H_3PO_4 and H_2SO_4/H_3PO_4 at various concentrations, using the "free" electrolyte test cell (Figure IV).



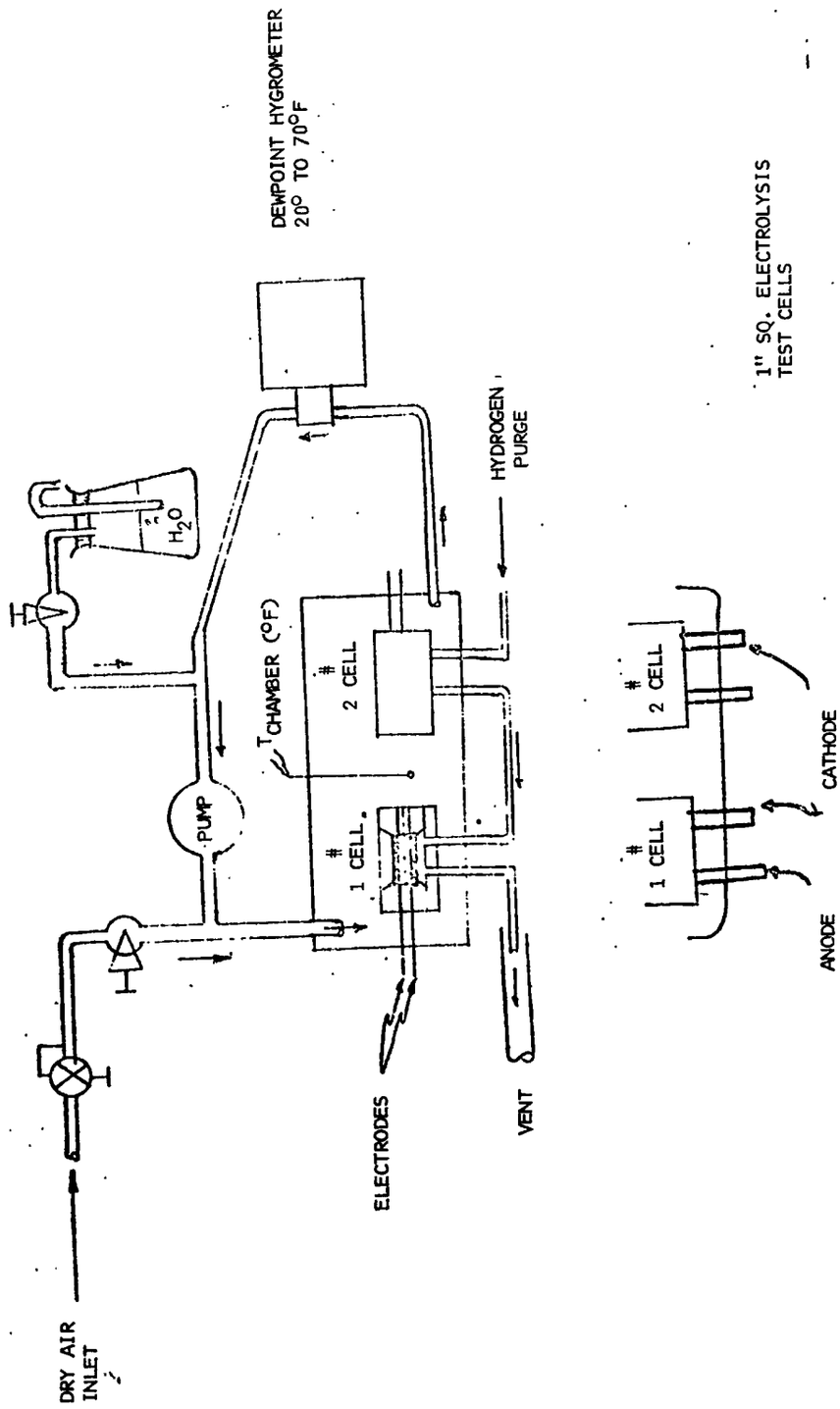


ELECTROLYTE	MATRIX THICKNESS	REMARKS
H_2SO_4/CAB-O-SIL	.074"	ELECTROLYTE HAS A THIN MEMBRANE OF SYNPOR IN CENTER.
H_2SO_4/TISSUQUARTZ	.034"	SATURATED (.14 GRAMS OF TISSUQUARTZ & 0.9 CC OF H_2SO_4)
H_3PO_4/ASBESTOS	.070	.3277 CC OF ACID ADDED (≈ 50% OF VOLUME)
H_3PO_4/TISSUQUARTZ	.034	SATURATED (.12 GRAMS OF TISSUQUARTZ & .8 CC OF H_3PO_4)
H_2SO_4- H_3PO_4/TISSUQUARTZ & FRITTED GLASS RESERVOIR	.034	SATURATED (.56 CC OF ACID MIXTURE)

