Hamilton DIVISION OF UNITED AIRCRAFT CORPORATION
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HYDROGEN DEPOLARIZED CARBON DIOXIDE CONCENTRATOR

PERFORMANCE IMPROVEMENTS AND CELL PAIR STRUCTURAL TESTS

by

J. C. HUDDLESTON AND DR. J. R. AYLWARD

PREPARED UNDER CONTRACT NO. NAS 9-12920

by

HAMILTON STANDARD

DIVISION OF UNITED AIRCRAFT CORPORATION

WINDSOR LOCKS, CONNECTICUT

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

JOHNSON SPACE CENTER

HOUSTON, TEXAS

SEPTEMBER 1973

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ABSTRACT

HYDROGEN DEPOLARIZED CARBON DIOXIDE CONCENTRATOR PERFORMANCE IMPROVEMENTS AND CELL PAIR STRUCTURAL TESTS

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J. C. HUDDLESTON and DR. J. R. AYLWARD

SEPTEMBER 1973

This report describes the investigations and testing associated with improving the CO_2 removal efficiency and voltage degradation of a Hydrogen Depolarized Carbon Dioxide Concentrator, and the vibration testing of a Water Vapor Electrolysis Cell Pair, under NASA Contract NAS 9-12920.

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FOREWORD

This report was prepared by the Hamilton Standard Division of the United Aircraft Corporation for the National Aeronautics and Space Administration's Johnson Space Center, in accordance with Contract NAS 9-12920. This report describes the work accomplished during the period of 21 June 1972 through 28 September 1973, in improving the performance of the hydrogen depolarized carbon dioxide concentrator cell pair. This development effort consisted of detailed cell analysis, cell and cell pair testing, and a structural vibration test of a water vapor electrolysis cell pair.

Personnel responsible for the conduct of this program were Mr. F. H. Greenwood, Program Manager; Mr. J. C. Huddleston, Program Engineer; Dr. J. R. Aylward, Technical Consultant from Hamilton Standard; and Mr. A. Behrend, Technical Monitor and Mr. R. J. Gillen, Overall Program Supervisor for the NASA Johnson Space Center.

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ABBREVIATIONS AND SYMBOLS

A	Area
A-3R	Code Name for Experimental Electrode
A-10R	Code Name for Experimental Electrode
AA-2	Electrode Manufactured by American Cyanamid Company
AR	As Required
asf	Amps/Sq. Ft.
Avg.	Average
°C	Degrees Celsius (Centigrade)
cc	Cubic Centimeter
cfm	Cubic Feet per Minute
cm^2	Centimeter Squared
CO	Carbon Monoxide
CO_2	Carbon Dioxide
CO_3^{2}	Carbonate Ion
$Cs+$	Cesium Ion
Cs_2CO_3	Cesium Carbonate
$CsHCO_3$	Cesium Bicarbonate
Cs_2SO_4	Cesium Sulfate
d	Electrode Separation
d'	Uncompressed Matrix Thickness
d^o	Maximum Compressed Matrix Thickness
dc	Direct Current
DP	Dew Point

ABBREVIATIONS AND SYMBOLS (Continued)

DS-16	Code Name for Improved HDC Cathode
E	Cell Voltage (IR Free)
f	Matrix Factor
$°F$	Degrees Fahrenheit
Ft	Foot
H_2	Hydrogen
HCO_3^-	Bicarbonate Ion
HDC	Hydrogen Depolarized Carbon Dioxide Concentrator
Hsg	Housing
Hg	Mercury
hr.	Hour
H_2O	Water
H_2S	Hydrogen Sulfide
H_2SO_4	Sulfuric Acid
Hz	Hertz (cycles per sec.)
I	Current
i	Current Density
in.	Inches
IR	Voltage Drop
KCl	Potasium Chloride
lb.	Pound
mg/cm^3	Milligrams per Cubic Meter
min.	Minute

ABBREVIATIONS AND SYMBOLS (Continued)

ml	Milliliter
mm	Millimeter
mV	Millivolt
N_2	Nitrogen
N/A	Not Applicable
Nom.	Nominal
NH_3	Ammonia
N_2O	Nitrous Oxide
NO	Nitric Oxide
NO_2^-	Nitrite Ion
NO_3^-	Nitrate Ion
O_2	Oxygen
OH^-	Hydroxide Ion
P	Pressure
psi	Pounds per Square Inch
psia	Pounds per Square Inch Absolute
PH_2O	Partial Pressure of Water Vapor
PO_2	Partial Pressure of Oxygen
PCO_2	Partial Pressure of Carbon Dioxide
PPF	Code Name for Improved HDC Anode (Mfg. by P&WA)
PPM	Parts Per Million
PVC	Code Name for P&WA Experimental Electrode
P&WA	Pratt & Whitney Aircraft, Division of United Aircraft Corporation

ABBREVIATIONS AND SYMBOLS (Continued)

R	Resistance (ohms)
R.H.	Relative Humidity
SSP	Space Station Prototype
SEC	Code Name for Experimental Electrode
sec.	Second
SO_3^{-2}	Sulfite Ion
scc	Standard Cubic Centimeter
SO_2	Sulfur Dioxide
SCFM	Standard Cubic Feet per Minute
SEM	Scaning Electron Microscope
T_{in}	Inlet Temperature
TMAC	Tetramethylammonium Carbonate
TMA	Tetramethylammonium
UAC	United Aircraft Corporation
VDC	Volts, Direct Current
WVE	Water Vapor Electrolysis
wt.	Weight
wt. %	Weight Percent
μ	Micro (10^{-6})
%	Percent
$\approx$	Approximately Equals
=	Equals

ABBREVIATIONS AND SYMBOLS (Concluded)

<	Less Than
~	Approximate
Δ	Delta
ρ	Resistivity (ohm $^{-1}$ cm $^{-1}$)
τ	Tortuosity

DEFINITIONS

Anolyte	Electrolyte in the immediate vicinity of the anode.
Cell	Electrochemical cell consisting of an anode, matrix with electrolyte, and cathode.
Cell Pair	Two cell packages with back to back hydrogen electrodes which share a common hydrogen chamber, housing and reservoir assemblies.
Concentration Over-Voltage	Voltage loss due to concentration differences between electrodes.
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.
Electrokinetic Effects	General phenomena associated with interaction of current with charged interfaces, i.e., electroosmosis, electrophoresis, streaming potential, sedimentation potential.
Electroosmosis	Movement of a liquid through a porous solid via a voltage gradient.
Electrophoresis	Movement of a solid through a liquid via a voltage gradient.
Flooding	The condition of the cell when the electrolyte has absorbed an amount of water which results in an electrolyte volume exceeding the capacity of the cell matrix and electrodes.
H_2 Crossover	Occurs at dry out of the matrix and allows hydrogen and oxygen to pass through the matrix.
IR Check	Measurement of IR drop by current interruption.
IR Drop	Voltage loss due to pure ohmic resistance.
Potential Sweeps	A programmed potential change--usually a triangular wave.

DEFINITIONS (Concluded)

Reservoir

A porous material which absorbs the excess electrolyte during cell flooding and returns it to the matrix during drying conditions.

Spreading Coefficient

The force which determines the wicking rate of a liquid into a porous solid--related to the interfacial tensions of the systems.

Steady State Operation

The operating condition when the cell voltage and current do not change significantly with time.

Tafel Slope

The slope of the Tafel curve.

Zeta Potential

Potential across the mobile part of the electrical double layer.

SUMMARY

The objectives of this program, which were to reduce the voltage decay rate and improve the CO_2 transfer rate of the hydrogen depolarized CO_2 concentrator (HDC) cell pair (Figure 1), were accomplished. HDC endurance tests of two cell pairs verified that the improved electrodes reduced the voltage decay rate from approximately $200 \mu v/hr$ to less than $50 \mu v/hr$ and improved the CO_2 transfer efficiency from 60 percent to 86 percent for cesium carbonate (Cs_2CO_3) electrolyte and from 45 percent to 70 percent for tetramethylammonium carbonate (TMAC) electrolyte at a current density of 18 asf and a CO_2 partial pressure of 3 mm Hg. This was accomplished by a thorough analysis of cell and electrode performance behavior, effects of trace contaminants, matrix characteristics, and electrode structure examinations. Results of this effort revealed that the PPF electrode reduced the migration rate of the anode platinum catalyst and Teflon binder. Electrophoretic migration caused the anode to lose effective area. The DS 16-0 electrode used as a cathode increased the circulation rate of the catholyte, resulting in increased CO_2 removal.

An evaluation of various HDC matrix materials was made to determine the tortuosity, optimum compression for minimum IR drop, compression set characteristics, and the effect of thickness on the CO_2 removal rate and cell power.

Performance testing of the various HDC cell pairs with Cs_2CO_3 electrolyte provided sufficient parametric and endurance data to size the six-man Space Station Prototype (SSP) CO_2 removal system as having 36 HDC cell pairs, and to verify a life capability exceeding six months. Subsequent improvements in electrode structure, as noted above, increased cell life by a factor of four and allowed a reduction in the number of cell pairs to 20. Testing also demonstrated that TMAC is an acceptable HDC electrolyte for operating over the relative humidity range of 30 to 90 percent and over a temperature range of 50°F to 80°F.

The water vapor electrolysis (WVE) cell pair which was assembled and operated for 3940 hours under a preceding NASA contract (NAS 9-11830)[1] was successfully restarted, after three and one-half months of storage, was vibrated at Apollo launch levels, and was operated after vibration testing, providing a total running time of 5280 hours. The successful vibration test demonstrated that the Hamilton Standard electrochemical cell pair design, including an electrolyte reservoir, is structurally qualified for flight.

The integrated WVE/HDC breadboard unit designed under the previous NASA contract (NAS 9-11830) was fabricated and assembled during this program. The breadboard unit consisted of two HDC cell pairs and two WVE cell pairs integrated into a single package, which incorporated a blower, valving and instrumentation for operating the system. This unit was not tested, however, because the test requirement was deleted from the contract.
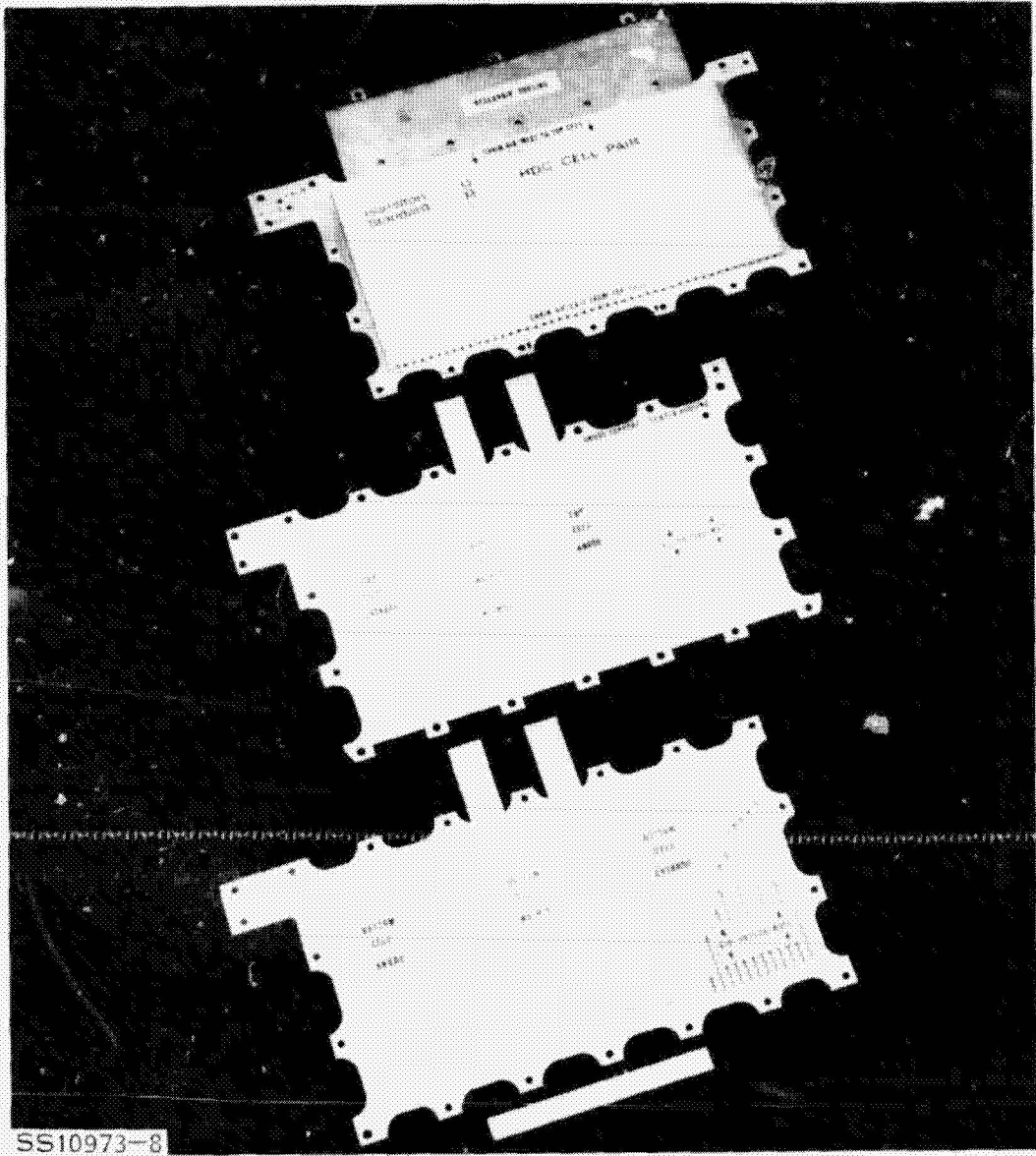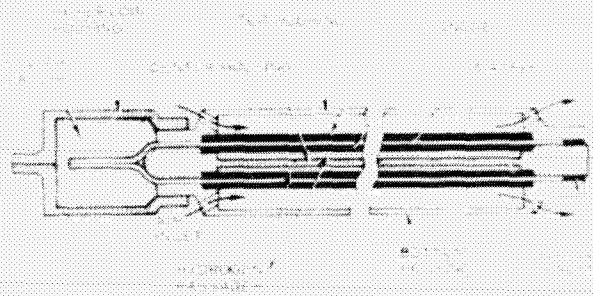
FIGURE 1.   WVE AND HDC CELL PAIRS

INTRODUCTION

This program was conducted in accordance with NASA Contract 9-12920 to improve the CO_2 removal rate and decrease the power decay of the HDC cell pair.

NASA Contract 9-12920 is a follow-on to NASA Contract NAS 9-11830 which resulted in the following accomplishments:

- Establishment of an HDC electrolyte (TMAC) for operation over a wide range in relative humidity.

- Initial testing of an integrated WVE/HDC.

- Design of a four cell pair integrated WVE/HDC breadboard unit.

The Improved Multi-Celled Breadboard Electrochemical Oxygen Generation and Carbon Dioxide Control System (NAS 9-12920) contract had as initial objectives:

- Investigation of various operating parameters of the HDC and WVE cell pairs.

- Fabrication and test of an integrated WVE/HDC breadboard unit.

The contract was revised twice to authorize the improvement of the HDC performance. As a result of the NASA's review of the HDC cell pair data, presented at the SSP Approval Design Review (ADR) meeting, this program was redirected to obtain additional HDC parametric test data and to demonstrate cell pair performance repeatability. This SSP oriented task established that:

- The required number of HDC cell pairs to maintain the P_{CO_2} level, for a six-man SSP cabin, below 3 mm Hg, would be 36.

- The degradation in cell voltage, which occurred over six months of testing, decreased with time and assured a six month cell life for a 36 cell pair unit.

- A CO_2 removal performance map was obtained for a range of P_{CO_2} from 1.2 to 3.0 mm Hg and current densities from 12 to 24 asf.

The evaluation of the HDC design and testing for this SSP oriented task are summarized in a separate report, SVHSER 6229. dated March 1973.[2]

Following completion of the above HDC testing, the NASA redirected this program in an attempt to impr we the HDC CO_2 removal rate and to decrease the rate of cell voltage degradation. The final program consisted of the following tasks:

HDC Cell Pair Definition - Determine the HDC cell pair configuration for use on the SSP, as described in Report No. SVHSER 6229.

HDC Power Decay Investigation - Determine the cause for the degradation in HDC cell pair power which was experienced in units S/N 017 and S/N 018.

HDC Analytical Testing - Obtain a more detailed knowledge of the HDC cell operation and evaluate new concepts to improve its performance using Cs_2CO_3 and TMAC as the electrolytes in a small (1/24 sq. ft.) analytical cell.

Improved CO_2 Transfer - Improve the CO_2 removal efficiency of the HDC.

HDC Electrolyte Properties - Determine the various physical properties of the HDC electrolytes Cs_2CO_3 and TMAC.

Special Tests - HDC matrix and hydrogen flow tests.

HDC Verification Test - Demonstrate performance improvements with a 90-day verification test on two HDC cell pairs (one Cs_2CO_3 and one TMAC).

WVE Vibration - Evaluate the effects of vibration on performance and structural integrety of the WVE cell pair developed under the NAS 9-11830 contract.

WVE/HDC Breadboard - Fabricate and test a WVE/HDC breadboard unit. This task, however, was deleted after the basic cell pair rack assembly had been fabricated.

The successful completion of the previously described tasks provided:

● A more through understanding of the operation of an HDC cell.

● Detailed characteristics of the HDC electrolytes.

● A new HDC anode and cathode.

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● An HDC which has an improved CO_2 transfer rate and a lower power degradation rate.

● Verification that the present HDC and WVE cell pair design is capable of withstanding launch vibration levels.

CONCLUSIONS

The results of this program effort to improve the performance of the HDC, vibrate the WVE cell pair, and perform various HDC oriented research type tasks lead to the following conclusions:

- The factors which contribute to the internal cell resistance and determine cell power output are electrode polarization, electrolyte concentration polarization, and matrix-electrolyte resistance.

- Anode concentration overvoltage is a major contributor to low HDC cell power output and is the only source of internal cell power loss which offers hope for significant improvement in the future.

- Power output is very sensitive to cell temperature because of the temperature effect on electrode polarization, and there is some drop-off in rower when the hydrogen flow rate is reduced below three times stoichiometric (normal flow rate is five times stoichiometric).

- The main reason for HDC cell power decay was a change in the anode structure with time, caused by electrophoretic migration of the catalyst and Teflon binder.

- Some HDC cell power decay was attributed to matrix dissolution at the matrix-anode interface, because of the low anolyte pH.

- Trace impurities at the maximum allowable cabin level will have little effect on HDC cell power performance. In an environment typical of the present day urban terrestrial atmosphere, where impurity levels are much higher, some power loss could result from sulfur dioxide (SO_2) absorption. This power loss could be recovered by a closed circuit nitrogen purge of the HDC hydrogen chamber.

- The rate of CO_2 transfer is controlled mainly by the mass transport processes within the cathode structure. Thus, the cathode should be designed to obtain a high rate of electrolyte circulation within the electrode structure.

- The CO_2 transfer rate is essentially independent of cell temperature, hydrogen flow rate, and hydrogen pressure.

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- For maximum CO_2 transfer efficiency the optimum catholyte pH is in the range of 12.3 to 13.0. Within limits, the catholyte pH can be controlled by proper choice of air flow rate and current density.

- The minimum electrolyte-matrix resistance is obtained when the matrix is compressed to a thickness which results in a 50 percent matrix void volume.

- A matrix thickness (electrode separation) of 20 to 30 mils provides optimum HDC performance based on a compromise between CO_2 transfer and cell power.

- Compressive relaxation of the asbestos matrix is ⸱⸱⸱ after two weeks, and the final pressure is more than enough to maintain sufficient contact between the cell components (current collector-electrode-matrix).

- The HDC and WVE cell pair design can withstand launch vibration loads without structural damage or loss of performance.

RECOMMENDATIONS

Based on the various HDC and WVE test results of this program, the following recommendations are made:

- The recommended HDC electrodes which will provide increased CO_2 removal rates and reduced voltage decay rates are:

  Anode   - PPF Electrode

  Cathode - DS 16-0 Electrode

- Continue investigation of HDC anode and anode-matrix interface behavior for possible ways to further improve the cell power and life.

- Change the HDC matrix material from fuel cell chrysolite asbestos to a more acid resistant material. A possible replacement material is Neoprene asbestos.

- Tetramethylammonium carbonate (TMAC) is recommended as the HDC electrolyte for units which are required to operate over a wide range of inlet air temperatures and dew points.

- Determine the physical properties of TMA bicarbonate and measure the decomposition rate of TMAC in an operating cell. This task was not accomplished during this program because of program redirection.

- Continue the search for new HDC electrolytes which will provide increased performance and life.

- To provide an improved product two design changes of detail parts should be made:

  - The outer housings of the cell pair should have a 0.050 to 0.60 inch concave bend in them. This will provide a more evenly loaded matrix when the housings are assembled.

  - The air outlet holes and outer edges of the housings should be coated with Teflon to prevent any possible electrolyte creep.

DISCUSSION

The discussion of the results obtained from this program is separated into eleven sections. These sections correspond to the ten major tasks of the program, which are defined in the INTRODUCTION of this report, plus a discussion of the program's reliability and safety aspects, and are titled:

- SSP-HDC EVALUATION TESTING

- HDC POWER DECAY INVESTIGATION

- HDC ANALYTICAL TESTING

- IMPROVED CO_2 TRANSFER

- HDC ELECTROLYTE PROPERTIES

- MODIFIED ELECTROLYTES

- SPECIAL TESTS

- HDC VERIFICATION TEST

- WVE VIBRATION

- WVE/HDC BREADBOARD

- RELIABILITY AND SAFETY

SSP-HDC EVALUATION TESTING

This task pertained to the evaluation testing of the HDC cell pair concept to determine whether the Hamilton Standard design could be considered ready for inclusion in the Space Station Prototype (SSP) system. As a result of this test program, the NASA concluded that further cell development was necessary before a full scale system is constructed and that the remaining funding of contract NAS9-12920 should be directed towards this HDC development. A formal report on the SSP test program, SVHSER 6229, was prepared for NASA-JSC and is summarized in the following paragraphs.

Various tests, analyses and miscellaneous tasks were performed in support of this task.

Analytical and Miscellaneous Tasks

Objectives

The objectives for the analytical and miscellaneous tasks were:

● Perform instrumentation error analysis on various test data parameters.

● Normalize test data of Hamilton Standard's cell pair S/N 010 to show CO_2 removal performance versus time (226 days).

● Define the N_2 purge technique (if any) to be employed during cell pair testing.

● Develop a computer mathematical model to determine the number of cell pairs required to satisfy the SSP requirements, based upon the CO_2 removal performance achieved during the test program.

● Perform various rig modifications to improve cell pair current control and test chamber ambient conditions.

Conclusions

The conclusions reached as a result of the analytical and miscellaneous tasks were:

● The RSS Measurement error for determining CO_2 transfer rate was ±3.95% on the reported tests.

● Normalization of cell pair S/N 010 data revealed that CO_2 removal performance remained constant throughout the test.

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- A procedure for purging the cell with nitrogen once each day was developed and employed on each of the four tests of this test effort. This purge did not improve the CO_2 transfer efficiency, but it did decrease the rate of cell pair power degradation initially.

- An HDC system sizing computer program was developed based on actual cell pair performance. It established that 36 cell pairs would satisfy the SSP application.

- Modifications were made to the facility to enable constant current testing at selected current densities. Due to mechanical limitations, the test chamber to laboratory room air leakage rates were not improved. It was not established positively that the relatively high concentration of sulphur dioxide (20 ppm) which the test cells were consequently exposed to, contributed to the voltage degradation.

Cell Pair Test Program

Objectives

The objectives for all the cell pair test program were:

- Establish the adequacy of certain cell pair housings and electrodes.

- Evaluate the effects of varying matrix compression over a range compatible with cell assembly tolerances and matrix void volume variations.

- Determine the desirability of including Tissuquartz in the proposed SSP reservoir cell configuration to enable the cell to withstand a significant step change in inlet air temperature.

- Establish a map of cell performance at varying current density, inlet air temperature, inlet dew point, and carbon dioxide concentrations and evaluate the change in this performance versus cell operating time.

Conclusions

Conclusions reached from the cell pair testing were:

- Both the annealed and non-annealed housings were found acceptable; electroplated electrodes should be used in all tests.

- Short term testing, during which the matrix compression was varied from .022 to .030 inches revealed that cell voltage and CO_2 removal efficiency were independent from this change in matrix thickness.