CELL CONFIGURATIONS

FIGURE I

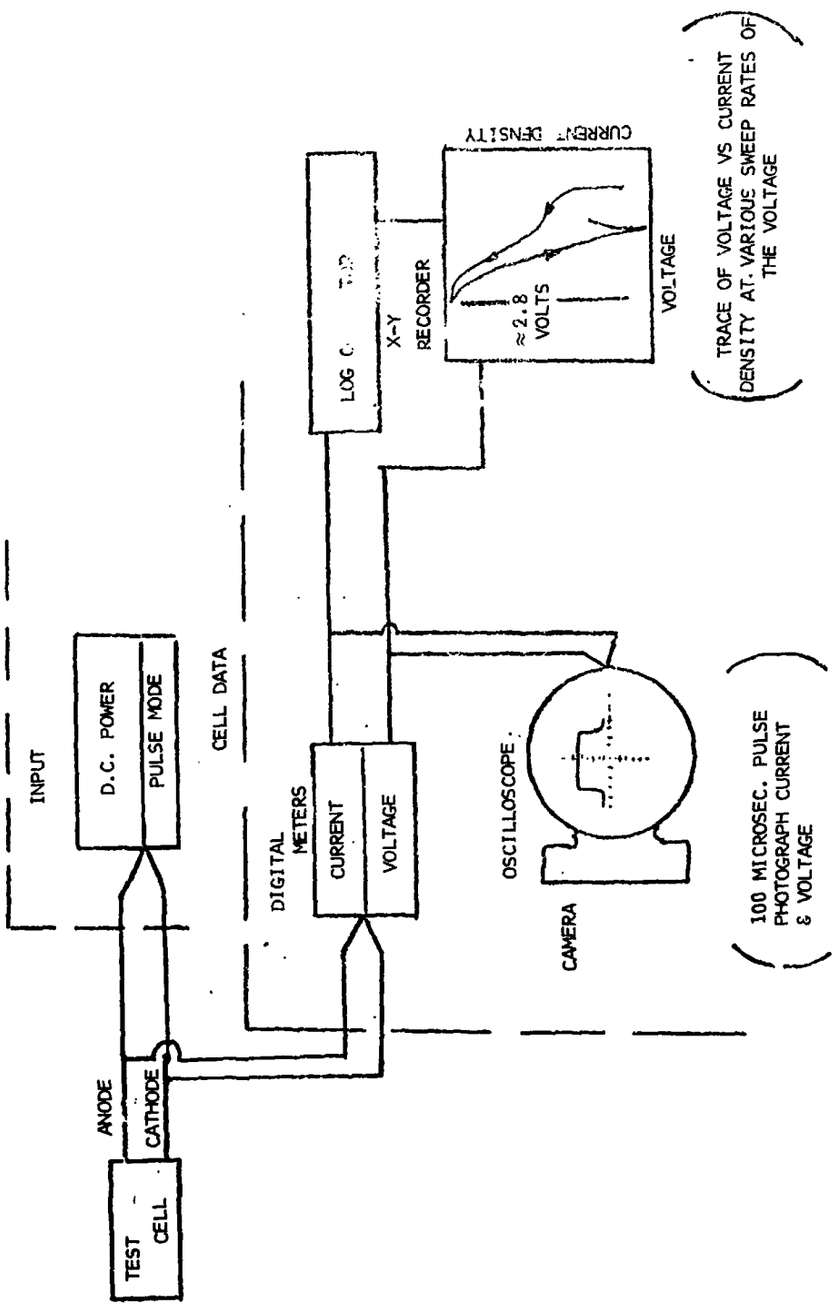
Page 40(C)