● Tissuquartz assembled in strips within the matrix resulted in the reservoir cell being able to withstand a ± 4°F air inlet step change...this configuration was subsequently employed.

● A map of CO_2 removal efficiency for different operating conditions was established over an extended test period. The originally planned test duration of six to eight weeks was extended. After five months of continuous testing, no permanent decrease in cell current efficiency (performance) occurred. Cell voltage decreased with time over five months, but remained sufficiently high to accommodate the necessary CO_2 transfer rate of 13.2 lbs/day for the SSP application.

HDC POWER DECAY INVESTIGATION

During HDC cell testing under the SSP program, cell power degradation was found to be associated mainly with the anode. Potential decay curves of the cell anode and cathode versus a reference probe showed that the effective anode surfac. .r̄ a was decreasing with time, whereas the cathode area remained about the same.

Floating electrode te.̄. on anodes from cells that had decayed in performance gave almost the same performance as an unused electrode.* This indicated that most of the initial performance capabilities of the anode were still avaⅰⅼaⅮle, but for some reason were not being utilized in the full cell. ᵢⁱeveral possible causes for the loss in anode performance were postulated.

- Anode Poisoning from oxidizable trace impurities.

- Electrolyte imbalance resulting in flooding or dry-out.

- Change in electrode structure due to electrophoretic migration, which would affect the electrolyte take-up capabilities of the anode.

To distinguish between the various possible causes of anode performance decay, a number of experiments was conducted on full size cells. In addition, the behavior of selected trace impurities in the electrolyte was investigated in half cells, and a micrographic examination of used and unused anodes was conducted to determine if the electrode structure had changed upon use. The effect of matrix thickness on cell performance also was investigated.

Trace Impurities

The effectiveness of a closed circuit nitrogen purge of the anode chamber in restoring a significant part of the cell voltage for a short period of time would indicate the presence of oxidizable or desorbable impurities in the electrolyte, the effects of which would not necessarily be detected in the floating electrode test because the electrode sample is exposed to air prior to the test.

The purpose of this study was to determine whether certain selected trace impurities and their oxidation or reduction products poison the HDC electrodes, and/or accumulate to significant levels in the electrolyte. It also was a goal of this study to define simple methods of recovering from any adverse effects due to trace impurities entering the HDC cell. The testing was conducted in accordance with the Master Test Plan, Section VI, included as the Appendix of this report.

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*The anodic overvoltage at 20 asf for the used and unused electrode was 103 and 91 mV, respectively.

The trace impurities chosen for study and their maximum allowable levels in cabin air are given below. *

Impurity	mg/m^3	ppm	moles/m^3
CO	29	25	1×10^{-3}
H_2S	1.5	1.3	5×10^{-5}
SO_2	2.6	1.0	4×10^{-5}
NH_3	3.5	5	2×10^{-4}
Freon 113	700	100	4×10^{-3}

* Numbers supplied by the NASA JSC, using SSP as a guideline.

In the cases where the impurity was added directly to the electrolyte for testing the poisoning effects, the amount added to 35 ml of electrolyte was equal to the number of moles in a cubic meter of air. All potentials are with respect to a hydrogen reference electrode in the same solution. Since the nature of the cation has little effect on the adsorption or electrochemical properties of a platinum electrode, cesium carbonate, rather than TMAC (tetramethyl ammonium carbonate) was used in this work for convenience in solution preparation and purification. Also, it would be difficult to prevent TMA ion decomposition with the test procedure employed and the presence of these decomposition products might have interfered with the test results.

Ammonia

Ammonia (NH_3) can be oxidized at the HDC cathode to a number of products; the most important are listed below along with their approximate standard potentials.

NH_3 Oxidation Product	Standard Potential (Volts)
N_2	0.1
N_2O	0.5
NO	0.7
NO_2^-	0.8
NO_3^-	0.9

These numbers are only a guide, since the actual potential range where a significant oxidation rate occurs will depend on the kinetics of the various reactions. Potentiostatic sweeps were made to determine the actual potentials for ammonia oxidation and to help in identifying the major reaction products. A typical

potentiostatic sweep curve on platinum in Cs_2CO_3 solution is defined by the sketch below and is used as a baseline for comparison.
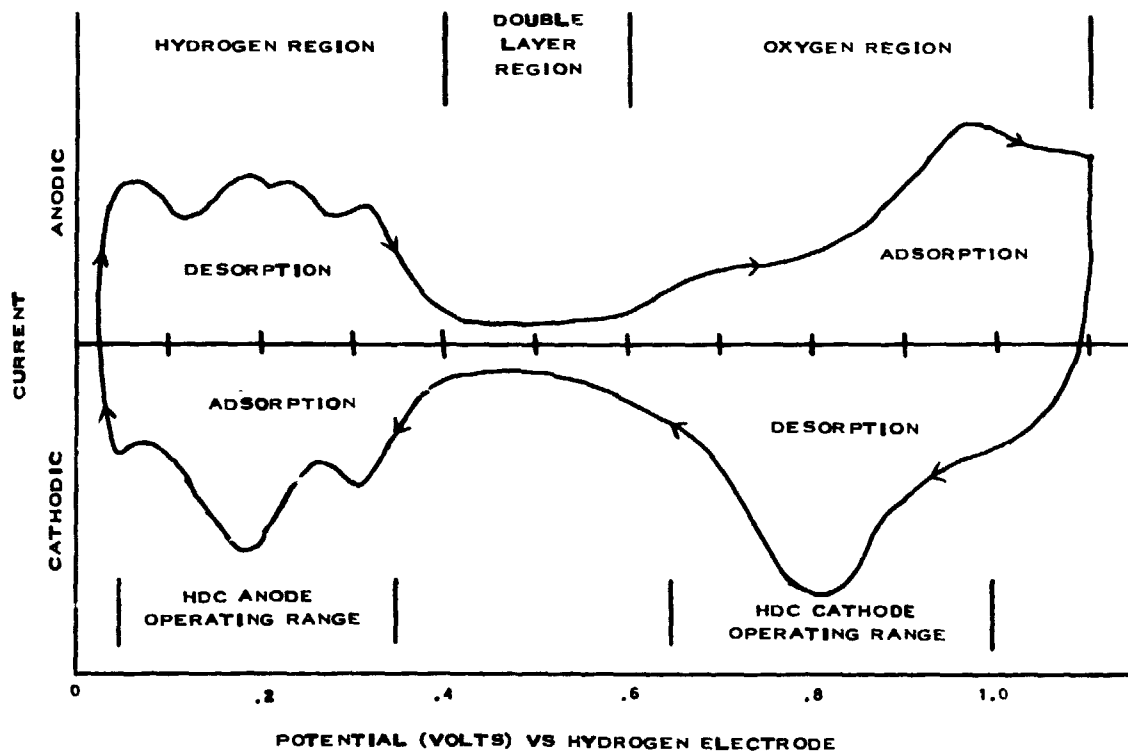
FIGURE 2. BASELINE POTENTIOSTATIC SWEEP ON
PLATINUM IN Cs_2CO_3 SOLUTION

It is desirable that the reaction products be gaseous rather than ionic to avoid accumulation of foreign ions in the electrolyte. The maximum solubility of ammonia in the catholyte in equilibrium with 5 ppm NH_3 in air is approximately 2×10^{-4} per liter. The initial potential sweep tests using this concentration showed no change in the sweep curves. Ammonium carbonate then was added to the cesium carbonate electrolyte at a level corresponding to approximately 30 times this maximum allowable NH_3 level in order to identify the reactions more readily. Figure 3 shows the potential sweeps with and without the addition of ammonia. There is definite evidence of some ammonia oxidation to nitrogen in the hydrogen adsorption region (below 0.4 volts) as expected, and a large anodic peak starts at 0.5 volts, which can be attributed to the oxidation of ammonia to N_2O. The formation of higher oxidation products (NO, NO_2^-) is not evident from these data and the excess cathodic current observed on the downsweep below 0.6 volts may be due to the reduction of any of these oxidation products formed during the upsweep. One interesting feature is the absence of an oxide reduction peak on the downsweep (0.8 volts) and it must be concluded that ammonia or its oxidation products inhibits the oxidation of platinum.

It will be shown later that the HDC cathode operates near 0.8 volts with respect to a hydrogen electrode in the same solution. This being the case, ammonia as a trace contaminant in the air stream would be oxidized mainly to N_2O and possibly some small amount of NO and NO_2^-. To determine if a significant amount of NO_2^-, an electrolyte contaminant, is formed under cathode operating conditions, the electrode was held at 0.8 volts for one-half hour followed by an upsweep to 1.1 volts and then down (figure 4, Curve 1). No increase in cathodic current was observed on the downsweep which could be attributed to NO_2^- reduction. To determine the potential range for NO_2^- reduction, 2×10^{-4} moles of sodium nitrite were added to the electrolyte and another sweep made (figure 4, Curve 2). A large reduction peak was observed in the hydrogen adsorption region (below 0.4 volts) so that even if some NO_2^- is formed at the HDC cathode it would be readily reduced to a gaseous product at the anode and therefore not accumulate in the cell electrolyte.

Since it was found that ammonia inhibits platinum oxidation, it was necessary to determine if trace amounts of ammonia would poison the cathodic reduction of oxygen by preventing oxygen adsorption on the platinum catalyst surface. For this test a floating SSP electrode was used with air, and 15 times the maximum amount of ammonia as ammonium carbonate was added to the cesium carbonate electrolyte. The results are shown in figure 5, where it can be seen that ammonia actually increases the cathode performance in the current density range of interest. This is reasonable because it was shown that ammonia inhibits platinum oxide formation and it is well known that bare platinum is a better catalyst for oxygen reduction than oxidized platinum. At low current densities ammonia oxidation results in a reduction of the net cathodic current.
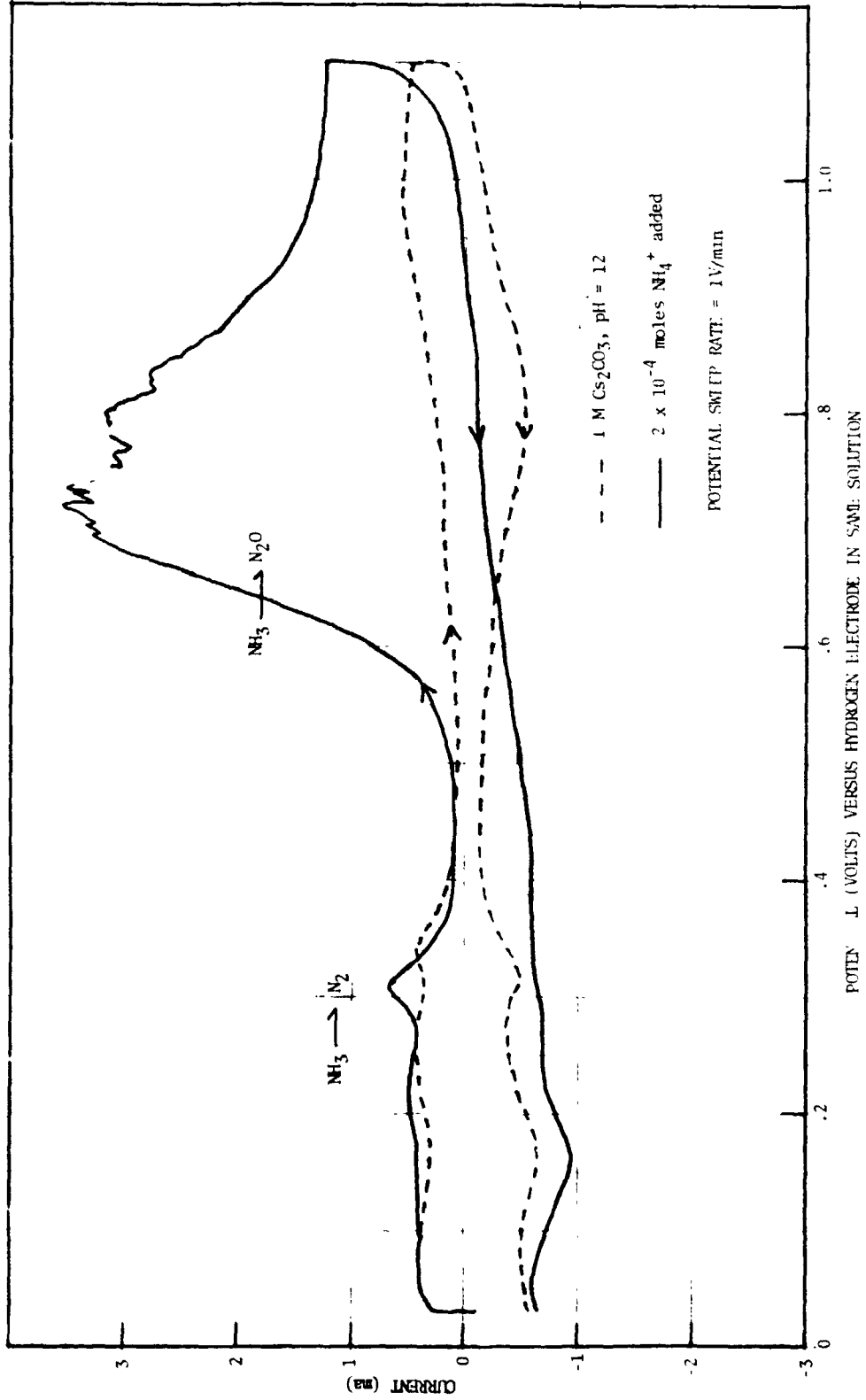
FIGURE 3. OXIDATION OF AMMONIA (NH₃) ON PLATINUM BLACK ELECTRODE

1 M Cs₂CO₃, pH = 12

——— TRACE NH₃⁺

− − − 2 x 10⁻⁴ moles NO₂⁻ added

POTENTIAL SWEEP RATE = 1V/min
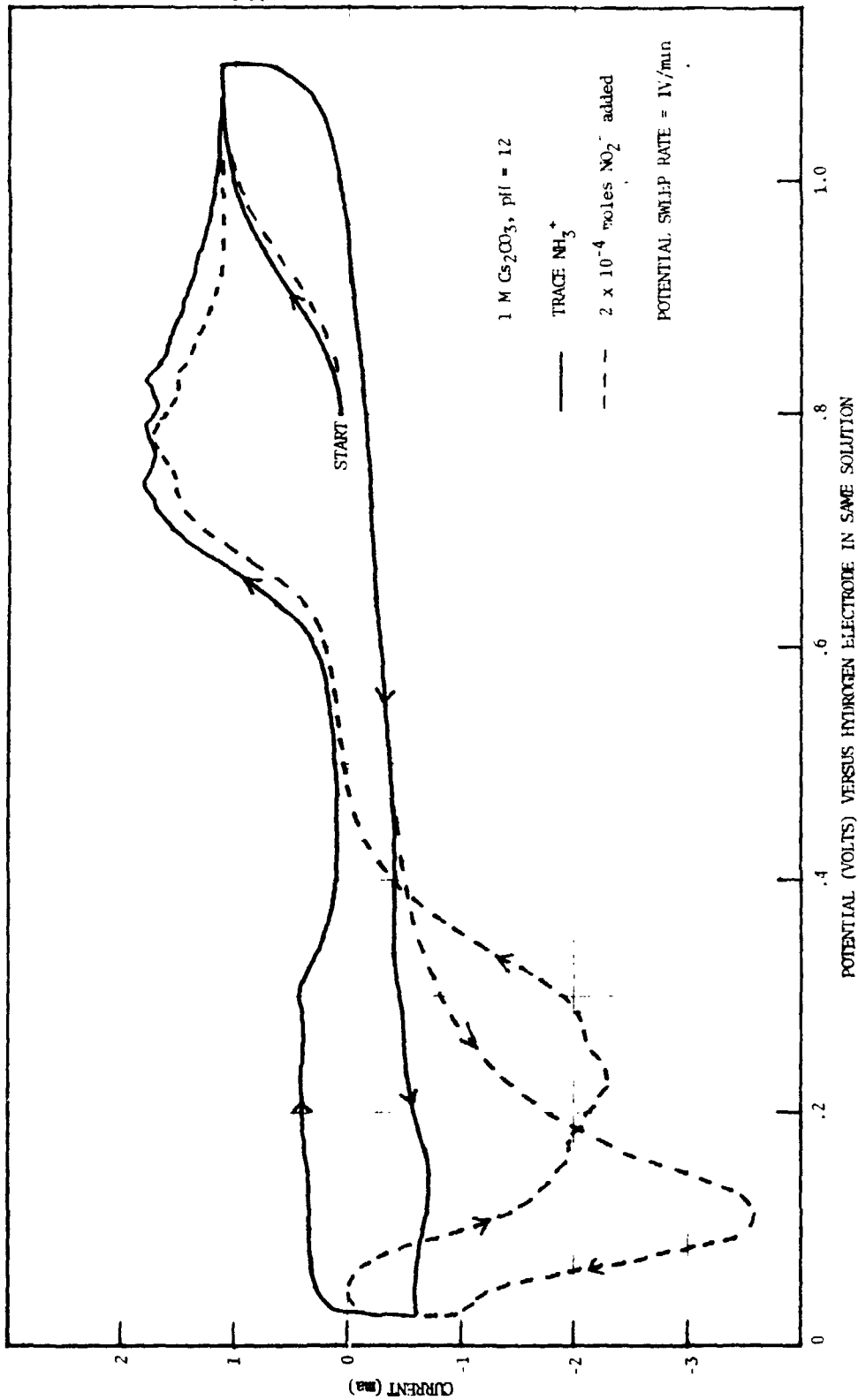
FIGURE 4. REDUCTION OF NITRITE ION (NO₂⁻) ON A PLATINUM BLACK ELECTRODE

POTENTIAL (VOLTS) VERSUS HYDROGEN ELECTRODE IN SAME SOLUTION

CURRENT (ma)
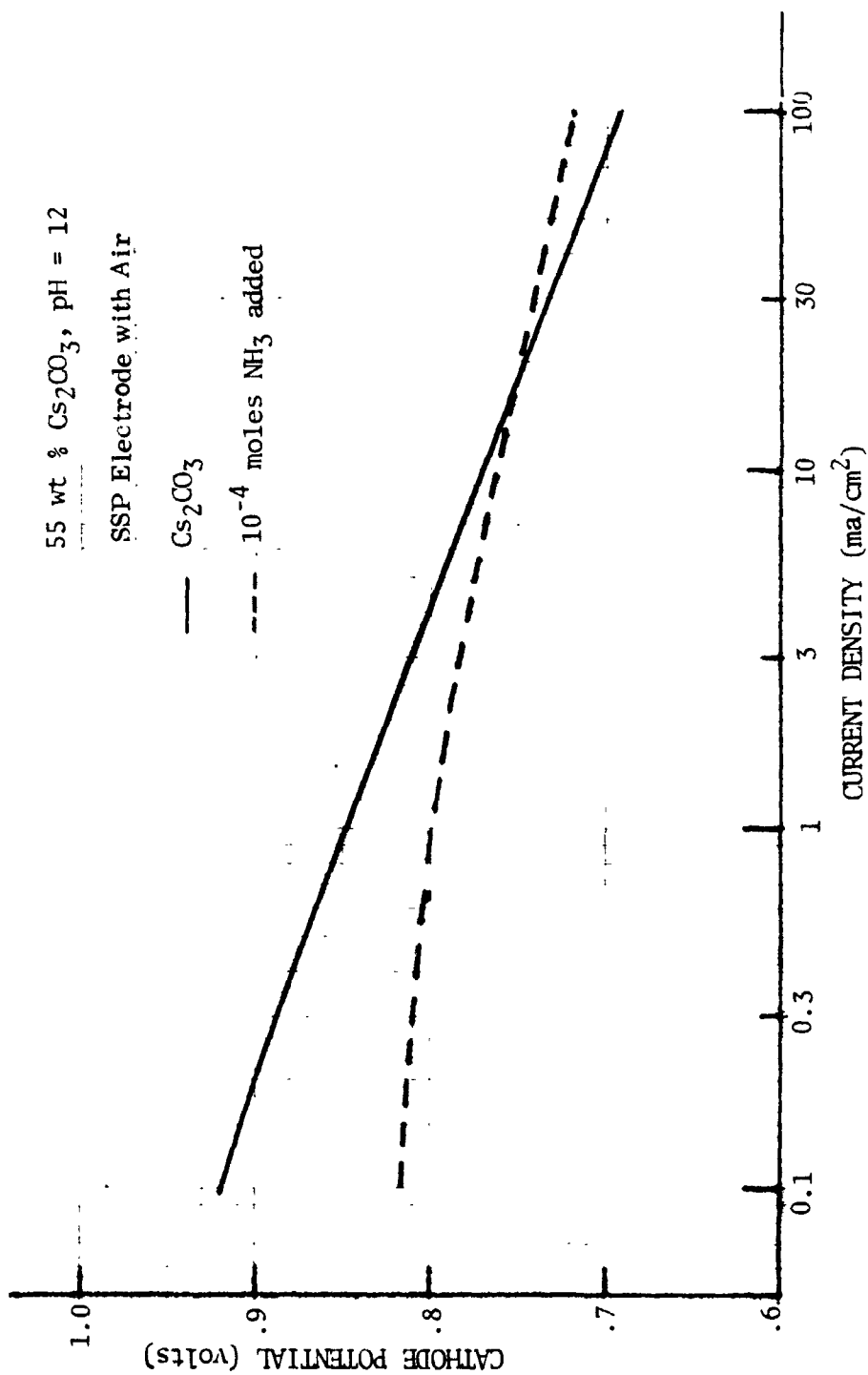
FIGURE 5. EFFECT OF AMMONIA ON CATHODE PERFORMANCE
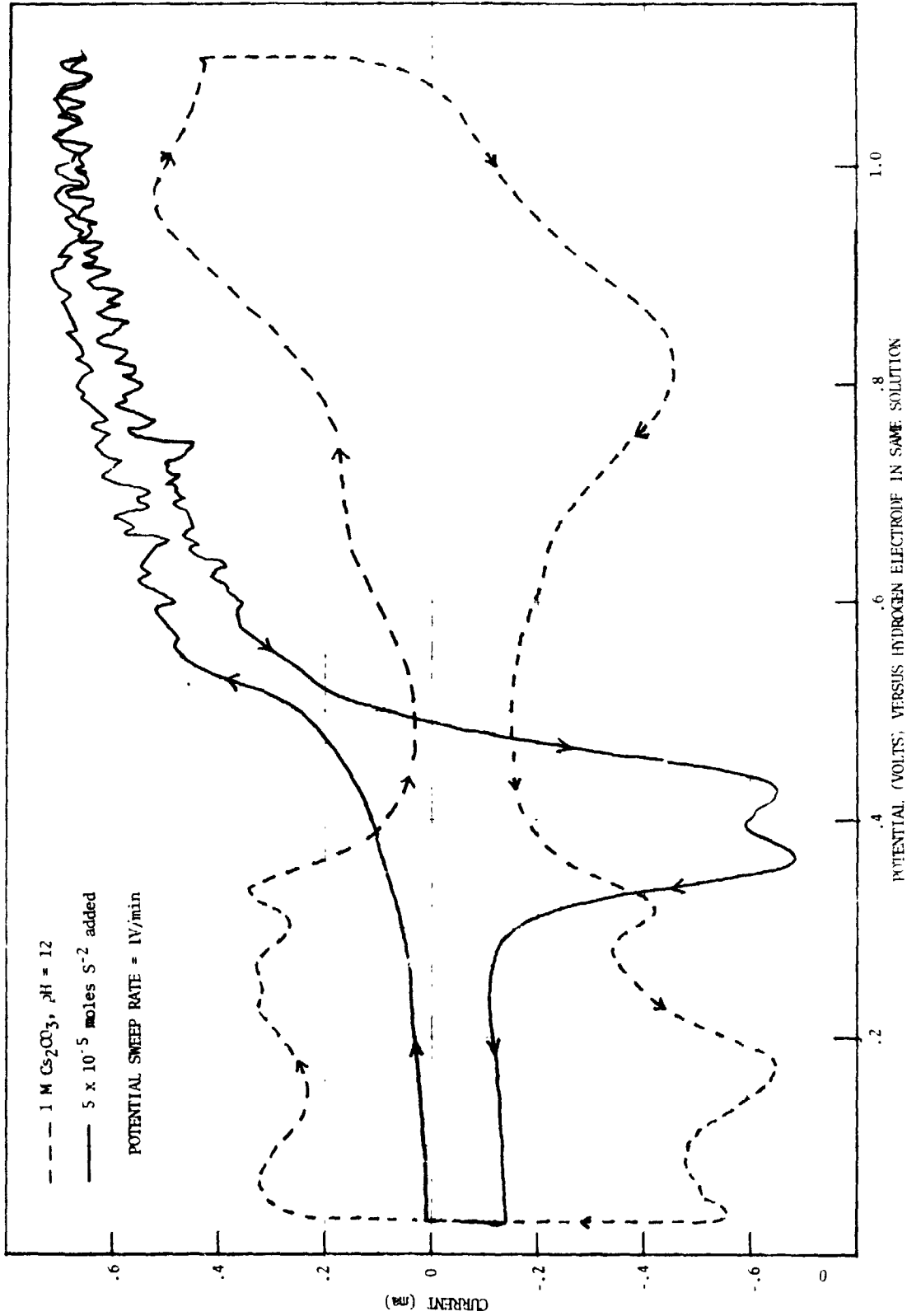
FIGURE 6. OXIDATION OF SULFIDE ION (S⁻²) ON A PLATINUM BLACK ELECTRODE.

POTENTIAL (VOLTS; VERSUS HYDROGEN ELECTRODE IN SAME SOLUTION)

CURRENT (ma)

- - - - 1 M Cs₂CO₃, pH = 12

──── 5 x 10⁻⁵ moles S⁻² added

POTENTIAL SWEEP RATE = 1V/min

Hydrogen Sulfide and Sulfur Dioxide

In addition to trace amounts of sulfur dioxide (SO_2) and hydrogen sulfide (H_2S) in the air stream, it is possible to get H_2S in the hydrogen gas from improper operation of the WVE. It is therefore necessary to consider the effect of H_2S on both the anode and cathode of the HDC. Both H_2S and SO_2 are acid gases and will react with the HDC electrolyte to form sulfide (S^{-2}) and sulfite (SO_3^{-2}) ions, respectively. Thus, these ions could accumulate in the electrolyte, reaching significant levels over a period of time. The only thermodynamically stable oxidation product of H_2S or SO_2 in the HDC electrolyte is the sulfate ion (SO_4^{-2}), although intermediate oxidation products such as sulfur and the various sulfur oxides will be produced at the HDC cathode during the oxidation of the sulfide or sulfite. The sulfate ion is not detrimental to the HDC electrolyte or electrodes; in fact, it was evaluated as an electrolyte additive to increase anolyte conductivity and lower anolyte pH under certain operating conditions. It can be calculated, considering mass transport limitations for H_2S and SO_2 in the air stream, that it would take about 10^5 hours for the sulfate concentration in the HDC electrolyte to reach a significant level (0.5 moles per liter).

A slow potential sweep, with and without the sulfide ion, is shown in figure 6. It can be seen that hydrogen absorption is strongly inhibited and sulfide oxidation becomes evident starting at ~0.15 volts and reaches a mass transport limiting current density at ~0.6 volts. On the downsweep, a reduction peak is seen at ~0.4 volts. In separate experiments it was determined that the number of coulombs corresponding to this reduction peak was 1/7 that of the coulombs for oxidation at 0.8 volts. Oxidation at more positive potentials resulted in a decrease in the number of coulombs for the reduction peak. From these results it can be concluded that this peak corresponds to the reduction of an adsorbed intermediate reaction product formed during sulfide oxidation.

The behavior of SO_2, which becomes SO_3^{-2} (sulfite) upon interaction with the electrolyte, is shown in figure 7. There is little effect on hydrogen adsorption and even after holding the potential at -0.16 volts for 10 minutes there was no evidence for the formation of SO_3^{-2} reduction products or a decrease in the amount of adsorbed hydrogen. Apparently the reduction of SO_3^{-2} is very slow under HDC anode conditions. Oxidation of SO_3^{-2}, presumably to sulfate, is obvious at 1.1 volts. Since the HDC cathode operates at 0.8 volts, it is expected that SO_2 would accumulate in the HDC electrolyte as SO_3^{-2} until the concentration reached a point where the rate of removal via oxidation, reduction and/or discharge in the hydrogen stream becomes equal to the rate of absorption. Over a relatively long period of time it is expected that sulfide would accumulate on the anode from sulfite reduction or H_2S adsorption, but this sulfide could be removed readily by increasing the anode potential to that of the air electrode (nitrogen purge, closed circuit) for a short period of time. The above reasoning is supported by gas analysis performed on the HDC cell. During normal operation it was found that approximately 1 percent of the SO_2 in the air stream (20 ppm) is transferred through the cell, but during a closed circuit nitrogen purge the amount of SO_2 in the HDC anode compartment increased by a factor of 100.
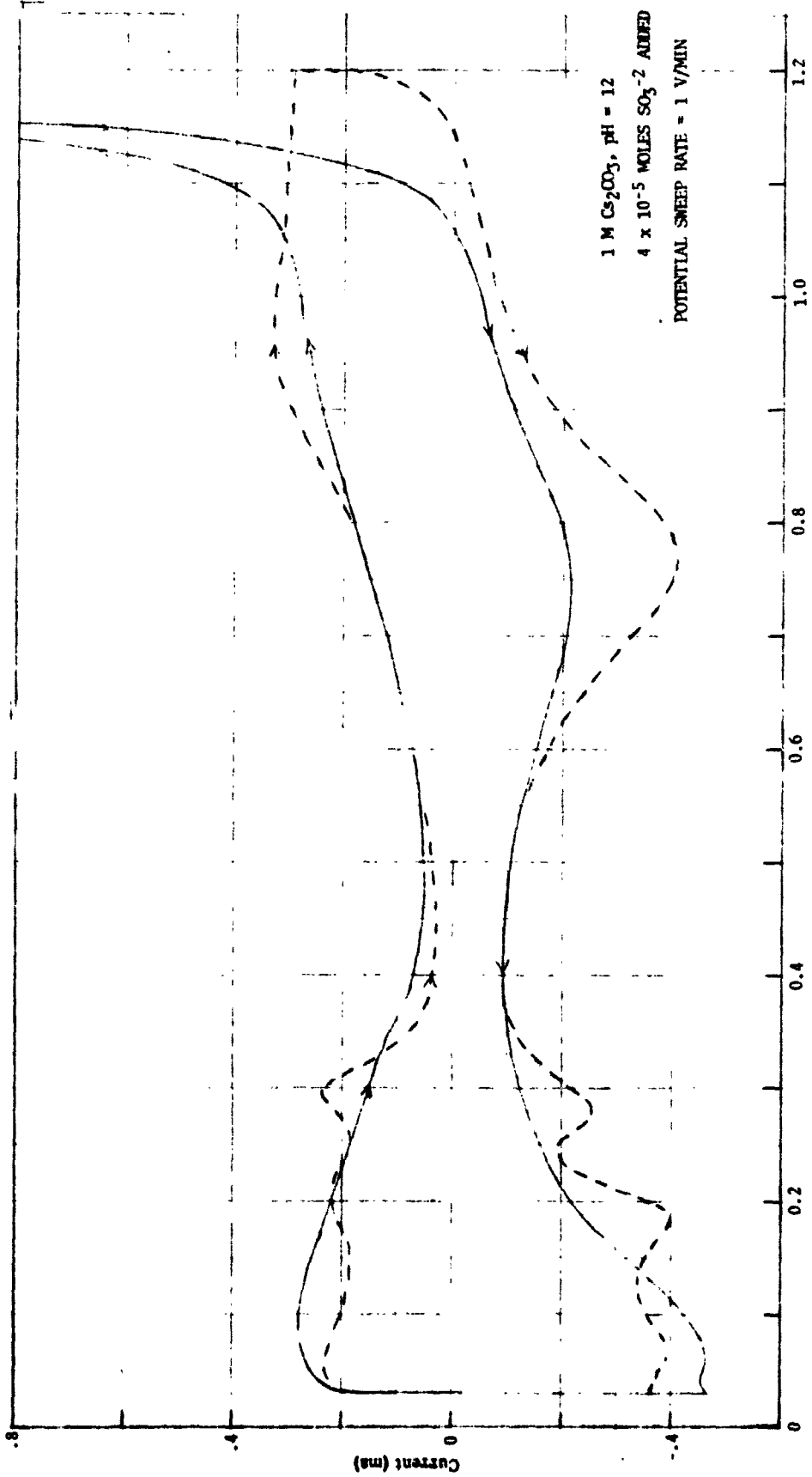
POTENTIAL (VOLTS) VS HYDROGEN ELECTRODE

FIGURE 7. OXIDATION OF SULFUR DIOXIDE (SO_3^{-2}) ON A PLATINUM BLACK ELECTRODE

1 M Cs_2CO_3, pH = 12

4 x 10^{-5} MOLES SO_3^{-2} ADDED

POTENTIAL SWEEP RATE = 1 V/MIN

Carbon Monoxide

The only stable oxidation product of carbon monoxide (CO) under HDC cell conditions is carbon dioxide (CO_2). It is well known that carbon monoxide poisons the hydrogen anode reaction but at the 22 ppm level only a slight reduction hydrogen adsorption was detected and no oxidation peak for CO could be found.

To determine the potential range for CO oxidation to CO_2 the cesium carbonate electrolyte was saturated with CO followed by a nitrogen purge for 20 minutes to remove the dissolved CO. Since the CO is strongly adsorbed on the electrode surface at +30 mV, little CO desorption would occur during the nitrogen purge.

Figure 8 shows the subsequent potential sweep compared to a sweep on a clean electrode surface. There is a significant reduction in the amount of adsorbed hydrogen, and the adsorbed CO gives an oxidation peak at ~0.7 volts. From these results it can be concluded that carbon monoxide would be readily oxidized at the HDC cathode and if a small amount eventually did get to the anode it could be easily removed by the nitrogen purge technique.

Freon 113

Freon 113 (CCl_2F-CF_2Cl) should be electrochemically inert under HDC cell conditions, since chemiadsorption is highly unlikely for such a stable chemical structure. Some physical adsorption may be possible in the double layer potential region (0.4 to 0.6 volts) but the Freon would be readily replaced by chemiadsorbed hydrogen or oxygen at the anode or cathode, respectively.

Potential sweeps with and without Freon 113 gave identical traces; therefore Freon 113 should have no detrimental effect on electrode performance. With HDC electrodes, which contain Teflon, the Freon may adsorb on the Teflon surface but the electrode wetting characteristics should not change significantly.

Conclusions

In view of these results it can be concluded that the above trace impurities at e levels normally present in cabin air will have little effect on HDC cell power performance. At much higher levels some poisoning could occur from H_2S, CO and SO_2, but an open circuit nitrogen purge of the hydrogen chamber would oxidize these materials off the electrodes and restore power. Sulfur dioxide (SO_2) would be the most difficult of the above impurities to remove, since it accumulates in the electrolyte as the sulfite ion (SO_3^{-2}). Fortunately, it does not poison the electrodes directly and its rate of reduction to sulfur or sulfide (both poisons) is very slow.
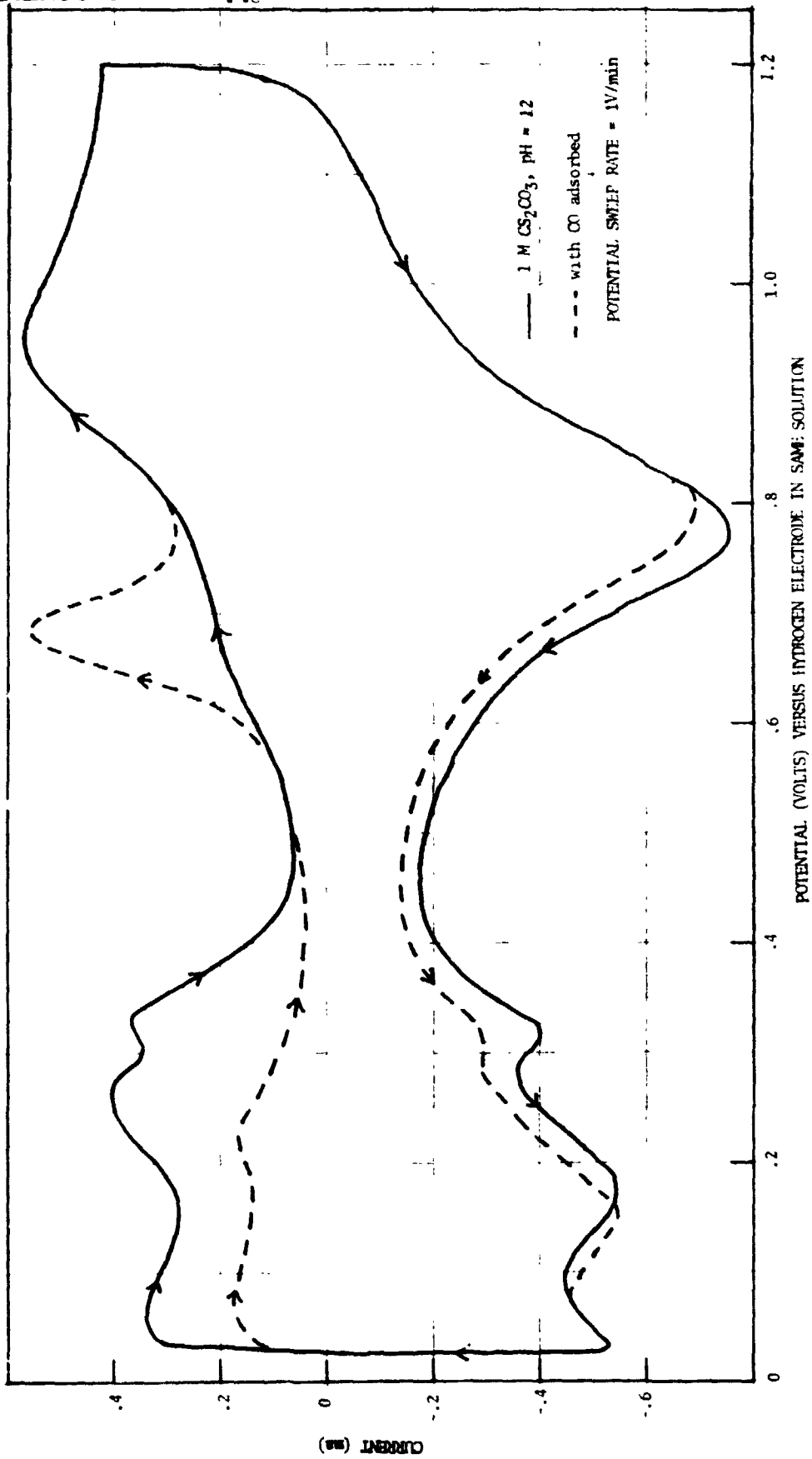
FIGURE 8. OXIDATION OF CARBON MONOXIDE (CO) ON A PLATINUM BLACK ELECTRODE

From our experience with the electrochemical behavior of organic materials it is predicted that their rate of oxidation to CO_2 and H_2O in the HDC or WVE cell would exceed their rate of adsorption from the air stream. Thus, no significant poisoning from organic impurities is expected for the normal cabin concentrations.

Experiments on Cell Pair S/N 017

Experiments were conducted on cell pair S/N 017 (per the Master Test Plan, Section X, reference Appendix) in an attempt to isolate the cause of the degradation in cell power (~200 μ volts/hr at 18 asf) experienced over a six month period.

The first approach was to establish whether electrolyte imbalance was a problem, although this is unlikely with the reservoir type cell unless the capacity of the reservoir is exceeded. A wet condition causing electrode flooding results in very little loss in cell power and would not persist because the relatively high air flow rate would remove the excess electrolyte from the cell. In order to be absolutely sure that excess electrolyte was not the problem, the air and hydrogen passageways were flushed with nitrogen at a high flow rate. The amount of electrolyte removed from the cell by this procedure was less than 1.0 ml, and there was no change in cell power. To determine if the cell was too dry, 20 ml of electrolyte were added to the reservoir, and after two days there still was no effect on cell power. To eliminate the possibility that the reservoir was not feeding electrolyte to the cell matrix, 5 ml of electrolyte were then added to the top cathode via the air passages. This resulted in a slight decrease (0.2 watts) in cell power, and potential decay measurements showed a smal loss in effective cathode area, as might be expected, but no change in anode area. At this point there was more than sufficient electrolyte in the cell and a vacuum was applied to the hydrogen side for four hours in order to force electrolyte into and through the anode. This procedure resulted in a substantial increase (1.8 watts) in cell power, which decayed slowly back to the original level after three days. Potential decay measurements after the vacuum treatment showed a large increase in effective anode surface area and a small increase in cathode surface area. The slow decay of cell power during the next three days was found to be associated with a gradual loss in effective anode surface area as determined by potential decay measurements. The results of this experiment definitely indicate that the anode gradually loses its ability to take up electrolyte from the matrix.

Since the vacuum treatment did not result in complete recovery to the original power level, it was reasoned that other factors could be contributing to the power decay. The accumulation of trace impurities in the electrolyte still was a possibility. If a closed circuit nitrogen purge of the anode resulted in the desorption rather than the oxidation of a given impurity, a temporary power recovery would be expected as observed.

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A temporary power recovery from a closed circuit nitrogen purge also would be expected if a relatively large amount of impurity, such as SO_2 (SO_3^{-2}) had accumulated in the electrolyte. To establish if either of these possibilities had contributed to the power decay of cell pair S/N 017, the anode was cleaned electrochemically (closed circuit nitrogen purge followed by an oxygen purge) and maintained in the oxidized state while the electrolyte was replaced by clean electrolyte. This was accomplished by flushing distilled water through the cell via the hydrogen passageways, through the anode, matrix, cathode and then out of the air passageways. This flushing was continued until the water pH and conductivity indicated all the electrolyte had been removed. The cell then was purged with nitrogen to remove most of the water and then filled with clean electrolyte by the above flushing technique.

Cell pair S/N 017 was operating at 1.3 watts (15 amps) before anode cleaning and electrolyte replacement. The initial power after electrolyte replacement was above 4 watts and had decayed to 2.3 watts after six days. Also, the voltage decay rate (630 μv/hr) at 15 asf decreased with time. This behavior would not be expected for power degradation caused by trace impurity pickup. Unfortunately, due to scheduling, the test could not be continued long enough to establish if there would be a leveling off of the power above the previous 1.3 watt value to give a net gain which might be attributed to the elimination of an anode poison. In any case the difference would have been less than 0.5 watts, which is small compared to the total power loss experienced with this cell pair.

Upon disassembly of cell pair S/N 017 everything seemed normal except for a slight embrittlement of the anode current collector screen. This embrittlement of the anode screen was observed also in the first analytical cell test. In both cases it was found that these particular screens had been coated with platinum by sputtering rather than the standard electroplating process. Apparently, something associated with the sputtering process or the characteristics of the resulting coating makes the tantalum screens susceptible to hydrogen embrittlement. It is believed that this screen embrittlement did not contribute significantly to cell pair S/N 017 decay because cell pair S/N 018, which had an electroplated screen not affected by embrittlement, showed the same power decay characteristics ($\sim$ 200 μ volts/hr at 18 asf).

Cell Pair S/N 019 Testing

Late in the SSP HDC program it was realized that one of the causes for HDC cell power decay might be associated with a change in the anode structure due to electrophoretic platinum migration. Therefore, a modified catalyst with reduced electrokinetic effects was used for the cell pair S/N 019 anode (P&WA PPF electrode). These anodes were run for a total of 3700 hours with Cs_2CO_3 electrolyte and showed a voltage decay at 18 asf of only 20 microvolts per hour (figure 9). The average CO_2 transfer efficiency, at 18 asf and 3 mm Hg P_{CO_2} was a low 52 percent because of the modified cathode structure used in this cell. After development of the improved DS 16-0 cathode, cell pair S/N 019 was rebuilt with the same PPF anodes and the CO_2 transfer efficiency increased to 86 percent. These results are discussed in more detail on page 52.
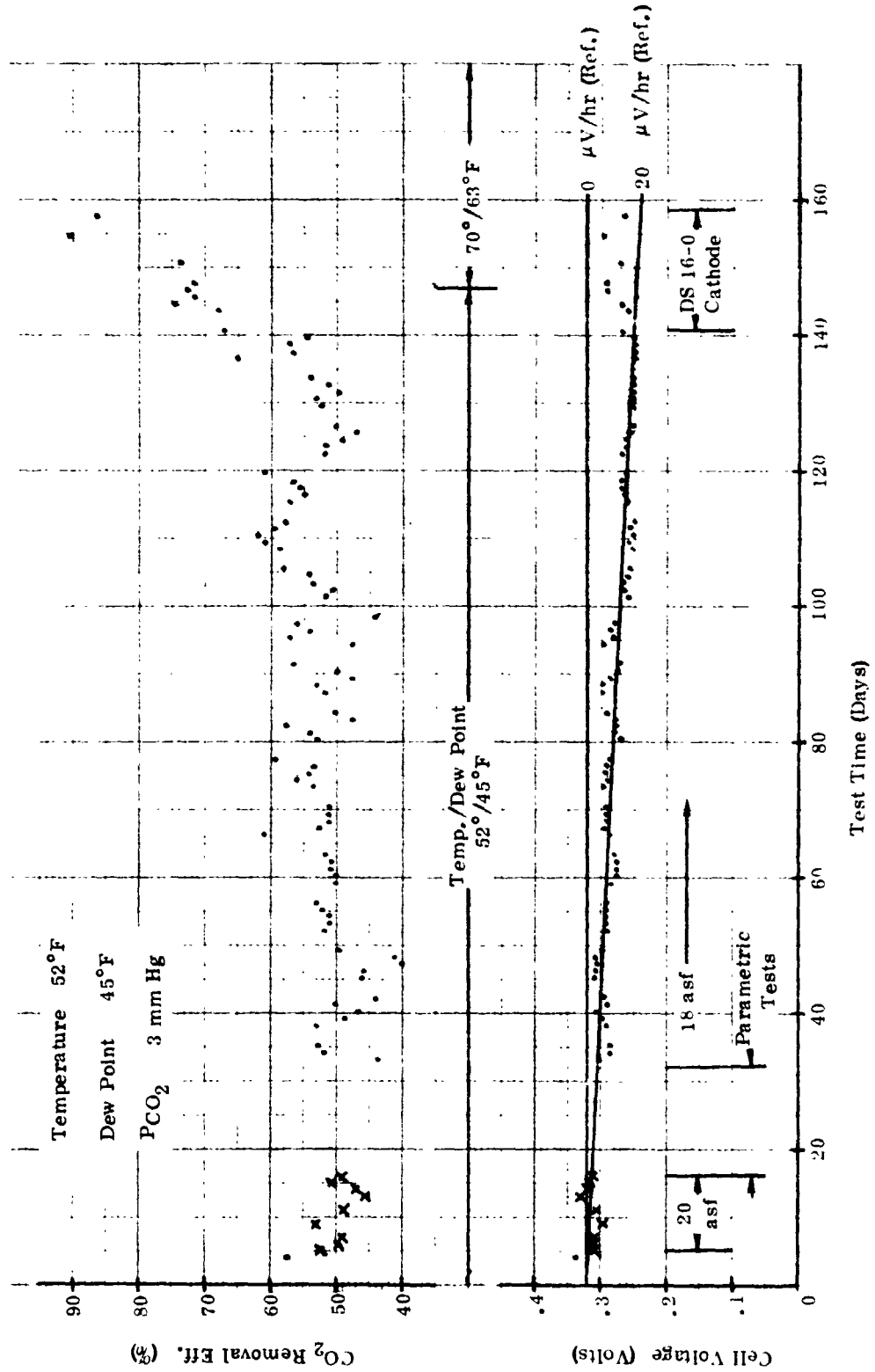
FIGURE 9. HDC CELL PAIR S/N 019 (Cs_2CO_3) PERFORMANCE VS TIME

Since the voltage decay rate of cell pair S/N 019 (20 μv/hr) was such a large improvement over that of previous cells (~ 200 μv/hr) it was deemed worthwhile to examine the anodes of cell pairs S/N 018 and S/N 019 to see if any structural differences could be detected. The anode of cell pair S/N 018 rather than S/N 017 was chosen as more representative of previous cells because of the screen embrittlement problem encountered with the cell pair S/N 017 anode.

Anode Examination

Micrographic examination of the anodes from cell pairs S/N 018 and S/N 019 included the use of optical, scanning electron microscope (SEM), X-ray microprobe, and replica election microscope analyses. The SEM and X-ray microprobe results were the most informative and only these pictures are shown here, in figures 10, 11 and 12. Both unused and used samples of the electrodes were examined in cross section.

The unused electrode (cell pair S/N 018) showed a fairly even platinum distribution (figure 10a) with a tight structure of low void volume (figure 11a). The outer surfaces of the electrode contained a slightly higher Teflon concentration than the interior (figures 10a and 12a).