1" SQ. ELECTROLYSIS TEST CELLS

CURRENT DENSITY VS VOLTAGE TEST SET-UP

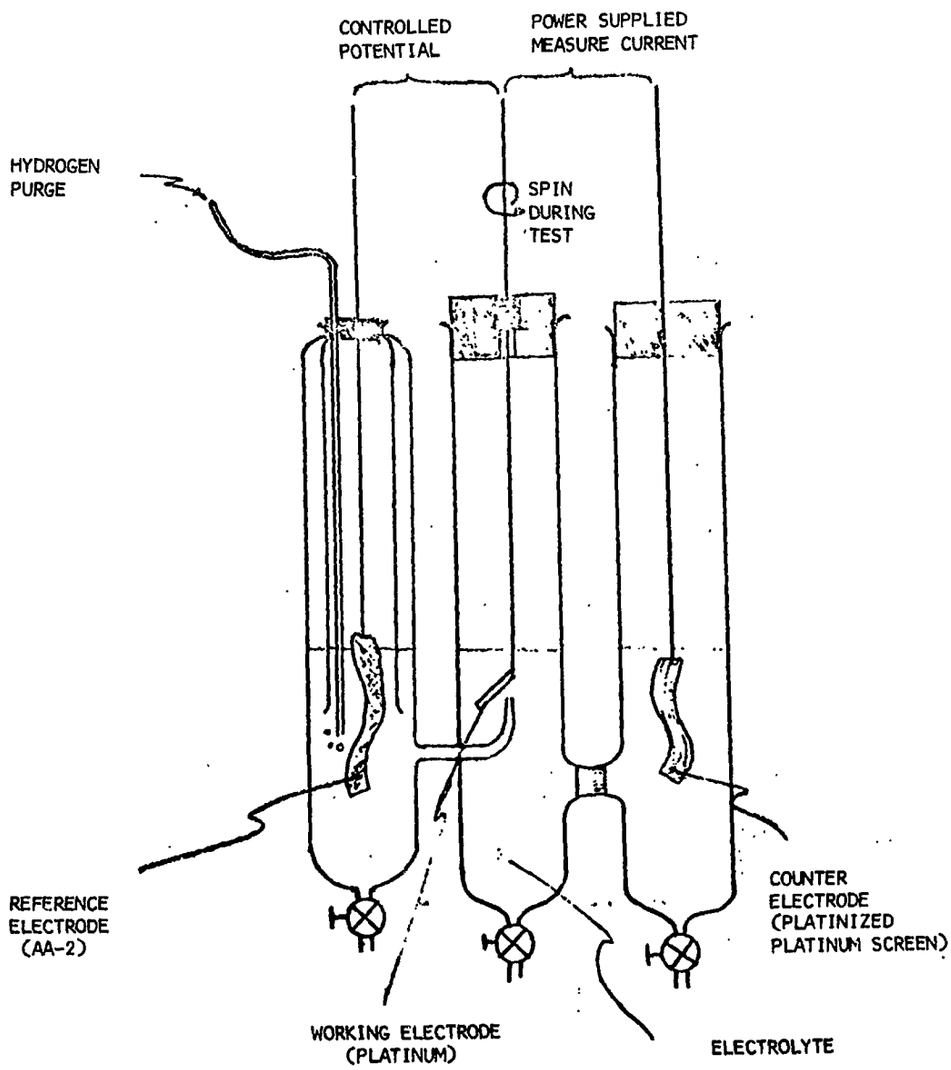
FIGURE II



TEST INSTRUMENTATION

FIGURE III

Page 6 of 6



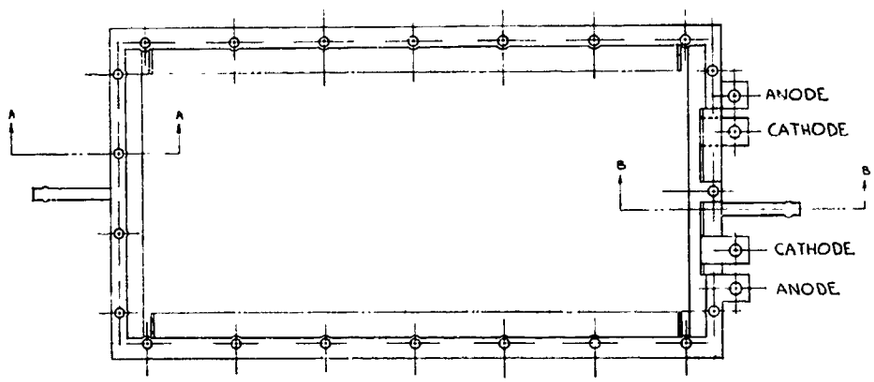
FREE ELECTROLYTE TEST CELL

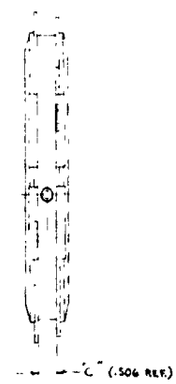
FIGURE IV

APPENDIX E

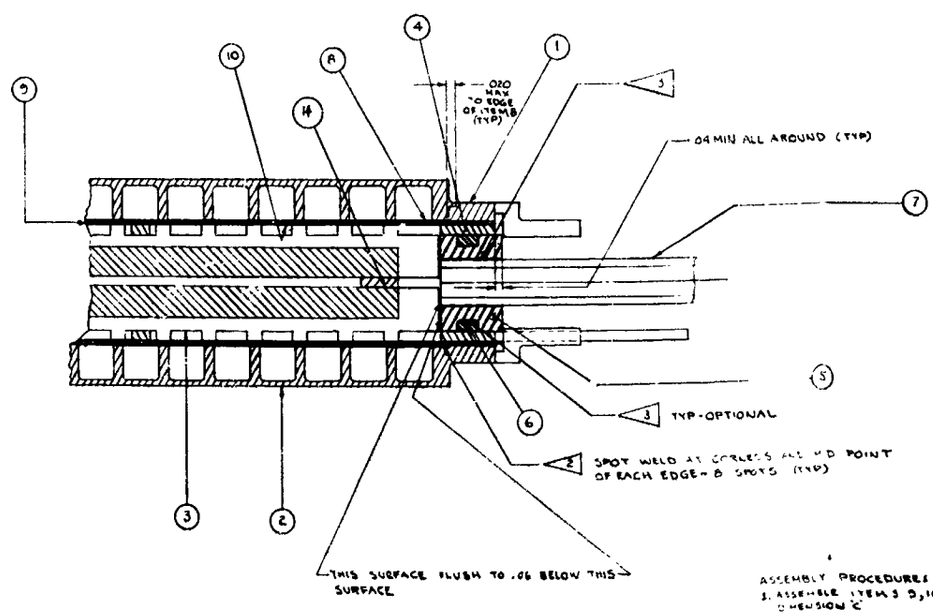
SVSK 81296, WATER VAPOR ELECTROLYSIS CELL PAIR ASSEMBLY

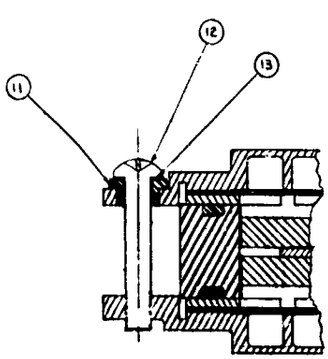
FOLDOUT FRAME 1





SECTION B-B
SCALE 4:1



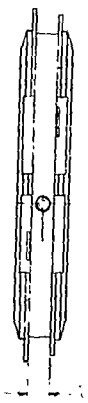


SECTION A-A
SCALE 4:1

ASSEMBLY PROCEDURES
1. ASSEMBLE (ITEMS 9,10,3,4,5 BETWE
DIMENSION 'C'
2. DISASSEMBLE AND REASSEMBLE WITH
3. TORQUE SCREWS (ITEM 12) EVENLY, SU
THAN THE RECORDED DIMENSION 'C'

FOLDOUT FRAME 2

REVISIONS							
GRAM NO	REV LTR	ZONE	LTR	DESCRIPTION	DATE	APPROVED	
			A	[illegible]	[illegible]		



SGG REF)

.04 MIN ALL AROUND TYP)

(1)

(5)

OPTIONAL

T CORNERS AND MI. POINT
C = 8 SPOTS (TYP)

ASSEMBLY PROCEDURE:

1. ASSEMBLE ITEMS 5,10,3,4,5 BETWEEN HOUSINGS ITEMS 1 & 2 AND RECORD DIMENSION C.
2. DISASSEMBLE AND REASSEMBLE WITH CHARGED ELECTROLYTE MATRIX.
3. TORQUE SCREWS (ITEM 12) EVENLY SUCH THAT DIMENSION C IS .010 LESS THAN THE RECORDED DIMENSION C IN STEP 1 ABOVE.

6. PEWA ACID ELECTRODE P/N SKN 42501
(TANTALUM SCREEN SPRAYED BOTH SIDES WITH TEFLON CATALYST REF)
PEWA RESEARCH LABS
EAST HARTFORD, CONN.

5. TISSUQUARTZ # 1700
PALLFLEX PRODUCTS CORP
PUTNAM, CONN.

4. PLATINUM-RHODIUM SCREEN (5% RHODIUM) 80 MESH-.003 WIRE DIA.
(PLATINUM COATED BY HSD REF)
ENGELHARD INDUSTRIES NEWARK, N.J.