The used anode (7 months of operation in cell pair S/N 018) had increased in thickness by almost a factor of two as can be seen from a comparison of figures 10b and 11b with figures 10a and 11a. In the expanded or "puffed out" area on the gas side, the platinum-Teflon ratio is essentially the same as the original; however, the catalyst density is about one-half that of the original electrode. This resulted in a large increase in void volume at the gas side and to a lesser extent on the electrolyte side of the anode, as can be most clearly seen from the platinum distribution scan of figure 10b compared to figure 10a. It is also obvious from a comparison of the Teflon distribution scans shown in figure 10 that the Teflon has migrated in the opposite direction; i.e., toward the matrix. This Teflon migration is even more dramatically evident from the characteristic X-ray distribution for platinum and fluorine (Teflon) shown in figure 12.

Since both the platinum and the Teflon are in the colloidal state, it is not surprising that they would tend to migrate in the electrical field (electrophoresis) of the cell. The platinum, having a negative charge, would be forced away from the cell cathode which is opposite to the direction of electrolyte pumping (electroosmosis). The Teflon, with a positive charge, would migrate toward the cell cathode. The migration of the Teflon in the anode toward the matrix would result in a large increase in the hydrophobicity at the electrode - matrix interface. This, combined with the increase in void volume, would greatly reduce the electrode capillarity and therefore its ability to pull back electrolyte from the matrix. The result is a reduction in the utilized catalyst area with time and therefore a decrease in anode electrochemical
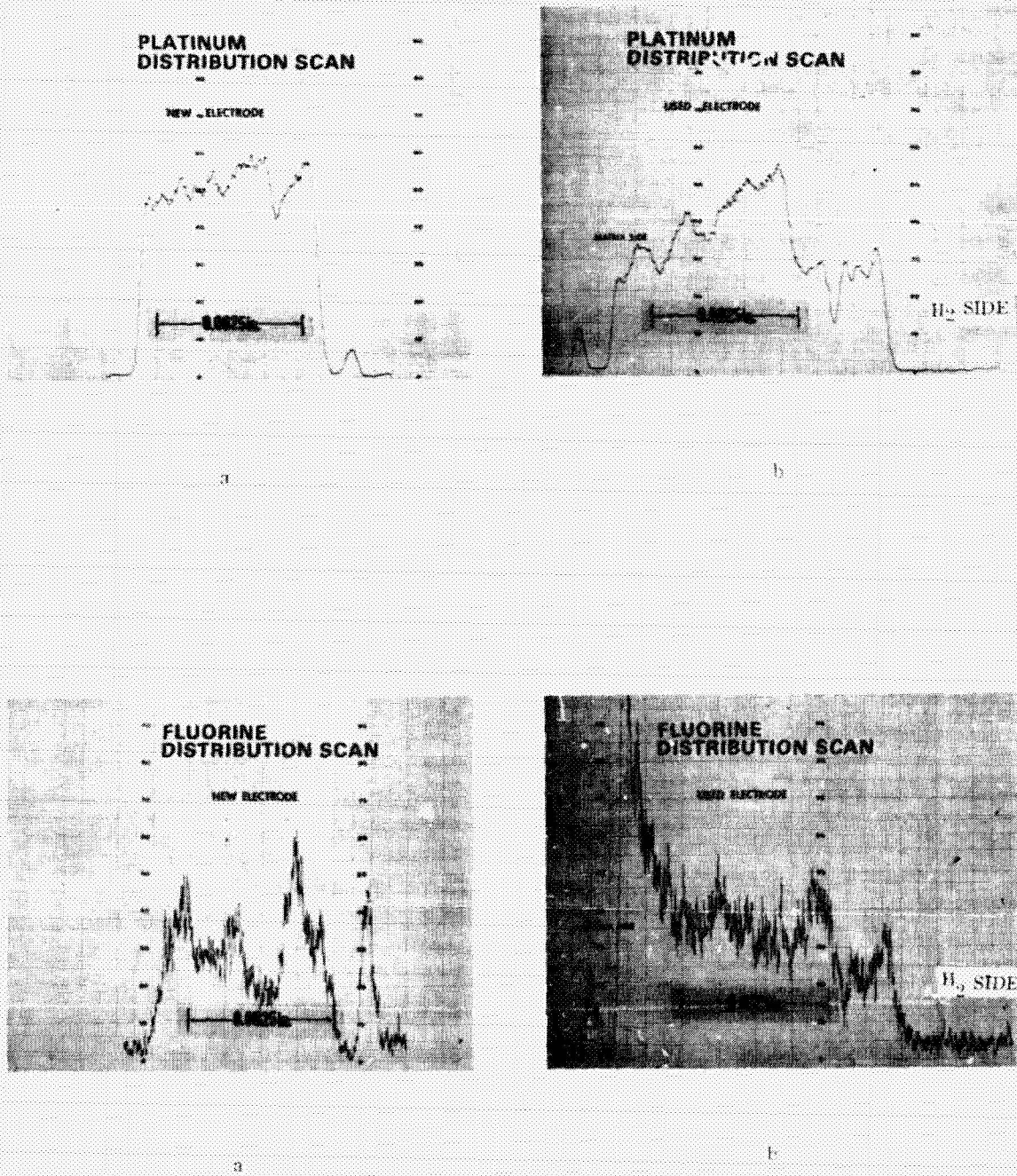
FIGURE 10. X-RAY MICROPROBE LINE SCANS OF CELL P2 IR S/N 018 ANODE

GAS SIDE
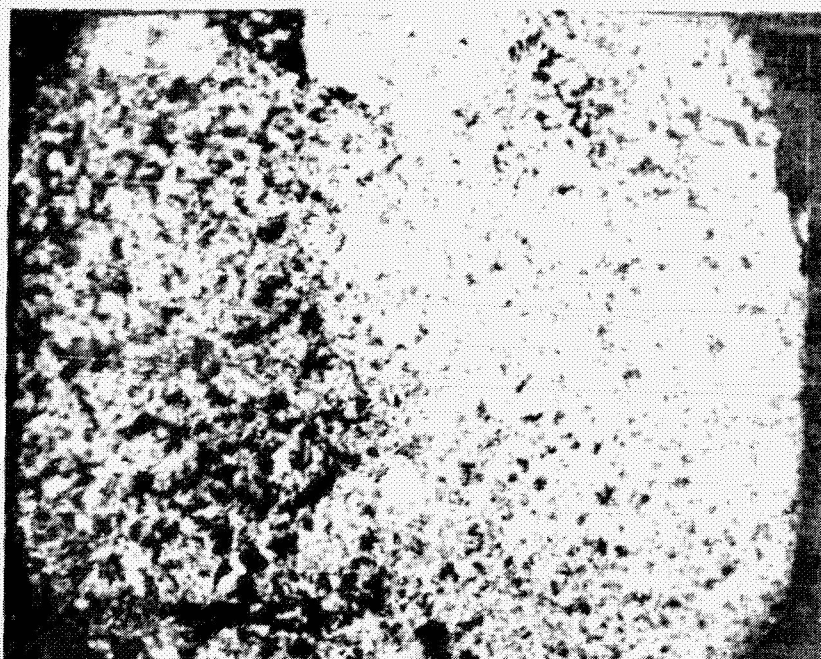
MATRIX SIDE

b

a

FIGURE 11. SEM EXAMINATION OF CELL S/N 018 ANODE ($\cdot$ = 750 X)

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a. UNUSED

b. USED

H₂ SIDE

ELECTRO LYTE SIDE
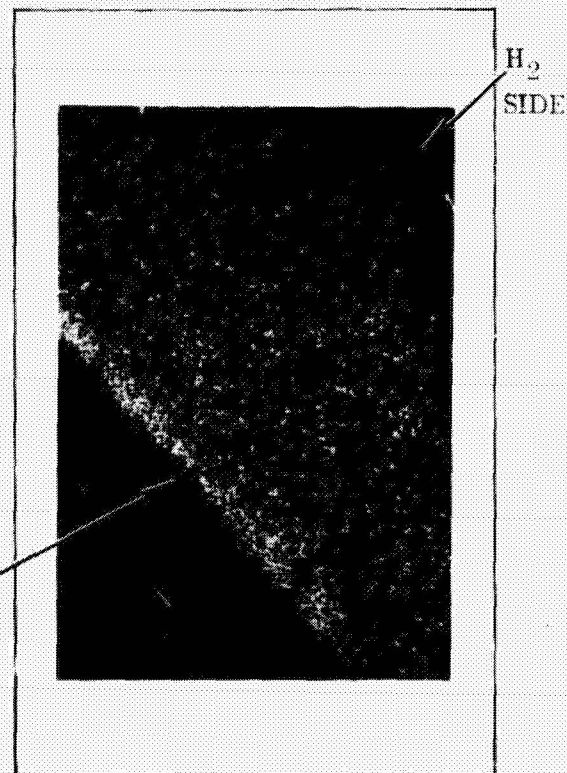
COLOR CODE: GREEN - PLATINUM
RED - TEFLON
YELLOW - PLATINUM AND TEFLON

FIGURE 12. CHARACTERISTIC X-RAY DISTRIBUTION FOR PLATINUM
AND FLUORINE (TEFLON) OF CELL S/N 018 ANODE

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performance. The performance can be increased temporarily by forcing electrolyte into the anode structure, e.g. by pulling vacuum on the hydrogen side. This has been demonstrated many times, but after a short interval of time the electroosmotic pumping restores the amount of electrolyte in the anode to its equilibrium level.

Most of the catalyst in the expanded area (gas side) is probably no longer effective because of the high void volume. This may account for the fact that complete recovery of the cell power to the original level was not obtained after the vacuum treatment or the electrolyte replacement.

The observation that the cell power decay rate after electrolyte replacement was slower than after a vacuum treatment can be explained by the difference in electrolyte availability in the two cases. After electrolyte replacement, there was initially an excess of electrolyte at a higher than equilibrium concentration so that the cell to anode electrolyte pressure differential was small. In contrast, the vacuum treatment decreased the amount of available electrolyte, thereby increasing the capillary pressure differential between anode and reservoir which would reduce the anode electrolyte take-up rate.

In view of the observed changes in electrode structure the mechanism by which either an open circuit treatment or a nitrogen purge causes temporary cell power increase can be explained. Reducing the cell current to zero via open circuit or a closed circuit nitrogen purge would stop electroosmotic pumping of electrolyte out of the anode structure and allow electrolyte replenishment into the anode structure via capillarity. The fact that a nitrogen purge is more effective than an open circuit treatment in increasing cell power may be related to the difference in anode potential in the two cases. At open circuit the anode potential goes to zero with respect to a hydrogen reference electrode in the same solution but during a closed circuit nitrogen purge the anode potential increases to ~1.1 volts with respect to the same reference. Since the electrocapillary maximum for platinum in aqueous solution is nearer to zero volts, a nitrogen purge would affect a greater reduction in the platinum-solution interfacial tension, thus making the spreading coefficient more positive which results in an increased anode wicking rate for electrolyte take-up.

The PPF anode from cell pair S/N 019 showed no evidence of any structural changes by optical or SEM examination. The X-ray microprobe line scan (figure 13) showed no evidence of Teflon migration and, at most, only a slight migration of platinum toward the hydrogen side. The gap near the matrix side in the scan of figure 13 b is due to a crack in the electrode which developed during the sample mounting process.
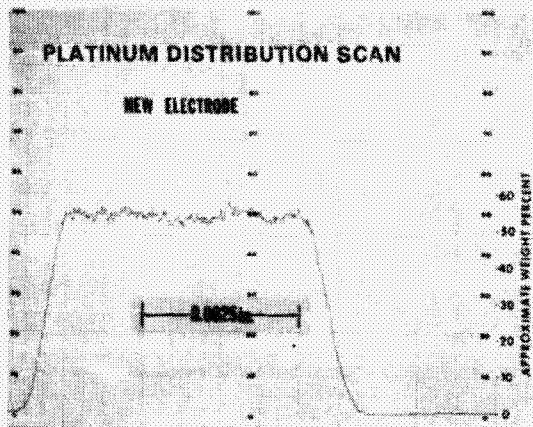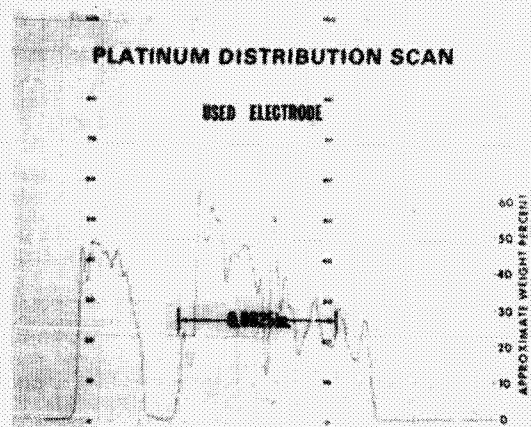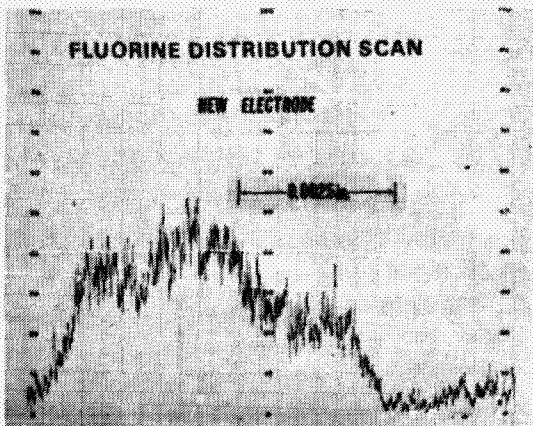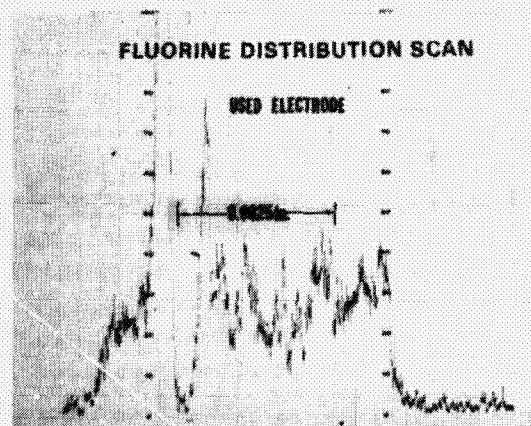
FIGURE 13. X-RAY MICROPROBE LINE SCAN OF CELL PAIR S/N 019 ANODE

Anode Evaluation

Four other electrodes (A-3R, A-10R, SEC and PVC) were screened for initial power performance as HDC anodes via floating electrode tests[3] (test set up, Reference figure 14). The A-3R and A-10R electrodes are intended to be more CO and possibly "reduced CO_2" tolerant, whereas the SEC and PVC electrodes represent a considerable reduction in catalyst loading and cost without a sacrifice in power performance at high temperatures. Table I gives the anode performance of these electrodes in addition to the SSP, PPF, and American Cyanamide AA-2 electrodes. The SEC electrode (4 mg cm^{-2}) gave the best power performance, but it was found that the catalyst was unstable in the $CsHCO_3$ electrolyte. The PPF electrode at a catalyst loading of 15 mg cm^{-2} had essentially the same power performance as the SEC and, from cell pair S/N 019R experience, the PPF performance degradation with time is very small.

If the data in Table I is normalized per mg cm^{-2} of catalyst, the PVC electrode containing only 1 mg cm^{-2} of active supported catalyst looks promising. Unfortunately, the catalyst-to-support ratio is at its maximum level and increasing the loading by a factor of three, to match PPF performance, would result in an electrode of unreasonable thickness.

From these results and from cell pair S/N 019 power performance, the PPF electrode was chosen for use as the anode in future cell tests.

Matrix Thickness and Cell Performance

The matrix thickness test was run under the following conditions: $P_{CO_2} = 3$ mm Hg, air velocity = 15 ft/sec, current density = 18 asf, air inlet temperature/dew point = 70°/63°F, ambient pressure = 14.7 psia.

One, two, and four layers of asbestos were used at the optimum compression thickness of 12.5, 25, and 50 mils, respectively. The electrolyte was Cs_2CO_3 (Reference Appendix, Master Test Plan, Section IX).

The results given in Table II show the expected trend in CO_2 processing efficiency; however, the high IR drop for one asbestos layer (compressed to 12.5 mils) is difficult to explain.* Also, in view of the anode and cathode pH values, the high cell voltage obtained with one asbestos layer seems peculiar.

* It is possible that a calculation error was made in the IR drop measurement such that the actual value should be 1/2 of that recorded, i.e. 25 mV. Unfortunately, the abnormally high value was not noticed until after the experimental series had been completed and time did not permit a rerun.

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RING

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SKETCH OF FLOATING ELECTRODE CELL

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ELECTRICAL SCHEMATIC OF
FLOATING ELECTRODE CELL TEST

FIGURE 14. FLOATING ELECTRODE CELL TEST SETUP

TABLE I

FLOATING ELECTRODE EVALUATION OF ANODE
PERFORMANCE IN CESIUM BICARBONATE

Electrode	PPF	SEC	A-3R	A-10R	PVC	AA-2	SSP
Catalyst Loading (mg cm^{-2})	15	4	20	13	1	10	10
Current Density (asf)	Overvoltage (mV)						
10	40	40	65	75	123	75	70
15	82	82	113	113	164	106	104
20	100	98	160	152	196	134	130
30	145	137	225	205	252	180	166
50	210	190	330	275	--	240	210

TABLE II

MATRIX THICKNESS TEST RESULTS

Thickness	CO_2 Efficiency	Voltage	IR Drop	Cathode	Anode
mils	Percent	Volts	mV	pH	pH
12.5	66.4	0.452	50	12.0	8.5
25	98	0.385	38	12.3	8.2
50	106	0.364	70	12.5	8.0

With the four layers of asbestos compressed to 50 mils it took about 30 hours to reach steady state conditions. The initial cell voltage was low and the pH high. Operation at 30 asf with the four layers resulted in a gradual increase in IR drop with time which increased the cell temperature. This test was discontinued when the ΔT between air inlet and outlet, normally $3.5^\circ F$, approached $8^\circ F$. At this time the IR drop was approaching 2 volts. The obvious cause of this behavior was that the electrolyte concentration at the anode was approaching zero under these conditions. Apparently, even under normal operating conditions with a matrix thickness of 25 mils the cesium ion concentration at the anode is significantly lower than had been anticipated. This information helps to explain two factors which were previously difficult to rationalize: the abnormally high anode polarization and the large anode electrokinetic effects.

The anode polarization measured in the analytical cell is typically one to two hundred millivolts greater than that measured in the floating electrode test even though the pH is the same (8.0) in each case. This discrepancy can be explained if the electrolyte concentration at the anode of a full cell is considerably lower than that in the floating electrode cell. This would result in additional intra-anode IR drop and concentration polarization for a full cell anode.

To determine the effect of electrolyte dilution on anode performance an electrode was tested in 9 weight percent cesium bicarbonate (58 weight percent diluted 10:1 by volume) and compared to the performance in 58 weight percent,* both solutions having a pH of 8. The dilute bicarbonate electrolyte gave 130 mv more anode polarization at 20 asf (corrected for IR drop) than the concentrated electrolyte. Thus, the unusually high cell voltage observed with one layer of matrix (12.5 mils) can be explained by a decrease in anode overvoltage due to the higher anolyte concentration obtained with

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* Evaluation of anode performance via the floating electrode test was done in 58 weight percent $CsHCO_3$ with a pH of 8.0.

the smaller inter-electrode distance. If a higher anolyte concentration could be maintained with a 25 or 30 mil electrode spacing, the cell voltage could be significantly improved without sacrificing the CO_2 transfer efficiency.

Two factors contribute to the overall anode polarization: intra anode IR drop, and intra anode concentration polarization.* The addition of an inert electrolyte such as cesium sulfate would decrease the intra anode IR drop but increase the concentration polarization so that the net effect on overall anode polarization is difficult to predict.

To determine the net effect of inert electrolyte addition, anode polarization was measured on a floating electrode in two electrolyte solutions: 0.5 mole $CsHCO_3$ and 0.5 mole $CsHCO_3$ + 1 mole Cs_2SO_4. With the inert electrolyte, the solution resistance was reduced by a factor of two and presumably there was a corresponding decrease in intra anode ᵀR drop. However, the total anode polarization at 20 asf was about 30 mv greater. From this result it seems that concentration polarization is the major factor limiting anode performance and the addition of an inert electrolyte would be expected to give a slightly lower cell power output.

The fact that electrokinetic phenomena seem to be more pronounced at the anode than at the cathode was difficult to understand, since it is well known that these effects on platinum are greatly enhanced by high solution pH, yet there has been no indication of cathode electrochemical degradation with time, as observed with the anode where the pH is low. In view of the evidence for a low electrolyte concentration at the anode it is now clear that the large anode electrokinetic effects must be related to the much greater zeta potential associated with the more dilute anolyte. Increasing the anolyte concentration by the addition of an inert electrolyte should suppress the zeta potential and thereby promote anode life by reducing the platinum and Teflon migration rates; however, some sacrifice in power would result due to the increase in intra anode concentration polarization.

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* Activation overvoltage would not normally be affected by electrolyte concentration changes when the major ions do not take part in the electrode reaction. Also, the activation overvoltage for hydrogen oxidation at normal HDC current densities should be negligible.

HDC ANALYTICAL TESTING

The purpose of the HDC analytical tests, described in Sections V and VII of the Master Test Plan, (reference Appendix) was to obtain more detailed information on the HDC cell to gain a better understanding of its operation. Such data are useful in the development of an HDC cell math model and in defining areas where improvements could be made to give better performance.

The analytical test cell illustrated by figure 15 was constructed from full cell pair hardware to give an electrode area of only $1/24 \, ft^2$ while keeping the full scale airflow path length of six inches. Reference electrodes were incorporated on both the anode and the cathode sides, and access ports were added to allow pH measurements at the anode or cathode during cell operation. The matrix consisted of one layer of Tissuquartz (18 mils) between two layers of fuel cell asbestos (20 mils each) compressed to 25 mils. All testing was done on the analytical test rig (figure 16) at an air temperature of $70°F$. The current density, air velocity, CO_2 partial pressure and air dew point were controlled at a given level. The more unusual parameters which were determined are listed below.

- Open circuit voltage

- IR drop

- Cathode overvoltage = cathode potential – cathode reference potential

- Anode overvoltage = anode potential – anode reference potential

- Anolyte and catholyte pH

- Concentration overvoltage and pH

The concentration overvoltage is equal to (open circuit voltage) – [(Cathode ref. – anode ref.) – (IR drop)]. The ΔpH is equal to concentration overvoltage divided by 0.06 volts.