3. BOND PER SVHS 3012 CURE 2 HRS AT 1,000°F, ETCH ITEMS 5/7 PER HS 3672.

2. SPOT WELD PER MIL W-6858

1. TRACEABILITY NOT REQ'D.

NOTES:

14	1	SPACER, RESERVOIR	SVSK 77298					
13	21	WASHER	NAS 620C6L					
12	21	SCREW	HSS1357-33					
11	21	BUSHING, SHOULDERED	SVSK81294-2					
10	2	CATHODE ELECTRODE	SVSK 77300					
9	2	ELECTROLYTE MATRIX	SVSK 8126 A6					CUT TO FIT IN SPACE AS SHOWN
8	2	ANODE SCREEN	SVSK81254-A6					CUT TO FIT IN SPACE AS SHOWN.
7	2	TUBE	SVSK81254-1					
6	2	PACKING, PERFORMED	SVSK81217					
								SVSK 81296
5	1	SPACER	SVSK81276					A
4	2	CATHODE	SVSK81273					
3	2	RESERVOIR ELECTROLYTE	SVSK 81272					
2	1	HOUSING, LOWER	SVSK81275					
1	1	HOUSING, UPPER	SVSK 81274					
PIECE NO.	NO. REQ'D	PART NAME	PART NUMBER	MATERIAL AND HEAT TREAT	HT. TREAT	NDT TEST		REMARKS

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ± ANGLES ± EXCEPT FOR SURFACES MARKED
& MARK PART CENTERLINES IN PLB MODES
CLEANING, PRESERVATION AND MARKING PER MIL-STD-1568 E.P.
SURFACE CHARACTERISTICS PER MIL-STD-1310 OR MIL-M-6
HEAT ARRY USED ON APPLICATION

MATERIAL
MACHINING
HEAT TREAT
EMC
SURFACE CONTROL
MIL-STD-1310
PAINT CODE
REQ SPECIFICATION
ALL AREAS

SAFETY CHECK

Hamilton Standard

WATER VAPOR ELECTROLYSIS CELL PAIR ASSY

SIZE A CODE IDENT NO. 73030 SVSK 81296

SCALE SHEET

APPENDIX F

WATER VAPOR ELECTROLYSIS CELL PAIR VERIFICATION TEST PLAN

Hamilton Standard DIVISION OF UNITED AIRCRAFT CORPORATION

SVHSER 5901
CR-115070

Revision A

TEST PLAN

PHASE II

WATER VAPOR ELECTROLYSIS CELL PAIR

VERIFICATION TEST

PREPARED UNDER CONTRACT NAS 9-10773

BY

HAMILTON STANDARD
DIVISION OF UNITED AIRCRAFT CORPORATION
WINDSOR LOCKS, CONNECTICUT

FOR

NATIONAL AERONAUTICS & SPACE ADMINISTRATION
MANNED SPACECRAFT CENTER
HOUSTON, TEXAS

I Test Item

NASA/MSC Water Vapor Electrolysis Cell Pair. Reference assembly drawing SVSK 81296

Cell Dimensions	Air Pass Length	-	5.3 inches
	Electrolysis Area	-	63.3 square inches/cell

II Purpose of Test

To demonstrate that the W.V.E. cell design which was established in Phase I (Ref. HSD interim report dated August 1970) will meet the performance requirements per Table I. The test results will be used for sizing W.V.E. units for specific crew/vehicle applications.

III Test Description

Testing will be done in three basic modes, initial check out, steady state and off design. The initial check out will determine cell resistance and voltage settings for the remaining tests; steady state testing will provide a performance map over the required operating range; and the off design testing will cover the 5 to 90% relative humidity conditions.

A. Cell Check Out

1. Cell Resistance: The IR drop of each cell will be determined by applying a high frequency A-C signal across the cell and measure the voltage.

2. Determine Operating Voltages: Operating voltage settings will be determined by obtaining a current-voltage curve of the cell. This will be made by operating the cells for 10 second intervals at various voltage settings from 1 to 2.2 volts and 53°F dew point.

B. Steady State Testing - This testing will consist of eight operating conditions, where the test unit will be equilibrated at the desired chamber dew point prior to powering the cells. The power will be applied to the cells for approximately 24 hours during which time the cell polarization, final cell equilibrating and steady state data will be obtained.

1. Test Conditions

	CHAMBER GAS *		CELL OPERATION	
Run No.	Dew Point (°F)	Dry Bulb Temp. (°F)	Voltage (Volts)**	Air Flow *** (CFM)
1	35	65	Min. (1.95)	15
2	35	65	Nom. (2.05)	15
3	35	65	Max. (2.20)	15

III B. 1. Test Conditions (Continued)

Run No.	CHAMBER GAS * Dew Point (°F)	Dry Bulb Temp. (°F)	CELL OPERATION Voltage (Volts)**	Air Flow*** (CFM)
4	53	65	Min.	15
5	53	65	Nom.	15
6	53	65	Max.	15
7	64	65	Min.	15
8	64	65	Nom.	15
9	64	65	Max.	15

A

*** The fan which provides this air flow will be precalibrated such that the air flow will be determined by reading fan power and ΔP.

** The min., nom. and max. cell voltage values will be determined from initial cell check out, values listed are reference only and correspond to the values of Figure 13 of Phase I interim report.

* These three chamber conditions correspond to the minimum (35% R.H. @ 63°F), nominal (50% R.H. @ 73°F) and maximum (65% R.H. @ 77°F) cabin conditions for which the W.V.E. unit must maintain cabin O_2 partial pressure.

2. Test Data - Reference Table II (O_2 production will be calculated from current, 1 Amp = 3.46 cc/min). Data points will be taken every 10 min. during the first 2 hrs. of each run and every 1/2 hr. thereafter. A
3. Test Schematic - Reference Figure 1
4. Instrumentation - Reference Table III

C. Off-Design - Operate the cells at nominal voltage (2.05 volts) with a chamber dew point of 64°F and 65°F dry bulb until steady state is obtained. Then drop the dew point in 10° steps (over an 8 hour period) and operate the unit for approximately 8 hours at each new level, until the cells indicate a drying out condition. After the unit has indicated drying out, increase the dew point in 10° steps up to 74°F and observe the performance at each change for 8 hours.

IV Special Instructions

1. Maintain chamber pressure between 0.3 ± .1 inches of H_2O above ambient.

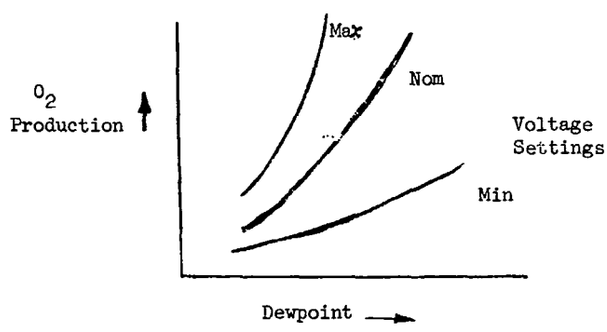
2. Maintain chamber O_2 % at 20 ± 2%.

3. When unit is shutdown, disconnect power leads from cells.

4. At dew points above 50°F observe cells for foaming or splattering of electrolyte.

V Data Presentation

The performance map data will be presented by the following sample curve: A



The off-design drying and flooding data will be presented as follows:

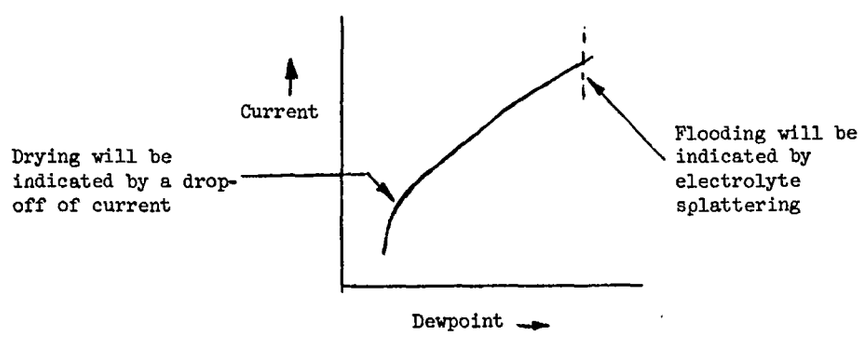


TABLE I

W.V.E. PERFORMANCE REQUIREMENTS

I The Phase II verification test W.V.E. unit will meet the following performance requirements when operated in the defined ambient:

(a) Ambient Conditions

Pressure	14.7 to 16 psia
Temperature	63 to 77°F
Relative Humidity	35 to 65%
% Oxygen	Approx. 20%
Air Flow Through Cell Pair	- 15 cfm

(b) Performance

Oxygen Production	- 1.18 to 3.125 lb/man/day (equivalent cell pair production = 57.8 to 153.5 cc/min)
Hydrogen Production	- 0.14 to 0.39 lb/man/day (equivalent cell pair production 115.6 to 307 cc/min)
Cell Pressure Drop	- To be determined (Approx. 2" H_2O expected)
Operating Voltage	- Not to exceed 2.20 volts

II The Phase II unit must also be capable of operating at off design conditions in an ambient as low as 5% relative humidity and as high as 90% R. H. The cell should not dry out or flood over this range of humidity and should not cause degradation in performance when operated at design conditions. A

TABLE II

W.V.E. Cell Pair Test Data Points

Tc_1	Inlet Air Temperature to Cell Pair
Tc_2	Outlet Air Temperature from Cell Pair
P_1	Chamber Pressure
P_2	H_2O Tank Pressure to Steam Generator
P_3	Steam Pressure
ΔP_1	Δ P Across Fan on Outlet of Cell Pair
W_1	H_2 Production Rate
W_2	N_2 Make-Up Gas Flow Rate
Dew Point	Record Dew Point °F
$Current_1$	Cell Pair Current Record
$Voltage_1$	Cell Pair Voltage Record (constant for a run)
$Voltage_2$	Fan Voltage
$Current_2$	Fan Current
ΔW_1	Change in Weight of Cell Pair
O_2	% Oxygen in Chamber Air (maintain 18-22%)
Chamber Gas Checks	Check Chamber Gas for Combustible Gas and Ozone
T	Test Time
H_2 Exhaust Gas	Check H_2 Exhaust Gas for Presence of H_2S and Ozone

TABLE III

INSTRUMENTATION

ITEM	RANGE/READ OUT
Oxygen Analyzer (Beckman)	0 to 100% (2% inc.)
Combustable Gas Dector (J-W)	0 to 1% (0.02% inc.)
Digital Voltmeter (United Sys. Corp.)	0 to 10 (.002 volts inc.)
Digital Amp Meter (United Sys. Corp.)	0 to 100 (.02 Amps inc.)
N_2 Flowmeter	
H_2 Flowmeter	
Dew Pointer (Cambridge 880)	-40° to 120°F (2°F inc.)
Temperature Recorder (Bristoc's)	0 to 250°F (1.0°F inc.)
Sandborne Recorder	N/A
H_2S Detector	Color change in sample tube
Scale	0 to 20 lbs (0.01 lbs inc.)
Slant Manometer (Merian Instr.)	0 to 4" H_2O (.01" inc.)
Slant Manometer	0 to 2" H_2O (.01 inc.)