The ΔpH value determined in this manner can be compared to the difference between the measured catholyte and anolyte pH values, and in most cases the correlation is good. (See Tables III and IV for Cs_2CO_3 electrolyte).
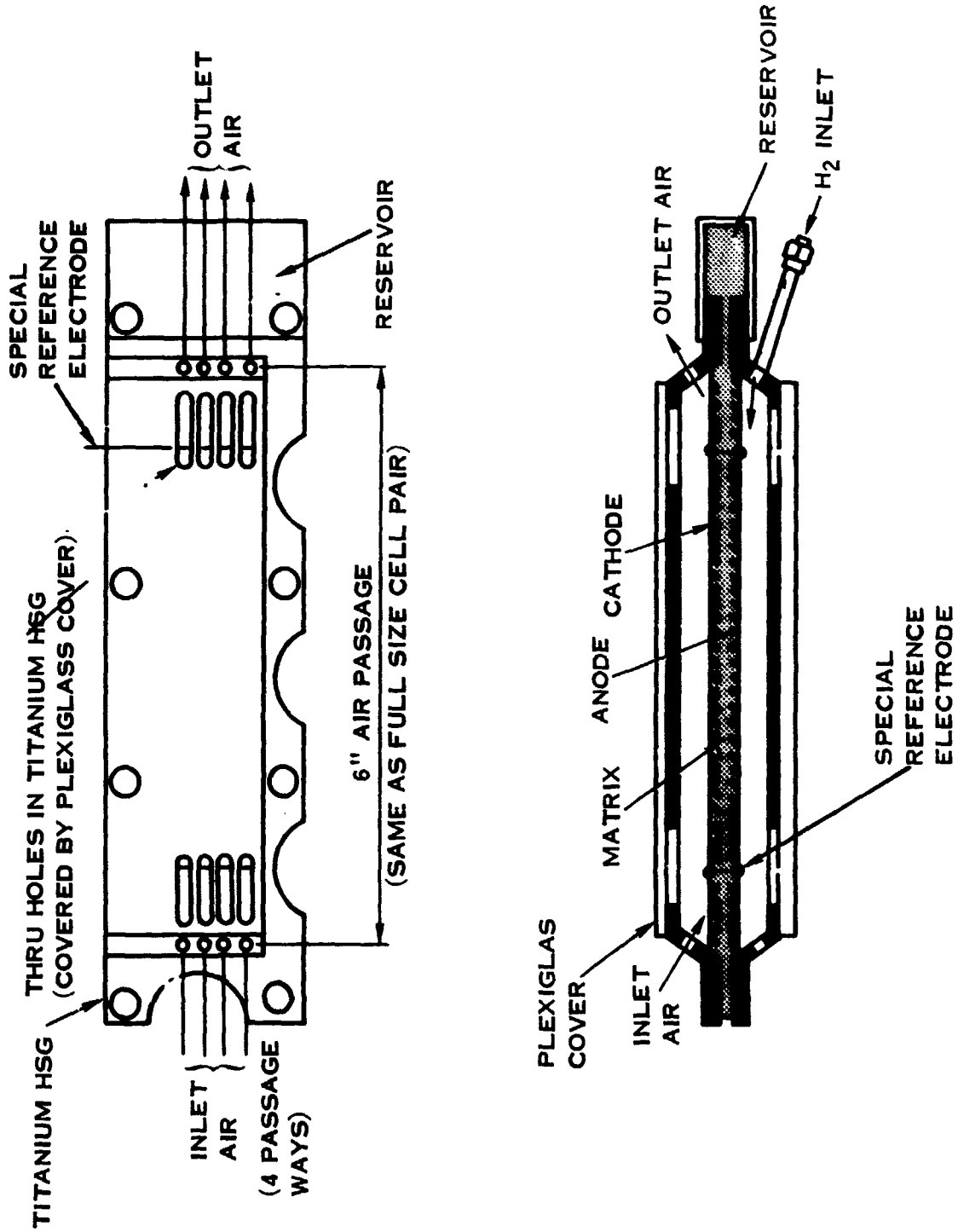
FIGURE 15. ANALYTICAL CELL
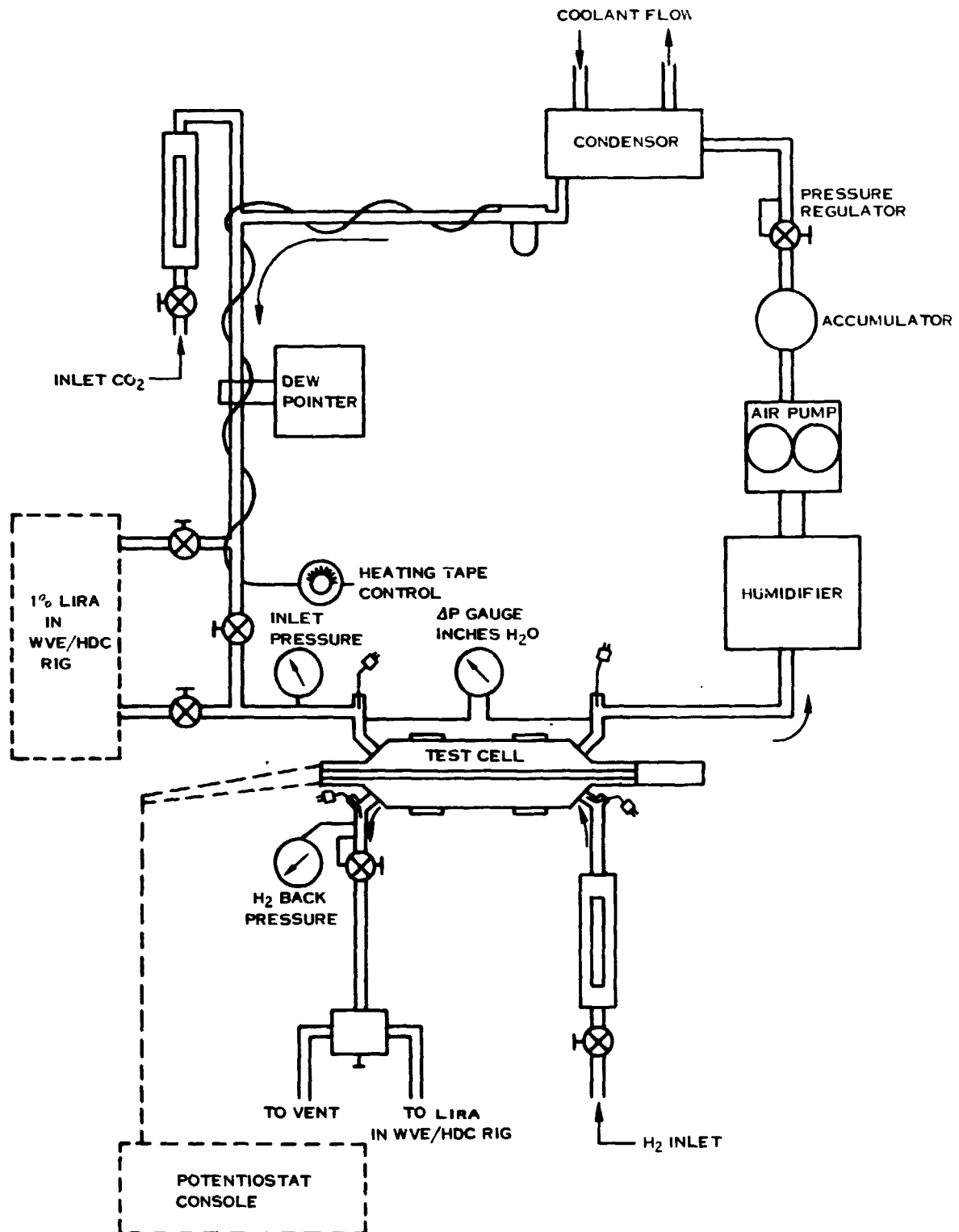
FIGURE 16. HDC ANALYTICAL TEST UNIT RIG

Cesium Carbonate Electrolyte

The analytical cell testing with Cs_2CO_3 electrolyte used the standard SSP electrodes as requested by the NASA-JSC and the air dew point was controlled at 63°F. The open circuit voltage was 1.122 volts and the electrolyte-matrix resistance, as calculated from the IR drop, was constant at 2.0 ± 0.1 milli ohms ft^2.

The anode overvoltage and cell voltage are not reported because, after a few weeks of operation, the anode performance decayed rapidly with time making these readings meaningless for the purpose at hand. The reported pH values were measured near the cell air exit. At low air flow velocities and low PCO_2 the pH values near the air inlet were somewhat lower due to significantly different gas phase mass transport rates at the extremes of the cell.

Table III shows the effect of CO_2 partial pressure and air velocity in the region where gas phase mass transport rates are important in determining the CO_2 processing efficiency. In this case increasing either the CO_2 partial pressure or the air velocity results in an increase in CO_2 processing efficiency, increased cathode polarization and a decrease in electrolyte pH. The increase in cathode polarization is a result of the decrease in catholyte pH. The results given in table IV also show this pH effect on cathode polarization and it is obvious that the pH effect predominates over the current density in determining the magnitude of the cathode overvoltage in this operating range.

TABLE III

EFFECT OF AIR VELOCITY AND CO_2 PRESSURE AT 18 ASF
WITH Cs_2CO_3 ELECTROLYTE

Air Vel. (ft/sec)	P_{CO_2} (mm Hg)	Efficiency %	Cathode pH	Anode pH	ΔpH	Cathode Overvoltage (mV)
5	0.25	12	14	11	3.1	-144
10	0.25	17	14	11.5	3.2	-153
20	0.25	18	13.8	11	3.0	-166
5	0.5	19	14	11	3.0	-160
10	0.5	22	13.5	10	3.0	-170
20	0.5	29	13.5	10	3.3	-185
5	1.0	33	13.5	11	2.5	-163
10	1.0	44	13.5	10	3.1	-172
20	1.0	51	13.5	9	4.5	-200

NOTE: Air Temperature 70°F, Dew Point 63°F.

TABLE IV
EFFECT OF CURRENT DENSITY AND CO_2 PRESSURE
AT AN AIR VELOCITY OF 7 FT/SEC

P_{CO_2} (mm Hg)	Current Density (asf)	Efficiency %	Cathode pH	Anode pH	ΔpH	Cathode Overvoltage (mV)
0.25	24	12	14	11.5	3.2	-152
0.25	12	20	13.3	11.3	2.1	-174
1.0	24	30	13.5	9.5	4.1	-169
1.0	12	42	13	9.5	3.3	-217
4.0	24	65	13	8.0	5.1	-195
4.0	12	75	12.3	7.5	4.8	-246
3.0	6	71	12.3	8.5	3.9	-245

NOTE: Air Temperature 70°F, Dew Point 63°F.

TMAC Electrolyte

The purpose of the TMAC electrolyte tests was to obtain more detailed information on cell operation with TMAC and to compare these results to those obtained with cesium carbonate electrolyte. Cell construction was identical to the Cs_2CO_3 analytical cell testing except for the electrodes which were PPF anode and DS 16-0 cathode.

Table V shows the effect of current density on cell performance. The decrease in cell voltage with increasing current density is due to an increase in the anode overvoltage (related to ΔpH) and IR drop. The cathode overvoltage decreases with increasing current density because of the increase in catholyte pH. Additional data on CO_2 transfer versus current density is given in figures 17 and 18. Although the CO_2 transfer efficiency decreases with increasing current density over the range studied (figure 17), the absolute amount of CO_2 processed increases as shown in figure 18.

TABLE V

EFFECT OF CURRENT DENSITY

$(P_{CO_2}$ = 1 mm Hg, RH = 49.3%, Air Velocity = 10 ft/sec)

Current Density (asf)	Cell Voltage (Volts)	Cathode pH	Anode pH	Overvoltage mV		Efficiency %	IR drop (mV)
				Cathode	Anode		
12	0.380	12.6	9.2	-226	249	80	60
18	0.348	12.6	9.2	- 91	358	55.6	100
30	0.284	13.0	9.2	- 49	430	36	110

Table VI shows the effect of air CO_2 partial pressure and relative humidity on cell performance. Increasing CO_2 partial pressures reduces cell voltage because of increased cathode overvoltage and IR drop which over-compensate the reduction in anode overvoltage. The decrease in anode overvoltage with increasing P_{CO_2} is not understood, since there seems to be no change in anolyte pH and the electrolyte resistance increase would indicate a decrease in anolyte concentration which has been shown to result in greater, not less, anode polarization. Admittedly the pH measurements are somewhat crude for this purpose and are that of the bulk anolyte rather than that at the catalyst surface which would determine the degree of anode polarization.

Increasing the air relative humidity increases the cell voltage because the reduction in cathode overvoltage and IR drop is greater than the increase in anode overvoltage and concentration polarization. With the more dilute electrolyte solution one would expect an increase in anode overvoltage, and a decrease in electrolyte resistance and anolyte pH as obtained. The lower cathode overvoltage observed with the more
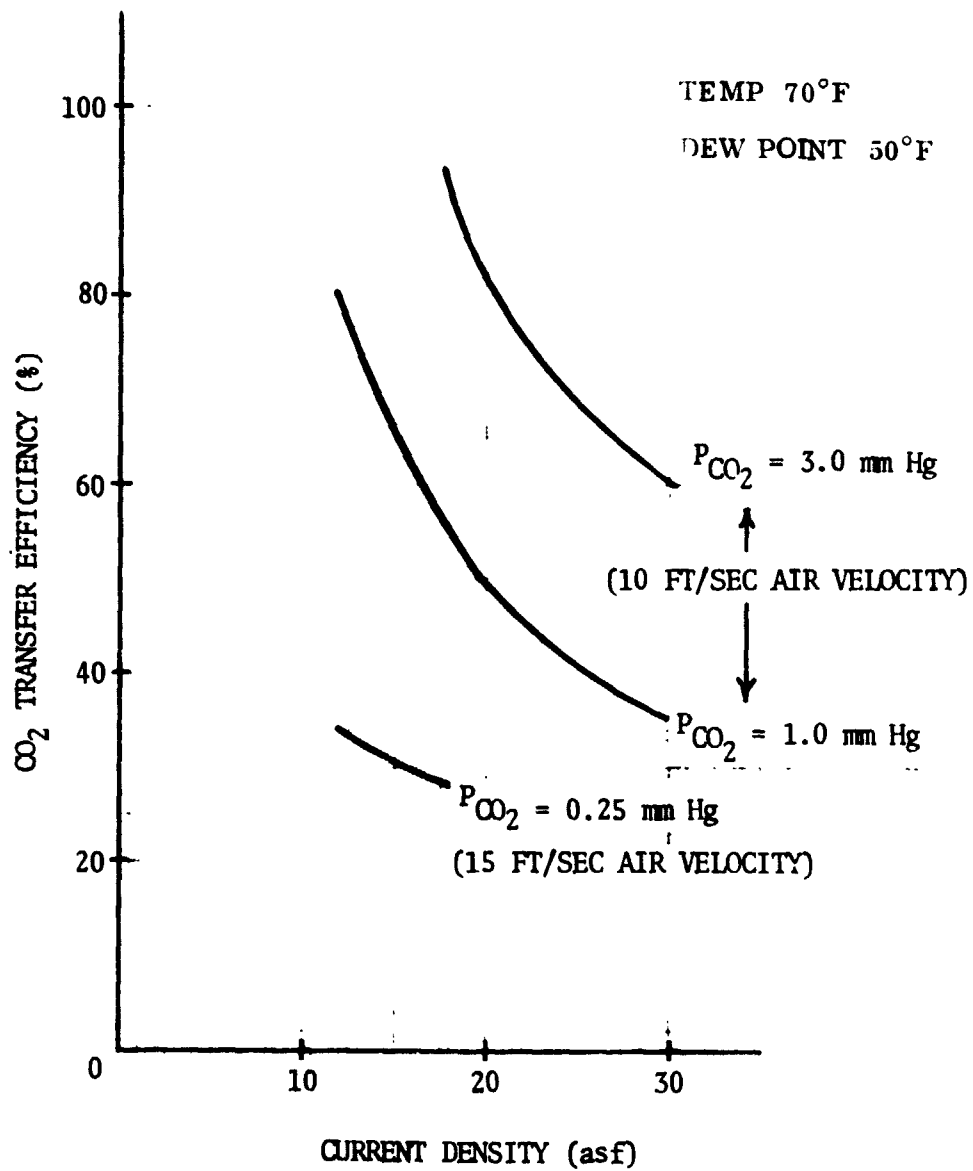
FIGURE 17. EFFECT OF CURRENT DENSITY AND P_{CO_2} ON CO_2 TRANSFER EFFICIENCY USING TMAC ELECTROLYTE
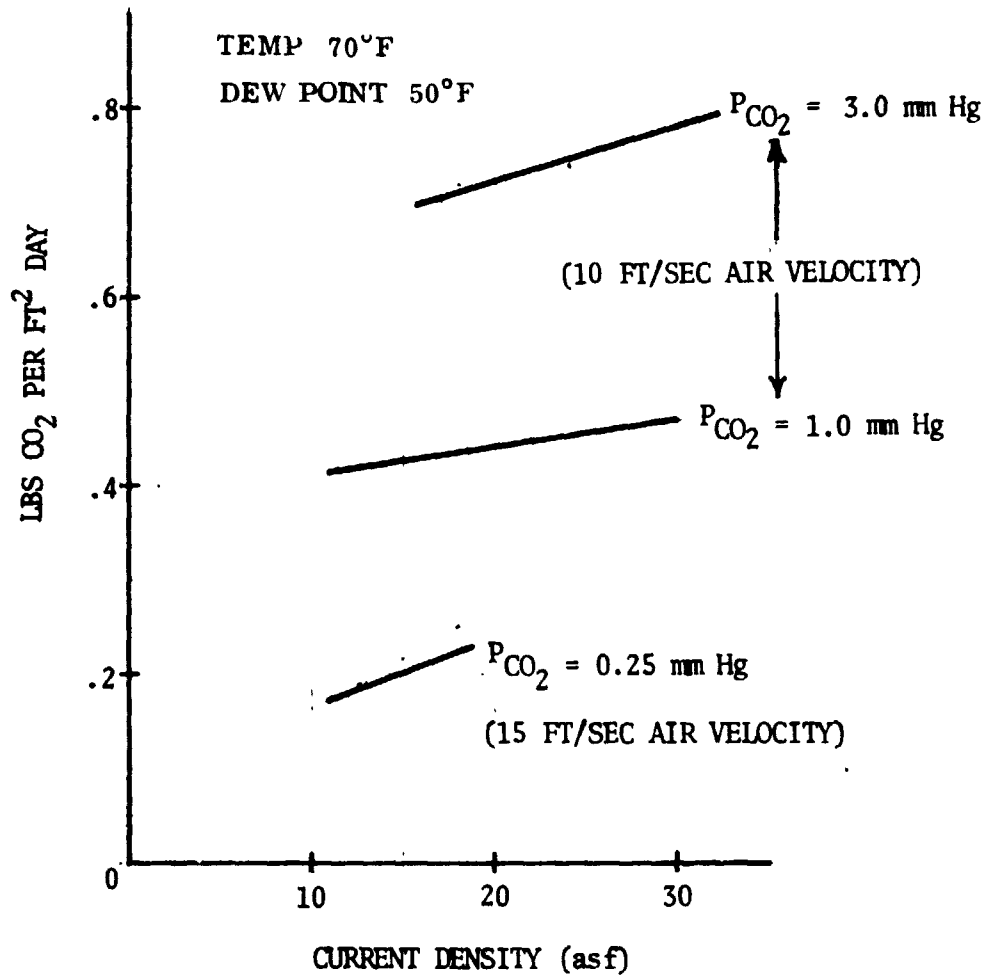
FIGURE 18. EFFECT OF CURRENT DENSITY AND P_{CO_2} ON THE ABSOLUTE

AMOUNT OF CO_2 TRANSFERRED WITH TMAC ELECTROLYTE

TABLE VI

EFFECT OF P_{CO_2} AND RELATIVE HUMIDITY
AT 18 ASF AND AT AN AIR VELOCITY OF 10 FT/SEC
WITH TMAC ELECTROLYTE (Air Temp = 70°F)

P_{CO_2} (mm Hg)	% R H (D. P.)	Cell Voltage (volts)	Cathode pH	Anode pH	Overvoltage (mV)		Efficiency (%)	IR drop (mV)
					Cathode	Anode		
0.25	33.6 (40°F)	0.354	13.5	10.5	- 46	415	19.0	100
1.0	33.6 (40°F)	0.246	13.3	10	-166	381	55.7	110
4.0	33.6 (40°F)	0.192	13.0	11	-294	368	92.4	120
0.25	71.2 (60°F)	0.387	13.5	9.2	-	-	19.6	65
1.0	71.2 (60°F)	0.274	13.3	9.2	-104	398	55.8	80
4.0	71.2 (60°F)	2.42	13.0	9.0	-150	383	97.8	90

TABLE VII

EFFECT OF AIR VELOCITY WITH TMAC ELECTROLYTE

(P_{CO_2} = 0.25 mm Hg, asf = 18, Relative Humidity = 49.3%, Temp 70°F)

Air Velocity (ft/sec)	Cell Voltage (volts)	Cathode pH	Anode pH	Overvoltage (mV)		Efficiency (%)	IR Drop (mV)
				Cathode	Anode		
5	0.423	13.5	10.5	- 36	375	18.0	80
10	0.370	13.5	10	-	-	19.3	100
15	0.364	13.0	10.5	- 98	395	28.8	90

dilute electrolyte (high air R.H.) is probably associated with a decrease in catholyte viscosity, since there is no change in catholyte pH. Air relative humidity (or electrolyte concentration) has little effect on CO_2 transfer efficiency.

Table VII shows the effect of air velocity, at ambient CO_2 levels, on cell performance. The decrease in cell voltage with increasing air velocity is caused by the lower cell temperature and the change in catholyte pH which is affected by the rate of CO_2 mass transport. At air velocities of 5 and 10 ft/sec, convective mass transport of CO_2 is definitely limiting as evidenced by the low CO_2 transfer efficiency.

Conclusions

The general conclusions from the results of the analytical cell testing are:

1. The cathodic overvoltage with both electrolytes is strongly affected by the catholyte pH.

2. For maximum CO_2 transfer efficiency the optimum catholyte pH for both electrolytes is in the range of 12.3 to 13.0. Within limits the optimum catholyte pH can be obtained by proper choice of air flow rate and current density.

3. The TMAC electrolyte results in a higher electrolyte resistance and anode polarization than Cs_2CO_3, and this accounts for the lower cell power output obtained with TMAC.

4. The anode overvoltage is a major contributor to low HDC cell power output and is the only source of cell power loss which offers hope for significant improvement in the future.

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IMPROVED CO$_2$ TRAN... ...R

Past experience has indicated that the overall CO$_2$ transfer process is controlled mainly by the reactions and mass transport processes at the cathode. The CO$_2$ must first be transported from the air stream to the gas-electrolyte interface within the cathode structure. Relatively high air velocities, approaching turbulent, are used to minimize the gas phase mass transport resistance. The next step is the absorption of the gaseous CO$_2$ into the catholyte as aqueous CO$_2$ followed by its reaction with hydroxide ions to form the bicarbonate ion. The subsequent, purely ionic, reaction to form the carbonate ion from hydroxide and bicarbonate ions is very rapid compared to the CO$_2$ (aq), OH$^-$ reaction. One can see from this sequence that the cathode structure should play an important role in determining the rate of these intra-cathode processes. The rate of gaseous CO$_2$ mass transport within the cathode structure would be determined by the hydrophobic gas pore geometry of the cathode. The absorption rate of gaseous CO$_2$ would be a function of the total available gas-catholyte interfacial area, and the CO$_2$, OH$^-$ reaction would be dependent on mass transport rates within the hydrophilic catalyst-matrix of the cathode.

To improve the overall CO$_2$ processing rate a catalyst for the CO$_2$ (aq), OH reaction was investigated and modifications of the cathode structure were made to increase the intra-cathode mass transport rates. The effect of electrolyte-matrix thickness (electrode spacing) on the overall CO$_2$ transfer rate also was determined.

Electrolyte Catalyst

Under certain conditions of cell operation it is possible for the reaction

$$CO_2 \text{ (aq)} \quad + \quad OH^- \quad \longrightarrow \quad HCO_3^-$$
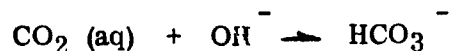
to be rate determining in the overall CO$_2$ transfer process, and under these conditions a catalyst for the above reaction would be beneficial. The most active known catalyst for this reaction is the enzyme carbonic anhydrase and it has been demonstrated qualitatively at Hamilton Standard that carbonic anhydrase at very low levels is effective in speeding up the absorption of CO$_2$ in both cesium carbonate and TMAC to form the respective bicarbonates. Also, potential sweeps showed no evidence of electrode poisoning or carbonic anhydrase instability over the potential range of 0 to 1200 m volts. However, long term stability and effectiveness in the HDC environment had not been evaluated.

Carbonic anhydrase was tested in the analytical test cell with Cs$_2$CO$_3$ electrolyte under the following conditions:

$$P_{CO_2} \quad = \quad 2.5 \text{ mm Hg}$$

Air velocity $\quad = \quad$ 7 ft/sec

Current density $\quad = \quad$ 12, 18 and 24 asf

Ambient pressure $=$ 14.7 psia

During a five day test with an SSP type cathode, there was no evidence of electrode poisoning from the carbonic anhydrase, however, there was no detectable improvement in the CO_2 transfer rate either. A repeat of this test using an American Cyanamide AA2 cathode, which gives a higher CO_2 transfer rate also gave a negative result. From this it can be concluded that either the CO_2, OH^- reaction is not rate controlling under normal operating conditions or that the carbonic anhydrase is not effective in the HDC environment. However, the fact that the CO_2 transfer rate varies with different cathode structures would indicate that intra-electrode mass transport, not the CO_2, OH^- reaction rate, is the controlling factor.

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Cathode Improvement and Evaluation

Since CO_2 is absorbed by the catholyte, an increase in catholyte volume and circulation rate would be expected to increase the CO_2 processing rate. Circulation of the catholyte within the cathode structure is determined by the local electroosmotic and capillarity pressure differentials. In the cathode, electrolyte tends to be pumped toward the air side in regions of high current density. In order for the capillary pressure differential to exceed the electroosmotic pressure, thus causing the electrolyte to be returned into the electrode structure, regions of low current density are required. It was reasoned that decreasing the density of the catalyst structure should increase its electronic resistance resulting in a significant drop-off in current density remote from the current collector screen. Thus, there would be a corresponding decrease in the electroosmotic pumping force while the capillary force would be independent of position from the current collector screen. Also microscopic examination and comparison of the SSP cathode with the American Cyanamid AA2 electrode, which gives a much higher CO_2 processing rate, showed that the AA2 electrode had a much less dense catalyst structure. The less dense electrode structure should also increase the intra-cathode $CO2$ gas mass transport rate.