APPENDIX G

RESERVOIR CHARACTERISTICS

Hamilton Standard — Division of United Aircraft Corporation

I. Reservoir Capacity

Reservoir	I Dry Weight (grams)	II Weight at Disassy. (grams)	III Weight After Disassy. When Saturated With H_2O (grams)	IV H_2O Saturated Weight (grams)
Top Unit	464.0	535.3	568.8	545.2
Bottom Unit	481.0	518.6	570.4	562.4

NOTES:

I. Dry weight of reservoir before Assembly No. 3.

II. Weight of reservoir as it was removed from cell at post test inspection.

III. Weight of reservoir as in II but after water had been added to fully saturate it.

IV. Weight of reservoir saturated only with H_2O.

Percent Removed From Reservoir - When the cell flooded during runs 7 and 8 it is assumed that the reservoirs were saturated and as the cell dried down electrolyte was removed from the reservoir. The following is a calculation of the percent of electrolyte which went back into the matrix during the drying phase.

$$\frac{\text{Weight III - Weight II} = H_2O \text{ Added}}{\text{Weight IV - Weight I} = \text{Total } H_2O \text{ Capacity}} = \%\ \text{Removed}$$

Top Reservoir $\frac{568.8 - 535.3}{545.2 - 464.0} = 41\%$ Removed

Bottom Reservoir $\frac{570.4 - 518.6}{562.4 - 481.0} = 64\%$ Removed

The theoretical delta in electrolyte weight/cell between a 65°F/32°F/DP and 65°F/57°F DP is approximately 40.5 grams. This would represent a percent removed of $\frac{40.5}{(562.4-481.0)\ \text{Total } H_2O \text{ Capacity} \times 1.43\ (\text{acid density})} = 35\%$.

I. (Continued)

The top reservoir percentage corresponds very well with the theoretical value, however, the bottom reservoir indicated a much larger percent removed. This means that the bottom reservoir was not saturated at the cell pair flooding condition of 65°F/56.5°F DP. From the examination of the anodes during post test inspection, it could be assumed that the excess electrolyte at flooding was trapped on the air side of the anode and could not be wicked back into the matrix and reservoir.

II. Reservoir/Matrix Interface Characteristics

A series of tests was conducted to determine if the electrolyte would be wicked between the reservoir and matrix against a one g force.

Test (A) The reservoir was saturated with water and a cell matrix (two sheets of Tissuquartz) was placed on top. Within five minutes this matrix was saturated and was replaced with a second matrix. The second matrix also became saturated after two hours and was replaced with a third matrix. This matrix did not become saturated and could not remove all the remaining water in the reservoir. Only 85% of the water was removed from the reservoir by the three series of matrices. Figure 1 presents a plot of percent water removed versus time for the above test.

Test (B) Half a cell pair unit was assembled and positioned with the matrix below the reservoir (simulation of the bottom cell). The reservoir was dry at assembly and the matrix was saturated with water. During the test the matrix was kept

II. (Continued)

saturated as the reservoir continued to wick water away from it. The reservoir became saturated within four hours (reference Figure 1).

From the above tests and Figure 1 a reservoir capacity factor of 80% was selected as the recommended value for future reservoir sizing.