Analytical Cell Test of DS 16-0 Cathode

The first approach was to modify the standard fuel cell electrode manufacturing process to achieve a less dense structure; however, the extent of this modification was limited and the CO_2 processing efficiency could only be increased from 50 percent to 60 percent. In discussion of our requirements with the Electrode Development Group at P&WA a different experimental electrode, was recommended. This electrode (DS 16-0), when used in the analytical cell with Cs_2CO_3 electrolyte gave a CO_2 processing efficiency of 98 percent at 18 asf and 3 mm Hg compared to an efficiency of 90 percent for the American Cyanamide AA2. Some parametric data for the DS 16-0 cathode obtained in the analytical cell is given in table VIII. The anode was a 15 mg/cm^2 PPF. Three additional Type DS 16-0 cathodes made for the analytical cell gave the same performance as the first DS 16-0 cathode. One of these cathodes was made with twice the platinum loading but showed no increase in CO_2 processing rate. Also, in an earlier test doubling the loading of the SSP type cathode did not affect the CO_2 processing rate. From these results it can be concluded that the catholyte volume is of minor importance compared to the catholyte circulation rate, assuming all the catalyst is being utilized.

Floating electrode tests of all the cathodes showed only small differences in electrochemical performance. The overvoltage at 20 asf was -250 ± 25 mV and there was a general trend toward increased overvoltage with decreasing catalyst density as would be expected.

TABLE VIII   ANALYTICAL CELL PARAMETRIC DATA WITH DS 16-0 TYPE CATHODE WITH Cs_2CO_3 ELECTROLYTE

Current Density (asf)	Efficiency (%)	Cell Voltage (Volts)	P_{CO_2} (mm Hg)	Air Velocity (ft/sec)	Air Dew Point(°F)	Cathode pH	Anode pH
12	82	0.416	3	10	64	12.3	8.0
18	98	0.400	3	10	62	12.5	8.2
24	92	0.290	3	10	63	12.7	8.3
30	84	0.231	3	15	65	12.5	8.2
36	75	0.183	3	15	65	12.3	8.2
42	68	0.110	3	15	65	12.5	8.2
54	53	0.050	3	15	63	-	-
18	97	0.374	3	10	62	12.2	8.2
18	63.4	0.364	1	10	65	13	8.2
18	24	0.365	0.25	15	63	>13.5	8.7

Note: Air inlet temperature was 70°F for all tests.

Full Size Cell Test of DS 16-0 Type Cathode

HDC cell pair S/N 019 operated for 3360 hours using Cs_2CO_3 electrolyte prior to its disassembly to incorporate DS 16-0 cathodes. During this time the degradation in cell power was approximately 20 micro volts per hour. The average CO_2 removal rate for the unit at 18 asf was 70 scc/min or a 52 percent current efficiency. After the reassembly, which incorporated the same PPF anodes, a new matrix and DS 16-0 cathodes, this cell pair (now S/N 019-2) was placed on test at an air inlet condition of 3 mm Hg P_{CO_2}, 52°F/45°F temperature/dew point and operated at 18 asf. The current efficiency was approximately 74 percent. Since the full size cell pair performance did not agree with the analytical cell performance (74 percent vs 98 percent), cell pair S/N 019-2 was disassembled and a section of the top cathode was removed and tested in the analytical cell. This testing revealed a current efficiency of 92 percent, which indicated a loss of 6 percent in current efficiency when scaling up to a large size electrode. Cell pair S/N 019-2 was reassembled with new matrix, the same electrodes and with new "O"-Seals on the hydrogen manifold block because it was suspected that the original seals may have leaked. Test results revealed a current efficiency of 86 percent after three days of testing* (figure 9).

The difference in CO_2 transfer efficiency between the full size cell pair and the analytical cell was investigated. It was established that differences in hydrogen back pressure, temperature, hydrogen velocity (Table IX), and housing resistance have little effec on the CO_2 transfer efficiency. Also the possibility of a hydrogen maldistribution in the full size cell pair was eliminated by reducing the hydrogen pressure drop and sealing the crossover points as shown in figure 19. The only apparent difference between the two cells is the partial pressure of CO_2 in the hydrogen gas coming out of the cells. The analytical cell has typically less than 1 percent CO_2 whereas the full size cell has greater than 5 percent CO_2. Changing the hydrogen flow rate in the full size cell to vary the percentage of CO_2 from 5 to 20 had no effect on CO_2 transfer efficiency (figure 43), and likewise the hydrogen flow rate in the analytical cell was varied from 0.1 to 1 percent CO_2 without affecting the CO_2 transfer efficiency (Table IX). A cell pair operated with a modified center housing to allow sampling of the hydrogen – carbon dioxide gas at various points showed no variation in the CO_2 transfer efficiency across the cell.

The discrepancy in CO_2 transfer efficiency between the full size cell pair and the analytical cell still has not been resolved.

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* Other full size cell pairs with Cs_2CO_3 electrolyte subsequently tested with DS 16-0 cathodes under the same conditions gave CO_2 transfer efficiencies varying from 82 to 92 percent.

TABLE IX   EFFECT OF HYDROGEN BACK PRESSURE, FLOW RATE
AND AIR TEMPERATURE ON CO_2 TRANSFER

	Cell S/N 019 Data		
H_2 Back Pressure (psia)	14.7	19.7	
CO_2 Transfer (scc/min)	59.7	59.0	
Air Temperature	52°F	70°F	
CO_2 Transfer (scc/min)	96.5	96.5	
			Analytical Cell Data
CO_2/H_2 Ratio Out * (x 10^3)	1.1	3	9.8
CO_2 Transfer (scc/min)	5.2	5.41	5.36

* Varied by changing H_2 flow rate.

A. BEFORE MODIFICATION TO CENTER HOUSING

H₂ FLOW GOES TO BOTTOM CELL · H₂ INLET INTO TOP CELL
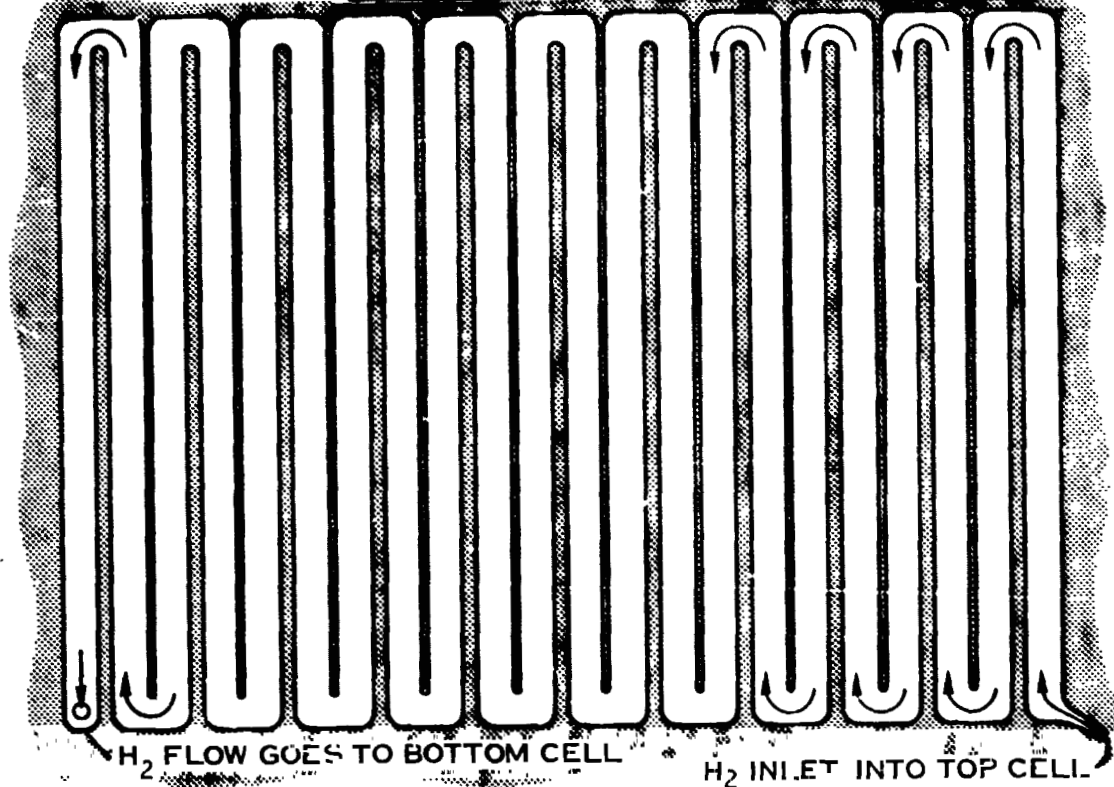
AFTER MODIFICATION TO CENTER HOUSING
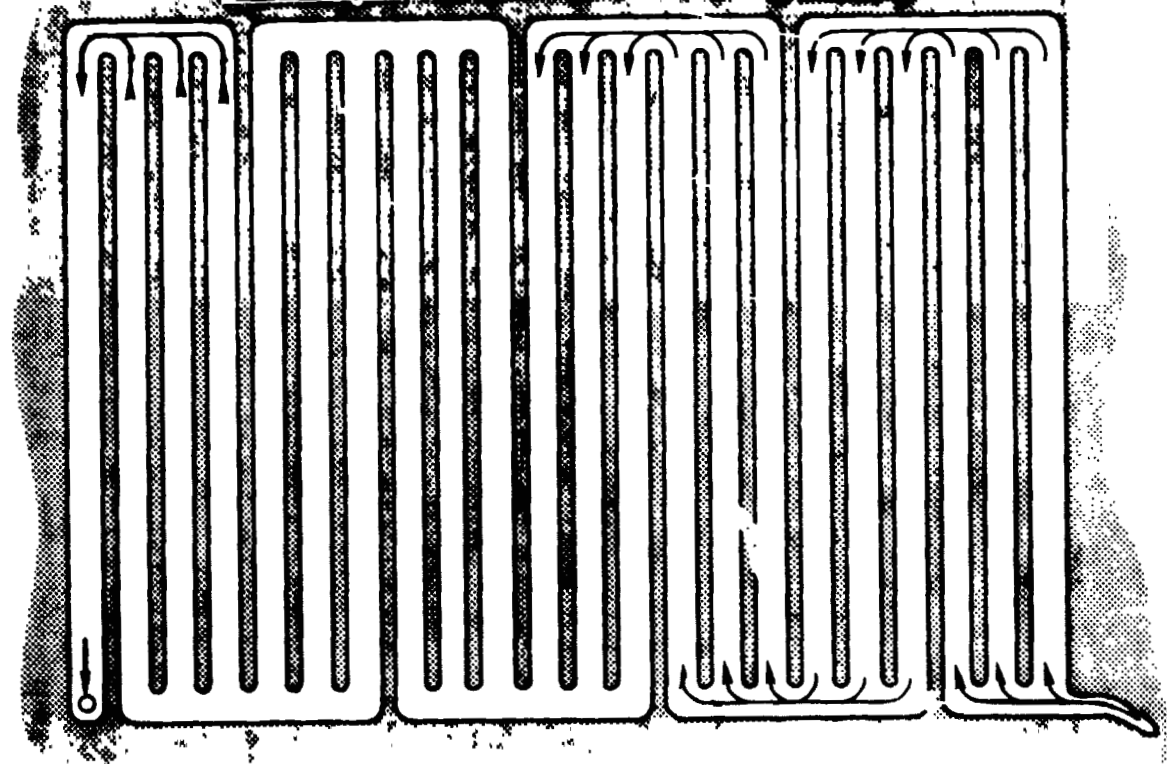
FIGURE 19. MODIFICATION TO CENTER HOUSING TO REDUCE H₂ FLOW Δ P

HDC ELECTROLYTE PROPERTIES

The physical properties of cesium bicarbonate and tetramethylammonium carbonate (TMAC) electrolyte were determined in order to provide the necessary design data for the HDC cell. The properties of cesium carbonate, except for viscosity, were determined under the previous contract (NAS 9-11830) but are included here for convenience. The viscosity data for TMAC, measured by Dr. C.H. Lin at the NASA-JSC, also is included. The data is presented in the following order:

Cesium Carbonate (Cs_2CO_3)

Table X	Vapor pressure versus concentration and temperature
Table XI	Specific conductivity versus concentration and temperature
Figure 20	Solubility versus temperature
Figure 21	Density versus concentration
Table XII	Viscosity versus concentration
Figure 22	Specific volume versus concentration
Figure 23	Moles per liter versus weight percent

Cesium Bicarbonate ($CsHCO_3$)

Table XIII	Vapor pressure versus concentration and temperature
Table XIV	Specific conductivity versus concentration and temperature
Figure 24	Solubility versus temperature
Figure 25	Density versus concentration
Table XV	Viscosity versus concentration
Figure 26	Specific volume versus concentration
Figure 27	Moles per liter versus weight percent
Table XVI	Relative humidity versus concentration and temperature

Tetramethylammonium Carbonate (TMAC)

Table XVII	Vapor pressure versus concentration and temperature
Table XVIII	Specific conductivity versus concentration and temperature
Figure 28	Density versus concentration

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TABLE X

VAPOR PRESSURE OF H_2O (in mm Hg) FOR Cs_2CO_3
AT VARIOUS CONCENTRATIONS AND TEMPERATURES

Cs_2CO_3 CONCENTRATION (WT - %)	Cs_2CO_3 TEMPERATURE -(°F)											
	45	50	55	60	65	70	75	80	85	90	95	100
0	7.7	9.22	11.1	13.25	15.77	18.76	22.2	26.1	30.8	36.2	42.2	49.8
40	7.1	8.5	10.2	12.3	14.4	17.3	20.6	24.2	28.8	33.7	39.3	46.0
50	6.4	7.7	9.25	11.2	13.3	15.7	18.7	22.2	26.1	30.6	35.7	41.8
60	5.52	6.65	7.95	9.55	11.3	13.5	16.2	19.0	22.2	26.2	30.7	36.0
65	4.83	5.8	6.95	8.36	10.0	11.7	14.0	16.5	19.5	23.0	27.0	31.5
70	4.02	4.82	5.75	6.88	8.22	9.8	11.5	13.6	16.2	19.0	22.1	25.8
73	3.42	4.11	4.9	5.88	7.0	8.3	9.84	11.5	13.5	16.0	18.6	21.8
75	2.83	3.39	4.02	4.82	5.75	6.82	8.03	9.45	11.1	13.0	15.2	17.7
77	2.38	2.84	3.37	4.02	4.8	5.7	6.68	7.85	9.23	10.7	12.6	14.6
78.5	2.03	2.43	2.88	3.46	4.12	4.88	5.72	6.74	7.92	9.25	10.7	12.5
80	1.75	2.09	2.49	2.97	3.56	4.16	4.87	5.72	6.73	7.9	9.2	10.6

TABLE XI

SPECIFIC CONDUCTANCE OF Cs_2CO_3 (OHM CM)$^{-1}$
AT VARIOUS CONCENTRATIONS AND TEMPERATURES

Cs_2CO_3 Concentration (Wt-%)	Cs_2CO_3 Temperature (°F)											
	45	50	55	60	65	70	75	80	85	90	95	100
40	0.144	0.154	0.163	0.173	0.183	0.192	0.202	0.211	0.221	0.230	0.240	0.249
45	0.161	0.172	0.182	0.192	0.202	0.213	0.223	0.233	0.243	0.253	0.263	0.274
50	0.170	0.180	0.191	0.201	0.212	0.222	0.233	0.243	0.253	0.264	0.274	0.285
55	0.171	0.181	0.192	0.203	0.213	0.224	0.234	0.245	0.255	0.266	0.276	0.287
60	0.165	0.175	0.185	0.196	0.206	0.217	0.227	0.238	0.248	0.258	0.269	0.279
62.5	0.158	0.168	0.178	0.189	0.199	0.210	0.219	0.230	0.241	0.250	0.261	0.270
65	0.148	0.158	0.168	0.178	0.188	0.198	0.208	0.218	0.229	0.239	0.249	0.258
68	0.130	0.137	0.149	0.159	0.169	0.178	0.191	0.200	0.211	0.221	0.231	0.241
70	0.113	0.123	0.134	0.144	0.154	0.164	0.175	0.185	0.195	0.205	0.215	0.226
71	0.104	0.115	0.126	0.136	0.146	0.156	0.167	0.177	0.187	0.197	0.207	0.217
72			0.118	0.128	0.138	0.148	0.157	0.166	0.177	0.186	0.196	0.207
73						0.138	0.147	0.155	0.166	0.175	0.185	0.194
74								0.144	0.154	0.163	0.173	0.183
75										0.150	0.160	0.170
76												0.152

Cs_2CO_3 SOLUBILITY LIMIT

USE LAST NUMBER IF LIMIT IS EXCEEDED
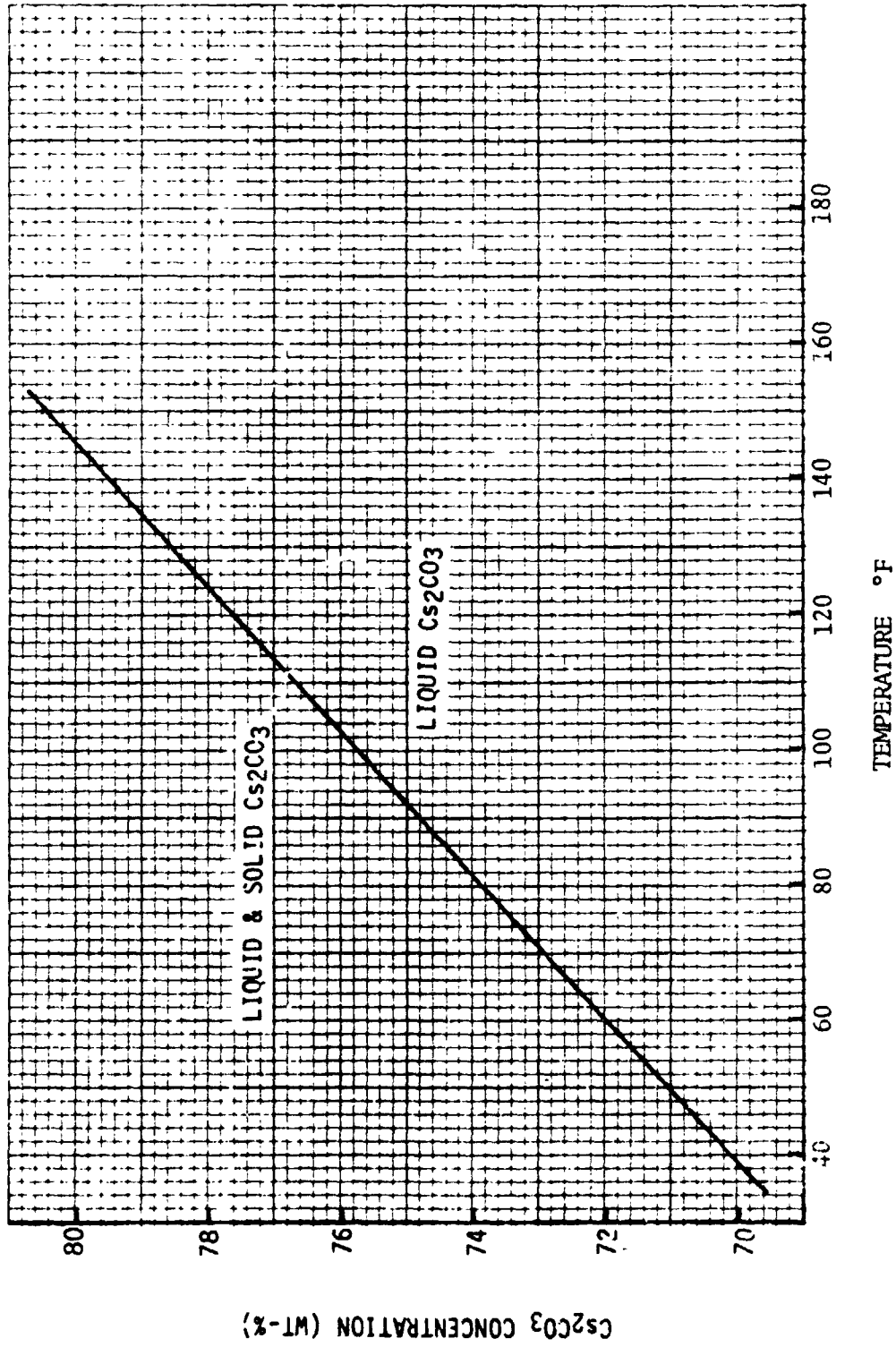
FIGURE 20.   SOLUBILITY LIMIT FOR Cs_2CO_3 VS TEMPERATURE
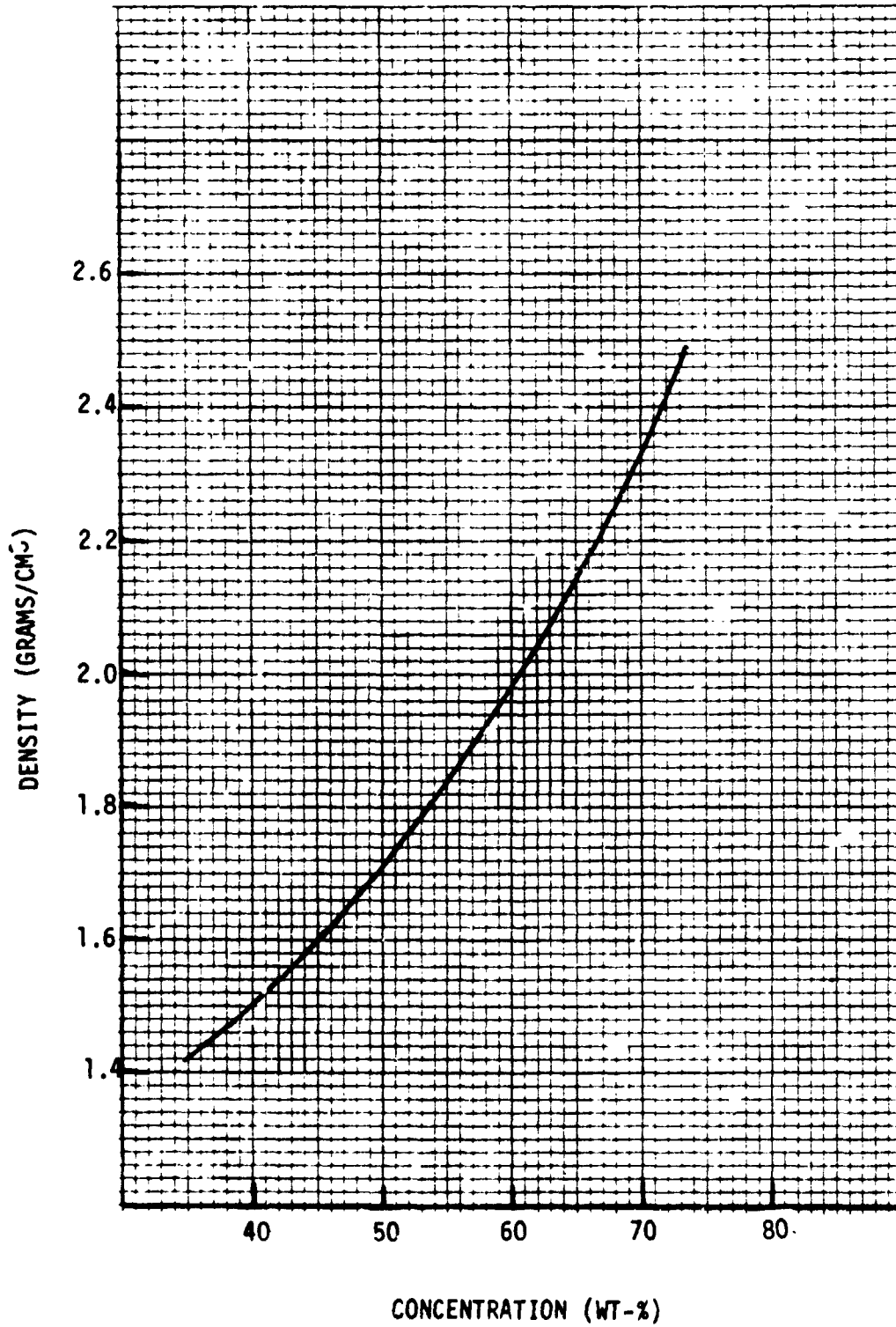
FIGURE 21. Cs_2CO_3 DENSITY VS CONCENTRATION AT 25°C

TABLE XII VISCOSITY OF CESIUM CARBONATE SOLUTION

CESIUM CARBONATE SOLUTION			
CONCENTRATION	DENSITY	ABS. VISCOSITY	KIN. VISCOSITY
Wt. %	Grams/cc	Centipoise	Centistokes
70	2.194	5.68	2.59
65	2.034	3.87	1.90
60	1.897	2.97	1.56
55	1.772	2.38	1.34
50	1.663	2.00	1.20
40	1.476	1.56	1.06
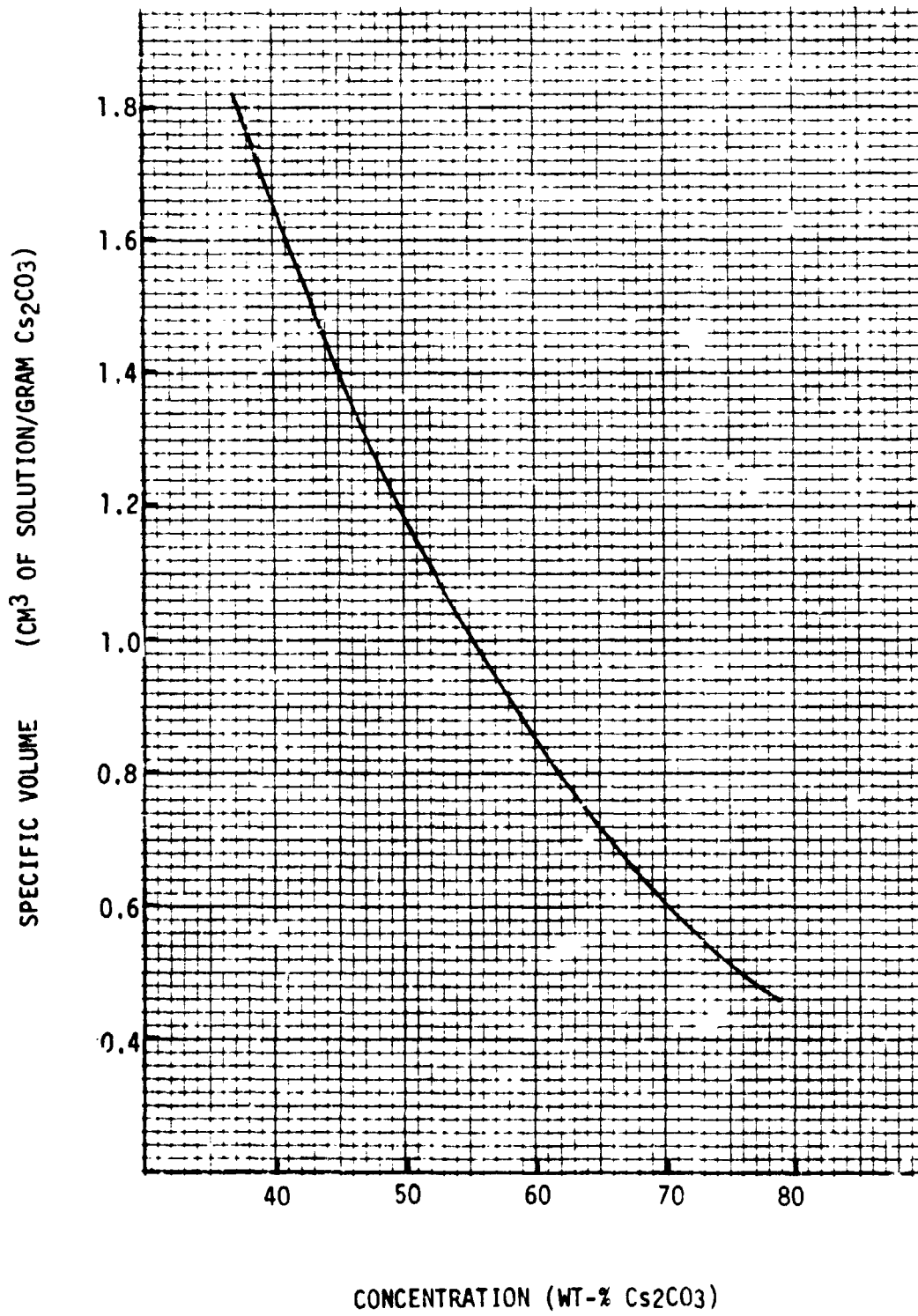
FIGURE 22. Cs$_2$CO$_3$ SPECIFIC VOLUME VS CONCENTRATION

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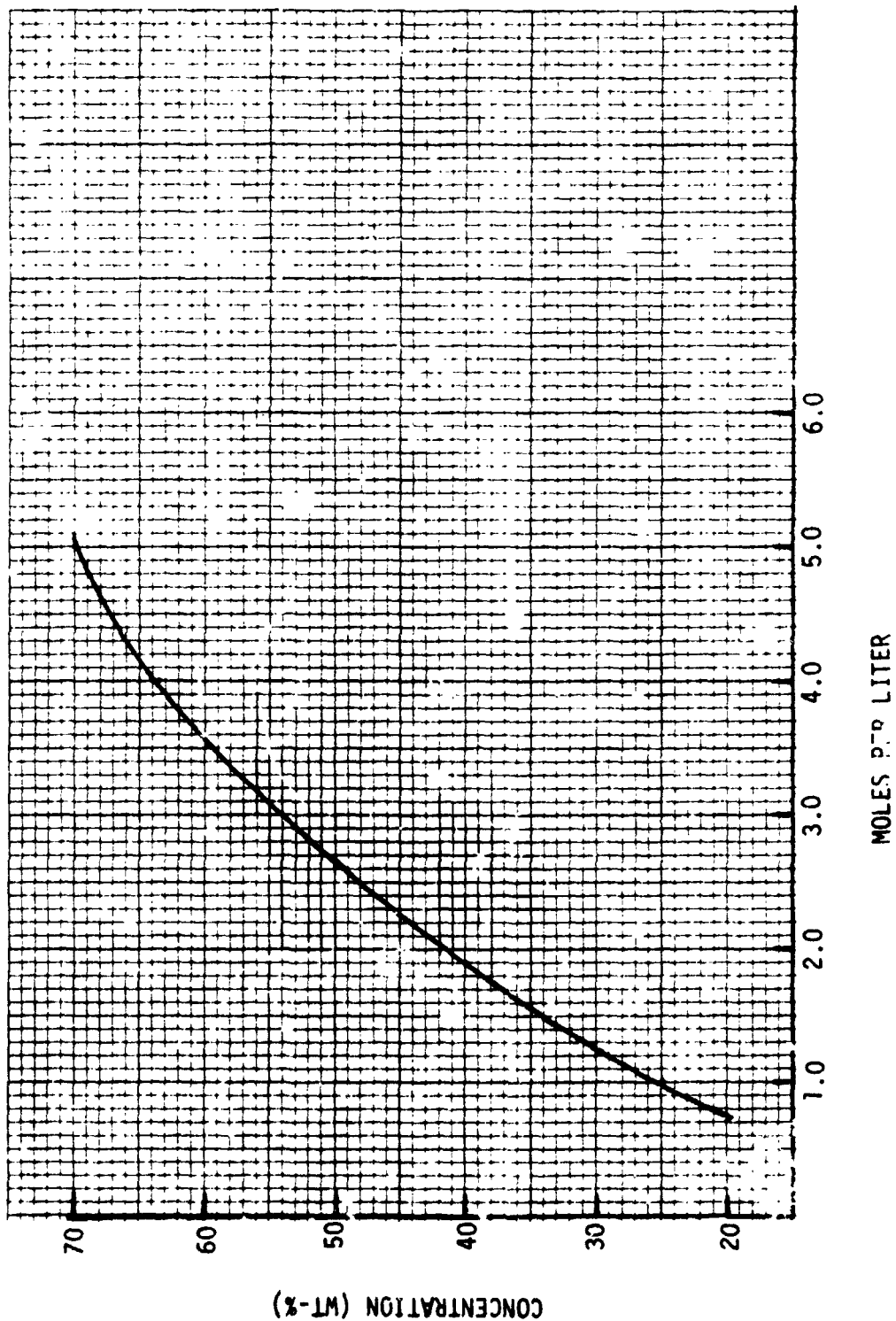
FIGURE 24. Cs$_2$CO$_3$ CONCENTRATION VS MOLE/LITER

CONCENTRATION (WT-%)

MOLES PER LITER

TABLE XIII   WATER VAPOR PRESSURE (mm Hg) OF CESIUM BICARBONATE SOLUTIONS
(pH = 8.0, P_{CO_2} = 1 atm.)

°F / Wt %	40	45	50	55	60	65	70	75	80	85
0	6.29	7.63	9.21	11.07	13.25	15.80	18.78	22.23	26.21	30.81
20	6.24	7.48	8.96	10.64	12.62	14.98	17.52	20.51	23.96	27.92
25	6.22	7.44	8.88	10.52	12.43	14.65	17.24	20.20	23.59	27.46
30	6.17	7.38	8.78	10.38	12.25	14.44	16.97	19.89	23.22	26.90
35	6.12	7.29	8.68	10.25	12.06	14.22	16.69	19.56	22.83	26.56
40	6.07	7.22	8.57	10.11	11.87	13.97	16.41	19.20	22.44	26.10
45	5.99	7.11	8.44	9.95	11.70	13.75	16.13	18.82	21.99	25.54
50	5.87	6.97	8.26	9.75	11.46	13.46	15.75	18.38	22.42	24.87
55	5.68	6.75	8.01	9.45	11.12	13.05	15.26	17.80	20.76	24.07
60	5.43	6.46	7.69	9.07	10.67	12.55	14.65	17.09	1C.89	23.05
65	5.11	6.09	7.25	8.56	10.07	11.84	13.84	16.14	18.77	21.72
70	4.69	5.60	6.66	7.87	9.29	10.89	12.77	14.87	17.30	20.04

TABLE XIV   SPECIFIC CONDUCTIVITY OF CESIUM BICARBONATE
(ohm cm)$^{-1}$ at pH = 8

Wt % \ °F	40	45	50	55	60	65	70	75	80	85
20	.0613	.0658	.0700	.0745	.0788	.0830	.0874	.0918	.0960	.1004
25	.0975	.0825	.0873	.0923	.0972	.1024	.1073	.1123	.1172	.1226
30	.0934	.0990	.1050	.1140	.1160	.1220	.1275	.1332	.1390	.1446
35	.1086	.1150	.1213	.1280	.1345	.1408	.1470	.1530	.1595	.1660
40	.1205	.1280	.1350	.1425	.1500	.1570	.1642	.1714	.1788	.1860
45	.1320	.1400	.1478	.1557	.1635	.1715	.1794	.1872	.1952	.2030
50	.1410	.1496	.1583	.1670	.1757	.1843	.1930	.2018	.2105	.2193
55	.1490	.1576	.1663	.1750	.1840	.1925	.2012	.2100	.2188	.2275
60	.1490	.1576	.1663	.1750	.1840	.1925	.2012	.2100	.2188	.2275
65	.1322	.1420	.1510	.1598	.1685	.1771	.1860	.1946	.2035	.2122
70	.1170	.1258	.1344	.1432	.1520	.1606	.1694	.1780	.1868	.1954
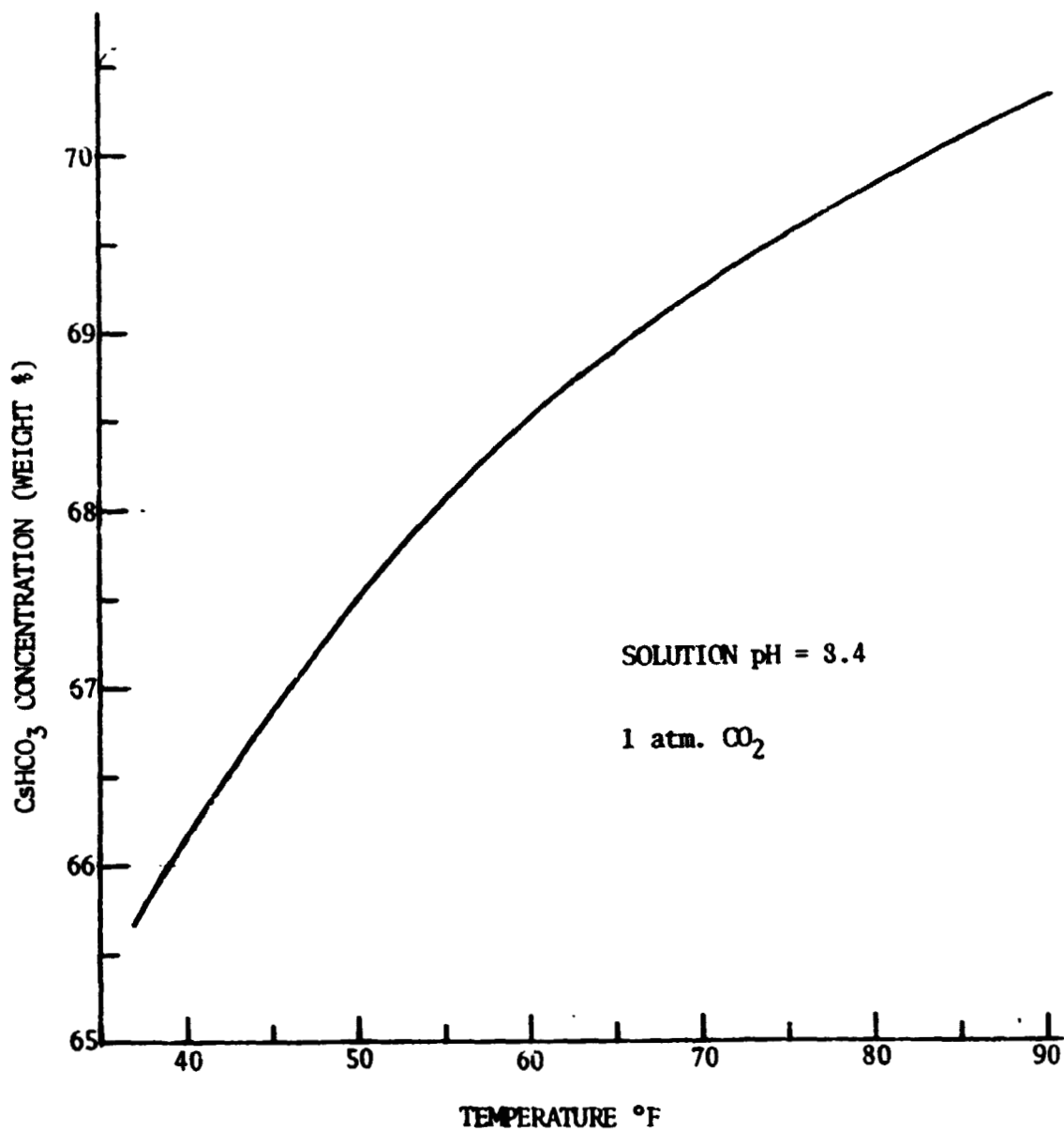
FIGURE 24. SOLUBILITY OF CESIUM BICARBONATE VS TEMPERATURE
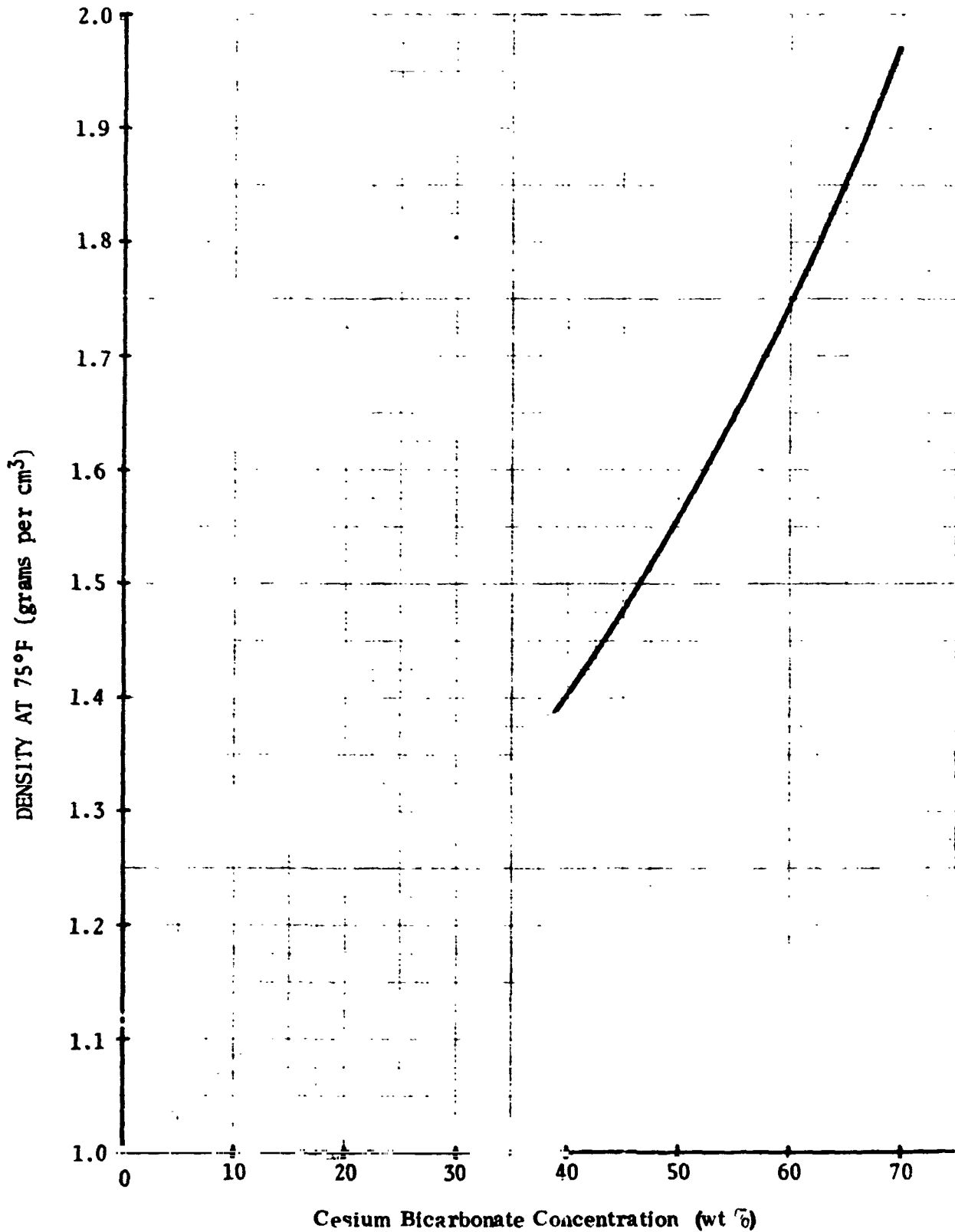
FIGURE 25.   DENSITY OF CESIUM BICARBONATE SOLUTIONS
VS CONCENTRATION AT 75°F

TABLE XV   VISCOSITY OF CESIUM BICARBONATE SOLUTIONS

CESIUM BICARBONATE SOLUTION*			
CONCENTRATION	DENSITY	ABS. VISCOSITY	KIN. VISCOSITY
Wt %	Grams/cc	Centipoise	Centistokes
69.4	2.006	3.92	1.955
65	1.892	3.00	1.59
60	1.774	2.38	1.34
55	1.672	1.99	1.19
50	1.580	1.77	1.12
40	1.421	1.44	1.01

* 1 atm. CO_2, 73°F
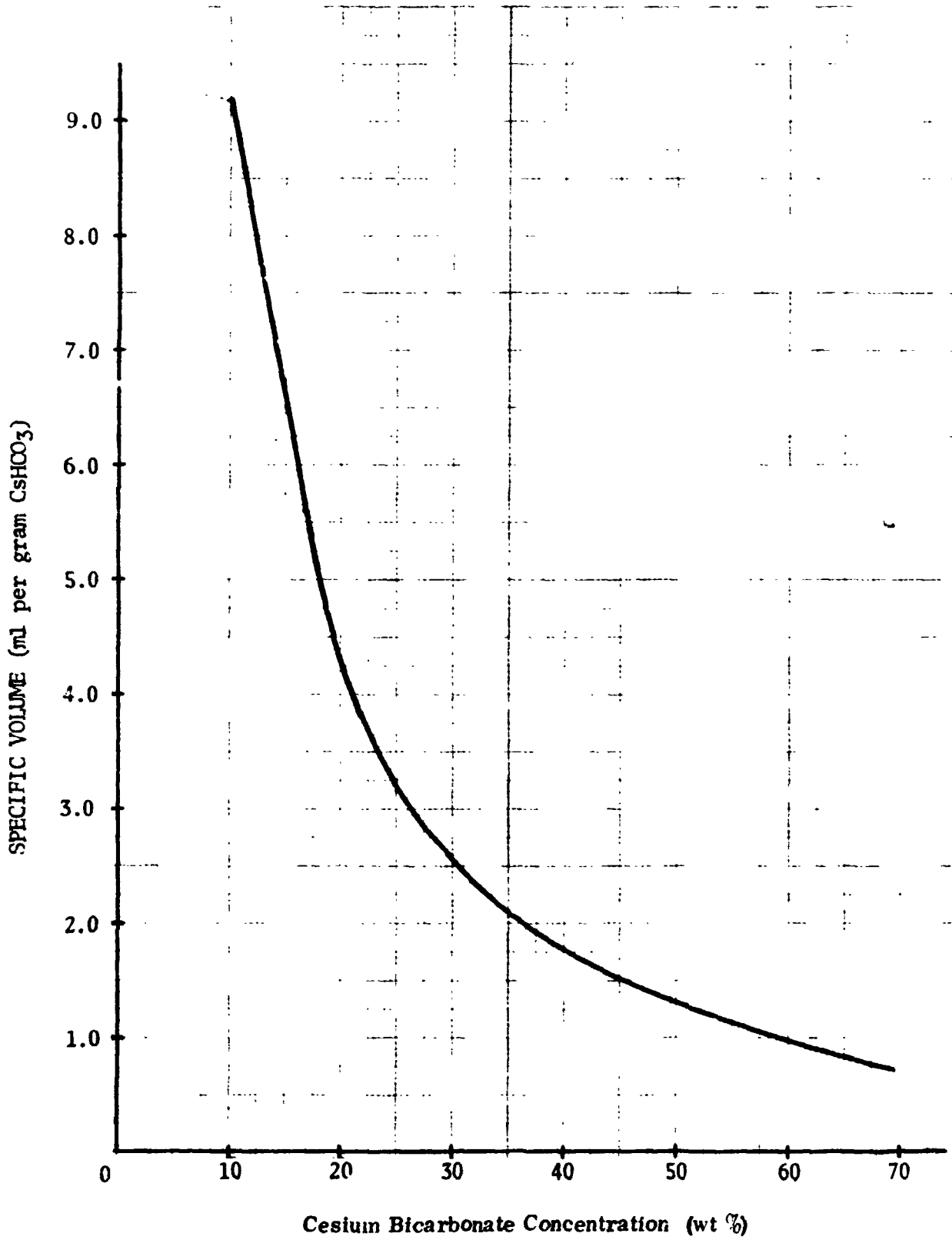
FIGURE 26.   SPECIFIC VOLUME OF CESIUM BICARBONATE SOLUTIONS
VS CONCENTRATION
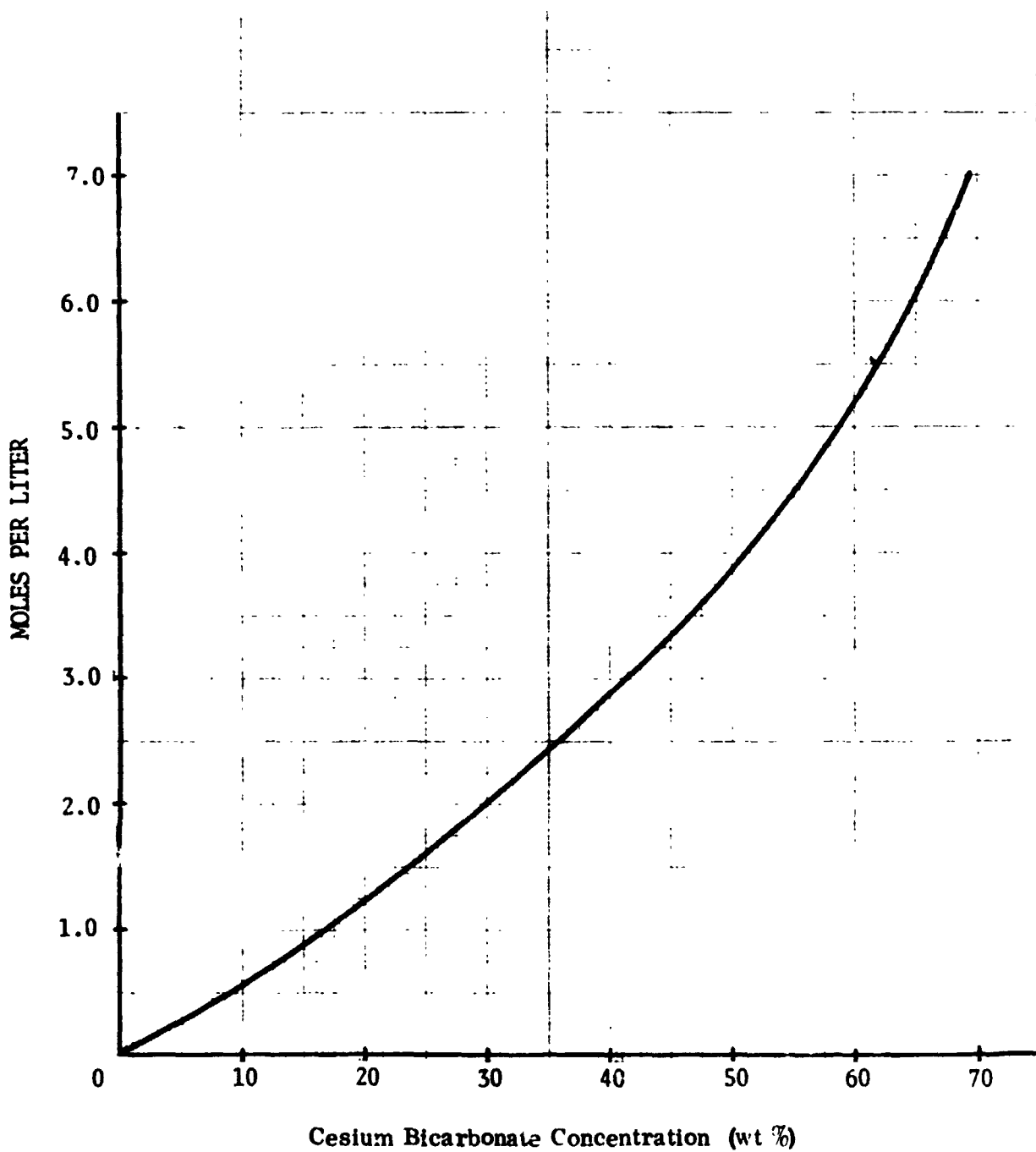
FIGURE 27. MOLES PER LITER VERSUS PERCENT CESIUM BICARBONATE

TABLE XVI   RELATIVE HUMIDITY OF CESIUM BICARBONATE SOLUTIONS (PERCENT) (pH = 8.0, P_{CO_2} = 1 atm.)