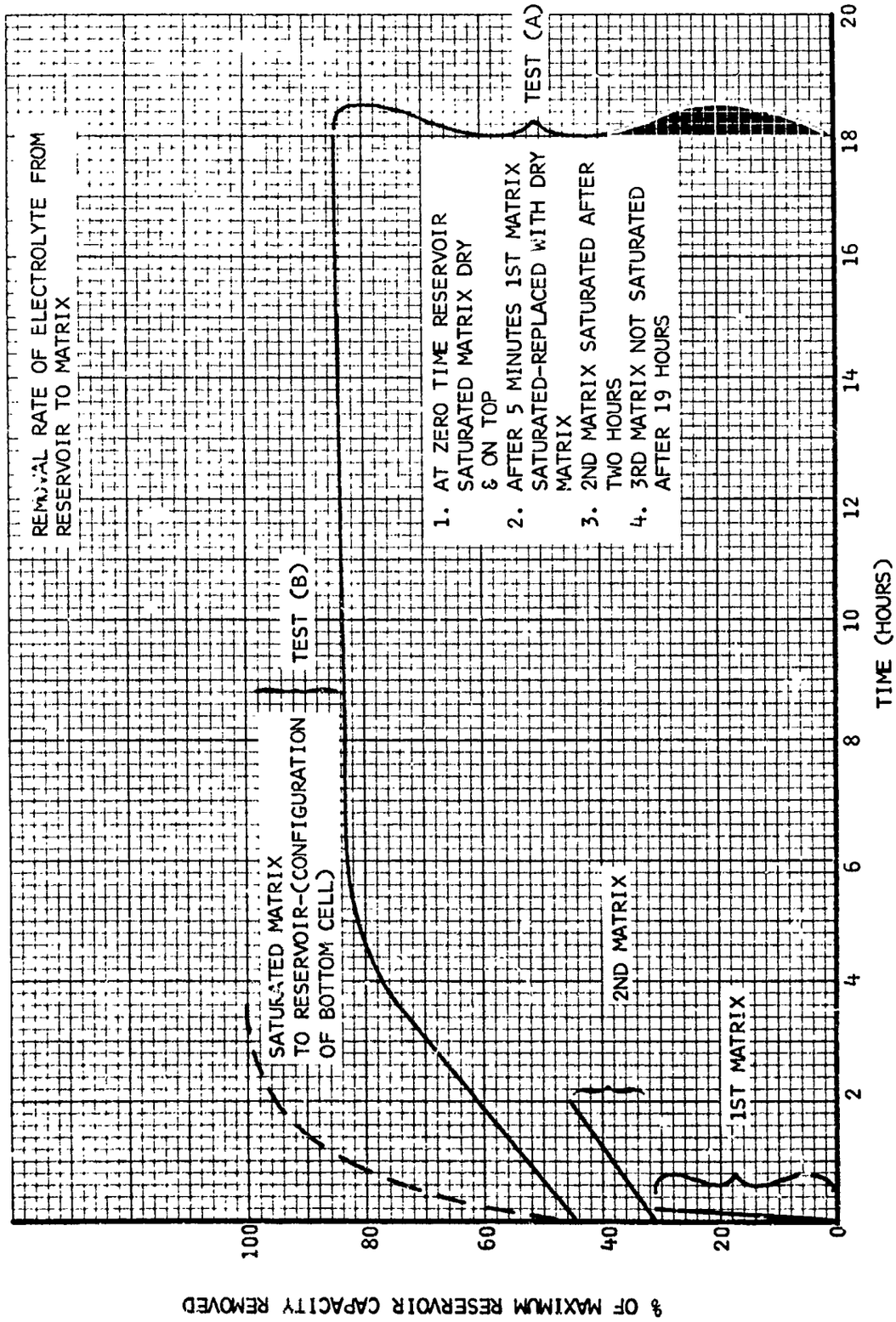

REMOVAL RATE OF ELECTROLYTE FROM RESERVOIR TO MATRIX
1. AT ZERO TIME RESERVOIR SATURATED MATRIX DRY & ON TOP
2. AFTER 5 MINUTES 1ST MATRIX SATURATED-REPLACED WITH DRY MATRIX
3. 2ND MATRIX SATURATED AFTER TWO HOURS
4. 3RD MATRIX NOT SATURATED AFTER 19 HOURS
TEST (A)
TEST (B)
SATURATED MATRIX TO RESERVOIR-(CONFIGURATION OF BOTTOM CELL)
2ND MATRIX
1ST MATRIX
TIME (HOURS)
% OF MAXIMUM RESERVOIR CAPACITY REMOVED
0
2
4
6
8
10
12
14
16
18
20
20
40
60
80
100

FIGURE 1

APPENDIX H

TEST PLAN TO DETERMINE WICKING CHARACTERISTICS OF VARIOUS MATERIALS

Hamilton Standard DIVISION OF UNITED AIRCRAFT CORPORATION

SVHSER 5901
CR-115070

HS F-927 6/56

TEST NO. _____

HAMILTON STANDARD

PAGE 1 OF 1

PLAN OF TEST

JOB: Wicking Characteristics of Various Materials

PLAN PREPARED BY: J Hoodleston

PROJECT & ORDER: NASA Contract - NAS 9-10773

APPROVED BY: [illegible]

INSTRUCTION: _____

TEST ENGINEER: _____

TIME PERIOD: _____ TO _____

1. WHAT IS ITEM BEING TESTED?

2. WHY IS TEST BEING RUN? WHAT WILL RESULTS SHOW OR BE USED FOR?

3. DESCRIBE TEST SET UP INCLUDING INSTRUMENTATION. ATTACH SKETCH OF INSTALLATION.

4. ITEMIZE RUNS TO BE MADE GIVING LENGTH OF EACH AND READINGS TO BE TAKEN.

5. SPECIAL INSTRUCTIONS: SAFETY PRECAUTIONS FOR OPERATORS AND HANDLING EQUIPMENT. OBSERVATIONS BY SIGHT, FEEL, OR HEARING. LIST POINTS OF OBSERVATION WHICH MIGHT CONTRIBUTE TO ANALYSIS OF (A) PERFORMANCE OF UNITS, (B) INCIPIENT TROUBLE BEFORE IT OCCURS, AND (C) CAUSE OF FAILURE.

6. HOW WILL DATA BE USED OR FINALLY PRESENTED? GIVE SAMPLE PLOT, CURVE, OR TABULATION AS IT WILL BE FINALLY PRESENTED.

NUMBER ENTRY AS LISTED ABOVE AND DESCRIBE BELOW

1. TEST ITEM: Materials of a W.V.E. cell which involve the wicking of the electrolyte:

Material	Pore Size
Tissuquartz	1-6 microns
Fritted Glass	30-40 microns
Porous Ceramic	31-40 microns
Microporous Rubber	-

2. PURPOSE OF TEST: To determine the wicking characteristics of various materials. This will enable material substitution in W.V.E. unit without affecting the flow of electrolyte in and out of the cell reservoir.

3. TEST PROCEDURE: Test specimen will be cut in 1/2 x 1 inch strips and its dry weight recorded. The specimen will be held vertically with the bottom edge in contact with distilled water. The height and weight of water which the material has wicked will be recorded. This will indicate the wicking strength of the material in inches of H_2O. The % absorption will be obtained by saturating the specimen with distilled water and determining the following:

$$\frac{\text{saturated wt.} - \text{dry wt.}}{\text{dry wt.}} \times 100$$

APPENDIX J

TEST PLAN TO INVESTIGATE THE PRODUCTION OF H_2S

Hamilton Standard DIVISION OF UNITED AIRCRAFT CORPORATION

SVHSER 5901
CR-115070

ECS-2067-L-001

HS F-927 6/56

TEST NO. _____

HAMILTON STANDARD

PLAN OF TEST

PAGE 1 OF _____

JOB: H_2S Investigation

PLAN PREPARED BY: J. Huddleston

PROJECT & ORDER: NASA Contract No. NAS 9-10773

APPROVED BY: _____

INSTRUCTION: _____

TEST ENGINEER: J. Huddleston 10/24/70

TIME PERIOD: _____ TO _____

1. WHAT IS ITEM BEING TESTED?

2. WHY IS TEST BEING RUN? WHAT WILL RESULTS SHOW OR BE USED FOR?

3. DESCRIBE TEST SET UP INCLUDING INSTRUMENTATION. ATTACH SKETCH OF INSTALLATION.

4. ITEMIZE RUNS TO BE MADE GIVING LENGTH OF EACH AND READINGS TO BE TAKEN.

5. SPECIAL INSTRUCTIONS: SAFETY PRECAUTIONS FOR OPERATORS AND HANDLING EQUIPMENT. OBSERVATIONS BY SIGHT, FEEL, OR HEARING. LIST POINTS OF OBSERVATION WHICH MIGHT CONTRIBUTE TO ANALYSIS OF (A) PERFORMANCE OF UNITS, (B) INCIPIENT TROUBLE BEFORE IT OCCURS, AND (C) CAUSE OF FAILURE.

6. HOW WILL DATA BE USED OR FINALLY PRESENTED? GIVE SAMPLE PLOT, CURVE, OR TABULATION AS IT WILL BE FINALLY PRESENTED.

NUMBER ENTRY AS LISTED ABOVE AND DESCRIBE BELOW

1. Test Item: H_2SO_4 and a mixture of H_2SO_4 with H_2PO_4 - The Electrolyte of an Electrolysis Cell.

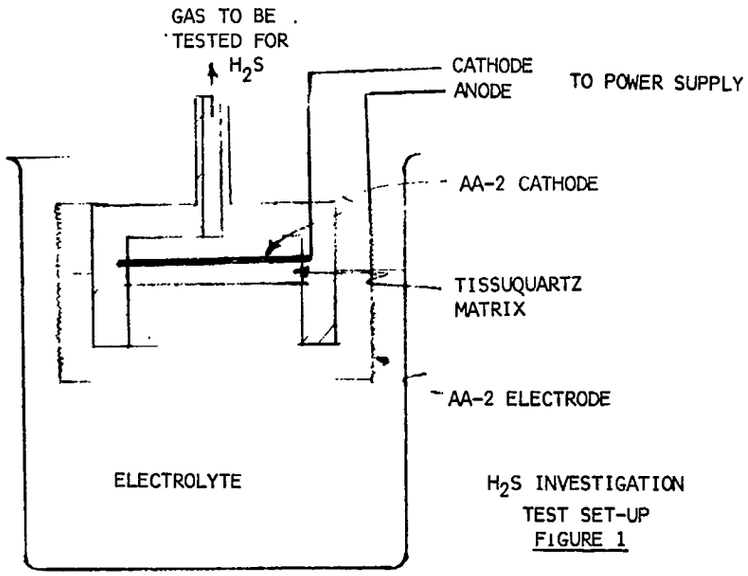
2. Purpose of Test & Use of Test Data: This test is being conducted to determine if H_2S is formed and if the cathode is poisoned when the above acids are used as the electrolyte of an electrolysis cell. Test data will determine if H_2SO_4 or the mixed acid will be used as the electrolyte in the Phase II test cell.

3. Test Setup: 1) A small electrolysis cell (reference Figure 1) will be operated and the gas produced at the cathode will be collected and passed over lead acitate paper to determine if H_2S has been produced.

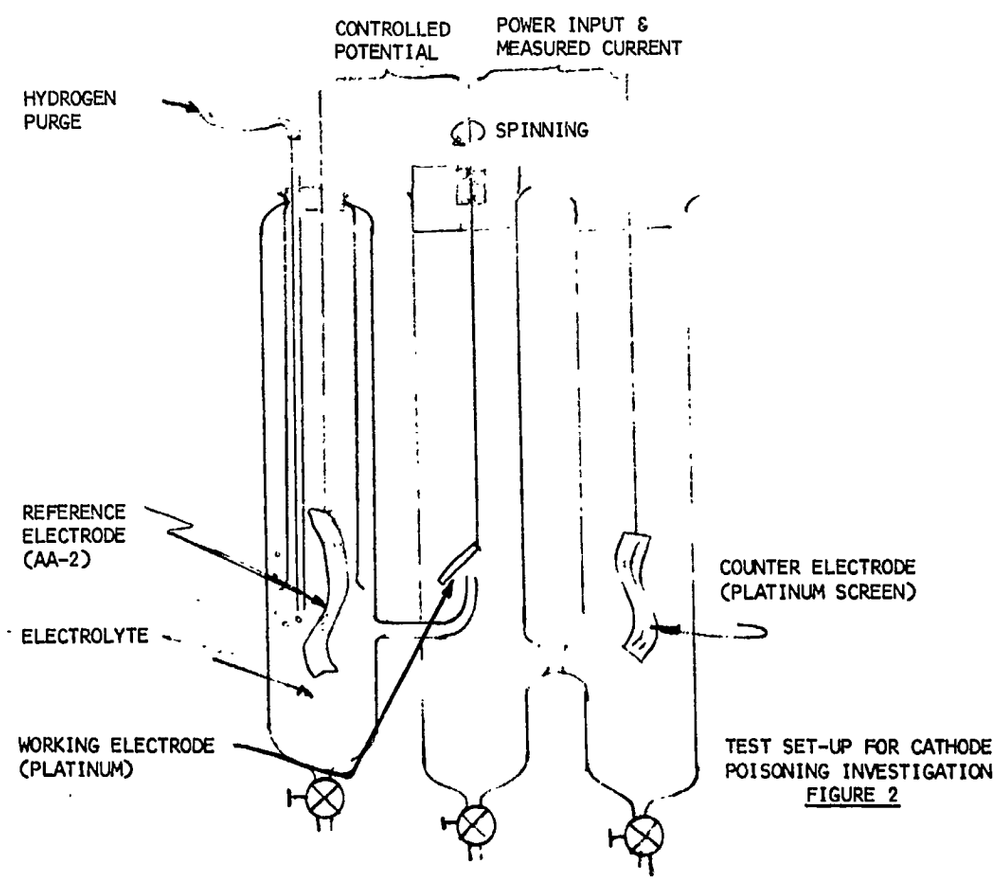
2) To determine the degree of cathode poisoning due to SO_4 reduction to sulfur or sulfide, a "half cell" (reference Figure 2) will be operated. The cathode poisoning will be determined by measuring the current decay with time at constant potential and with hydrogen adsorption checks.

4. Itemized Runs: Cell will be operated at current densities up to 100 amps/sq. ft. & at voltages between 1.95 and 2.3 volts. Specific current and voltage settings will be determined during the test.

ECS-2067-L-00



H_2S INVESTIGATION
TEST SET-UP
FIGURE 1



TEST SET-UP FOR CATHODE POISONING INVESTIGATION
FIGURE 2