°F \ Wt%	40	45	50	55	60	65	70	75	80	85
20	99.2	98.1	97.5	96.1	95.2	94.2	93.3	92.3	91.4	90.6
25	98.8	9?.?	96.4	95.0	93.8	92.8	91.8	90.9	90.0	89.1
30	98.1	96.7	95.3	93.8	92.4	91.35	90.4	89.5	88.6	87.3
35	97.3	95.6	94.2	92.6	91.0	90.0	88.9	88.0	87.1	86.2
40	96.4	94.6	93.0	91.3	89.6	88.4	87.4	86.4	85.6	84.7
45	95.2	93.2	91.6	89.9	88.3	87.0	85.8	84.7	83.9	82.9
50	93.2	91.4	89.7	88.1	86.5	85.16	83.9	82.7	81.7	80.7
55	90.2	88.5	87.0	85.4	83.9	82.6	81.3	80.1	79.2	78.1
60	86.3	84.7	83.5	81.95	80.5	79.4	78.0	76.9	75.9	74.8
65	81.2	79.8	78.7	77.3	76.0	74.9	73.7	72.6	71.6	70.5
70	74.6	73.4	72.3	71.1	70.1	68.9	68.0	66.9	66.0	65.06

TABLE XVII WATER VAPOR PRESSURE OF TMAC VERSUS CONCENTRATION AND TEMPERATURE

WATER VAPOR PRESSURE (mmHg) OF TMAC pH - 12.5

Wt% \ °F	55	60	65	70	75	80	85	90	% RH
0	11.09	13.29	15.77	18.76	22.24	26.27	30.75	36.07	100
10	10.77	12.91	15.32	18.23	21.61	25.55	29.87	35.02	97.1
15	10.54	12.62	14.98	17.83	21.13	24.98	29.20	34.27	95.0
20	10.14	12.14	14.41	17.15	20.33	24.02	28.10	32.97	91.4
25	9.44	11.31	13.42	15.96	18.93	22.37	26.18	30.70	85.1
30	8.47	10.14	12.04	14.33	16.99	20.07	23.48	27.56	76.4
35	7.32	8.76	10.41	12.38	14.68	17.36	20.28	23.81	66.0
40	6.11	7.34	8.71	10.36	12.27	14.52	16.98	19.87	55.1
45	4.71	5.65	6.70	7.98	9.45	11.17	13.07	15.33	42.5
50	3.60	4.32	5.13	6.09	7.23	8.55	9.99	11.72	32.5

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TABLE XVIII   SPECIFIC CONDUCTIVITY OF TMAC VERSUS CONCENTRATION AND TEMPERATURE

SPECIFIC CONDUCTIVITY OF TMAC $(ohm\ cm)^{-1}$ AT pH 12.5

°F \ Wt%	10	15	20	25	30	35	40	45	50	55
55	.404	.508	.561	.548	.491	.410	.305	.196	.104	.048
60	.440	.553	.609	.594	.538	.454	.345	.233	.132	.064
65	.475	.597	.656	.640	.585	.498	.386	.270	.159	.077
70	.512	.642	.703	.686	.630	.542	.427	.307	.187	.092
75	.547	.687	.750	.731	.675	.585	.467	.343	.215	.109
80	.583	.730	.797	.776	.721	.628	.508	.379	.243	.122
85	.624	.775	.844	.822	.767	.672	.548	.416	.270	.138
90	.655	.819	.891	.867	.813	.716	.589	.452	.298	.158

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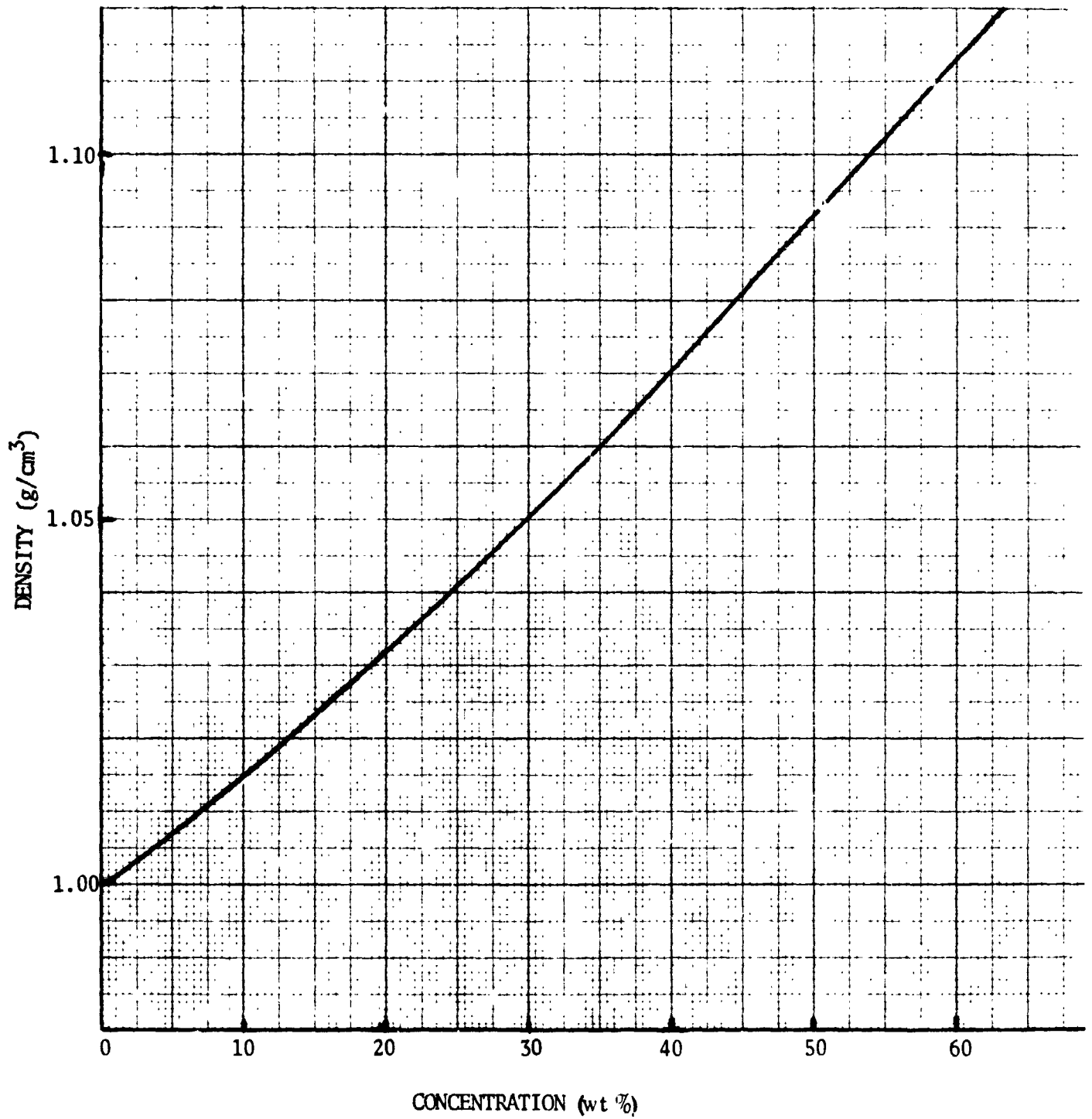
FIGURE 28. TMAC DENSITY VERSUS CONCENTRATION

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TABLE XIX   VISCOSITY OF TMAC SOLUTIONS

TMAC SOLUTION*			
CONCENTRATION	DENSITY	ABS. VISCOSITY	KIN. VISCOSITY
Wt. %	Grams/cc	Centipoise	Centistokes
51.5	1.092	32.8	30.04
39.9	1.07	7.67	7.17
30.0	1.05	3.09	2.94
20.0	1.032	1.86	1.80
10.0	1.015	1.31	1.29
5.0	1.008	1.l54	l.046

* pH ≈ 10.9, Data from NASA JSC
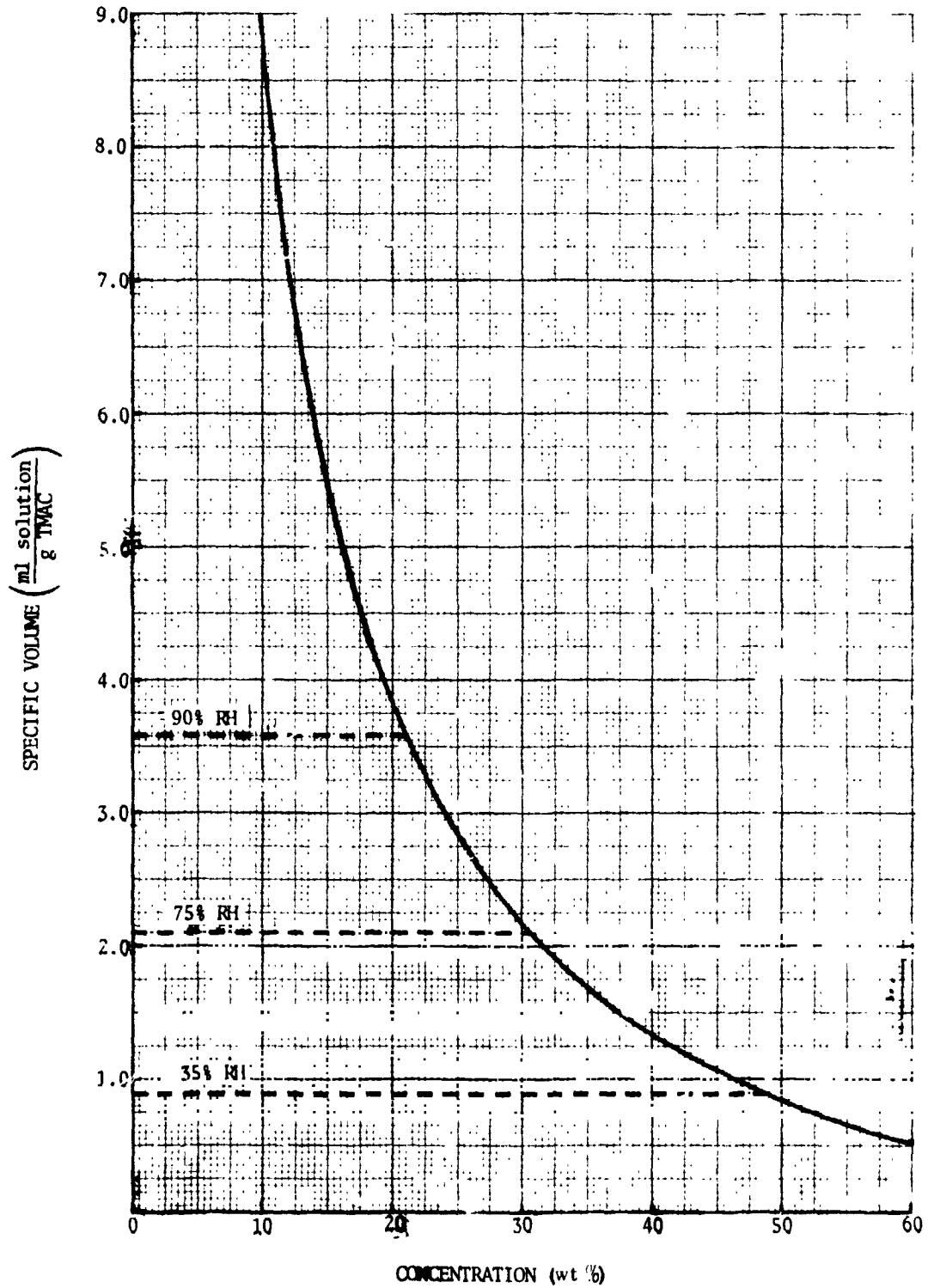
FIGURE 29. TMAC SPECIFIC VOLUME VERSUS CONCENTRATION
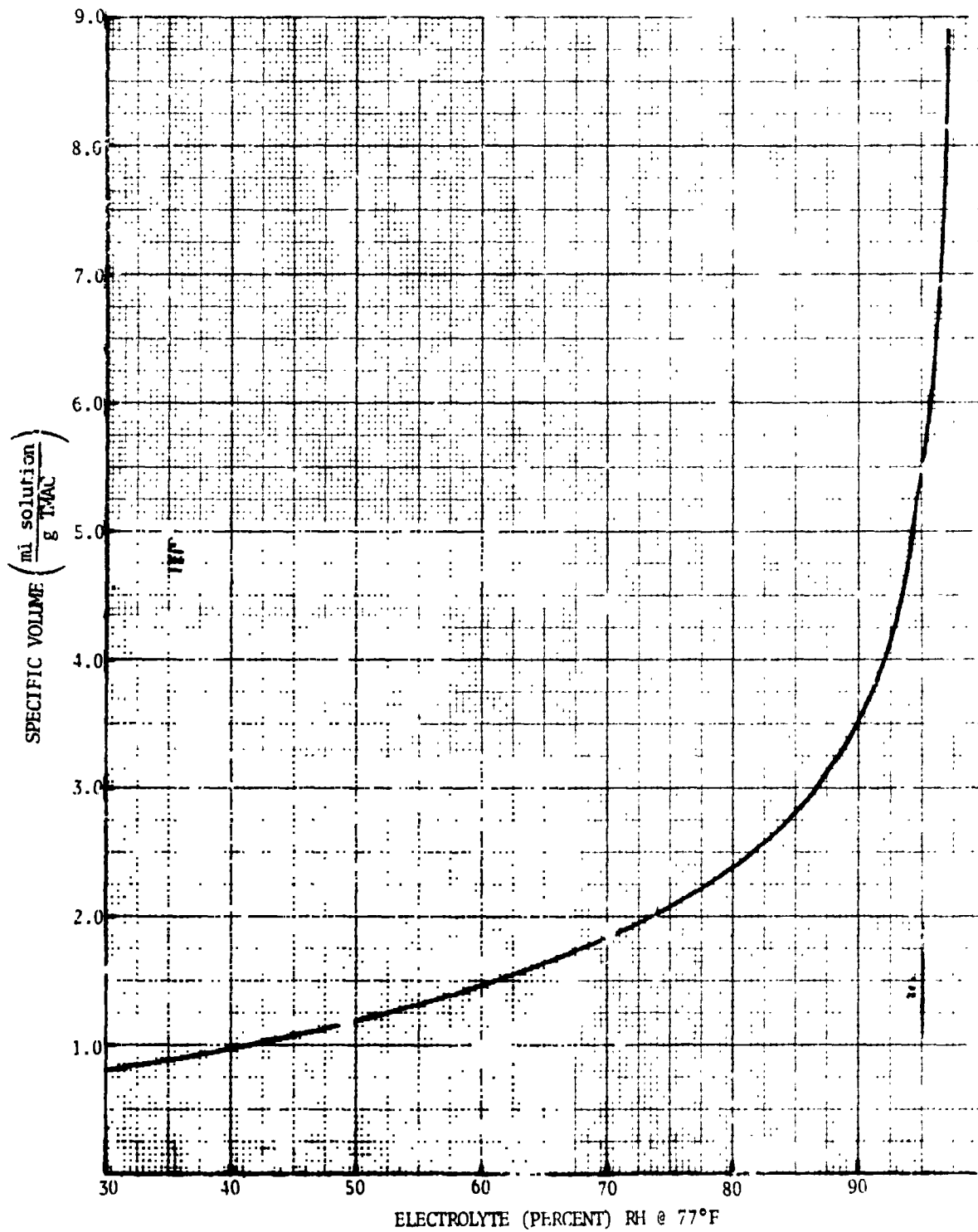
FIGURE 30. TMAC SPECIFIC VOLUME VERSUS ELECTROLYTE RELATIVE HUMIDITY
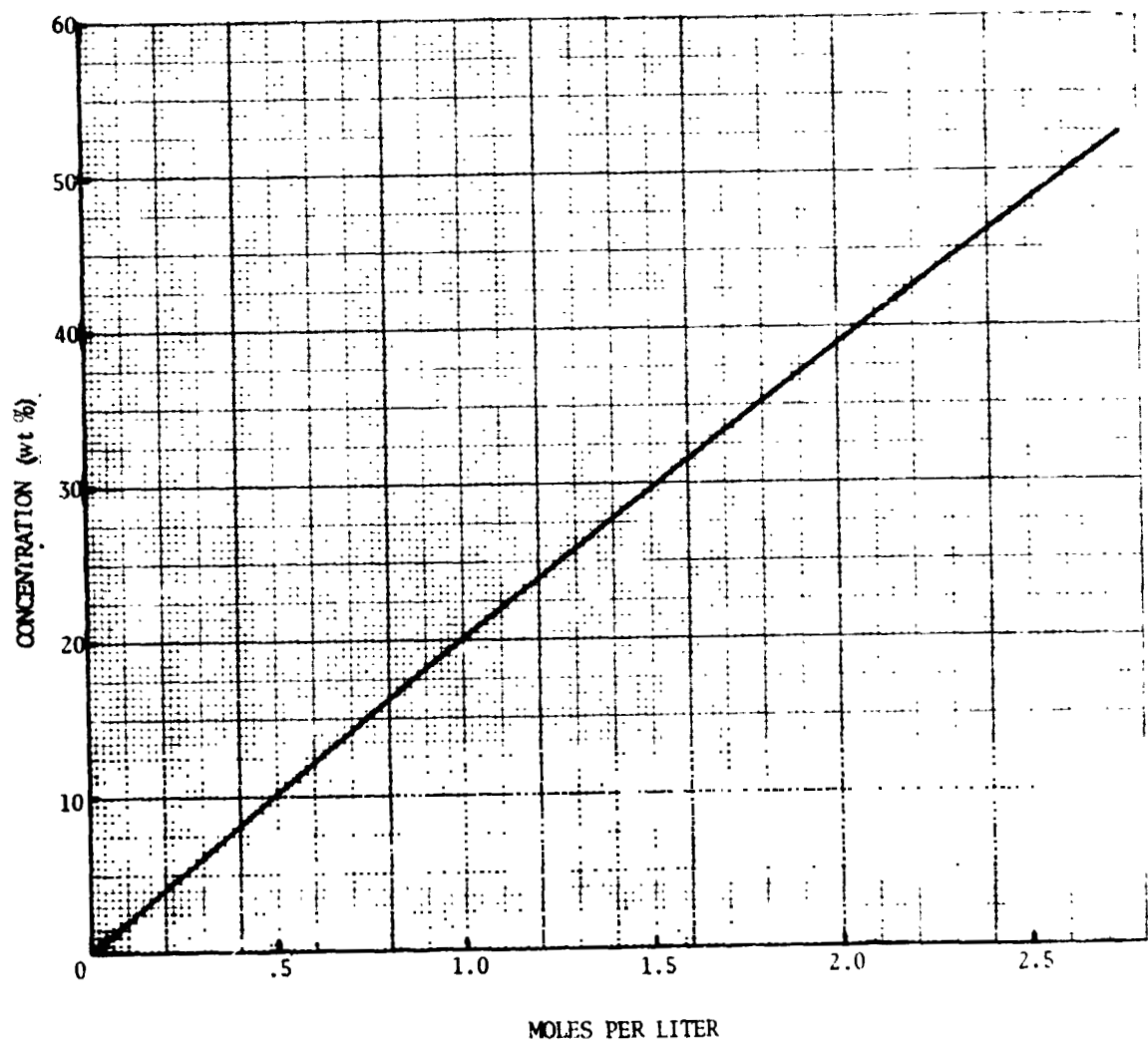
FIGURE 31.   MOLES PER LITER VS WEIGHT PERCENT (TMAC)

MODIFIED ELECTROLYTES

A search and evaluation of mixed (aqueous-nonaqueous) solvents to develop another electrolyte with low relative humidity capabilities was conducted.

One of the methods for obtaining an electrolyte with a low water vapor pressure is to replace a significant part of the water with a non-electrolyte solvent. The necessary properties for a non-electrolyte additive are:

- Negligible vapor pressure at cell operating temperature.

- Miscible with water.

- Resulting solution must dissolve a sufficient amount of electrolyte salt to give good electrical conductivity.

- Stable under cell conditions.

The initial literature search for materials with suitable properties was reported under the previous contract. NAS 9-11830. Only two materials, propylene carbonate and ethylene carbonate, were found which looked promising.

The solubility of propylene carbonate was found to be 3 ml per 10 ml of water* and this would result in a limited dew point depression. Ethylene carbonate has a melting point of 40°C (104°F) but a fifty-fifty volume mixture with water is soluble above 65°F where the HDC cell would operate.

The literature search revealed that propylene and ethylene carbonate had been employed as electrolyte solvents (anhydrous) and were relatively stable over a wide potential range. However, the stability should depend on the nature of the electrode material (catalyst) and probably the availability of water, which could induce hydrolysis of these cyclic esters to alcohol and acid or carbon dioxide.

A potential sweep with and without ethylene carbonate (three drops in 35 ml of 1 M Cs_2CO_3) is shown in figure 32 from which it is obvious that these materials are readily oxidized on a platinum black electrode in the presence of water. The relatively large anodic peak at ~0.75 volts on both the up and down sweeps cannot be due to the direct oxidation of ethylene carbonate. It is more reasonable to assume that the ethylene carbonate is rapidly hydrolyzed by base and platinum catalyst to alcohol followed by oxidation of the alcohol.

To date the only materials found that have sufficient solubility in water contain amine, hydroxy or ester groups, and all of these are readily oxidized under HDC conditions.

* The literature shows propylene carbonate as very soluble in water.

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SVHSER 6285

1M Cs_2CO_3

TRACE ETHYLENE CARBONATE ADDED

POTENTIAL SWEEP RATE = 1v/min
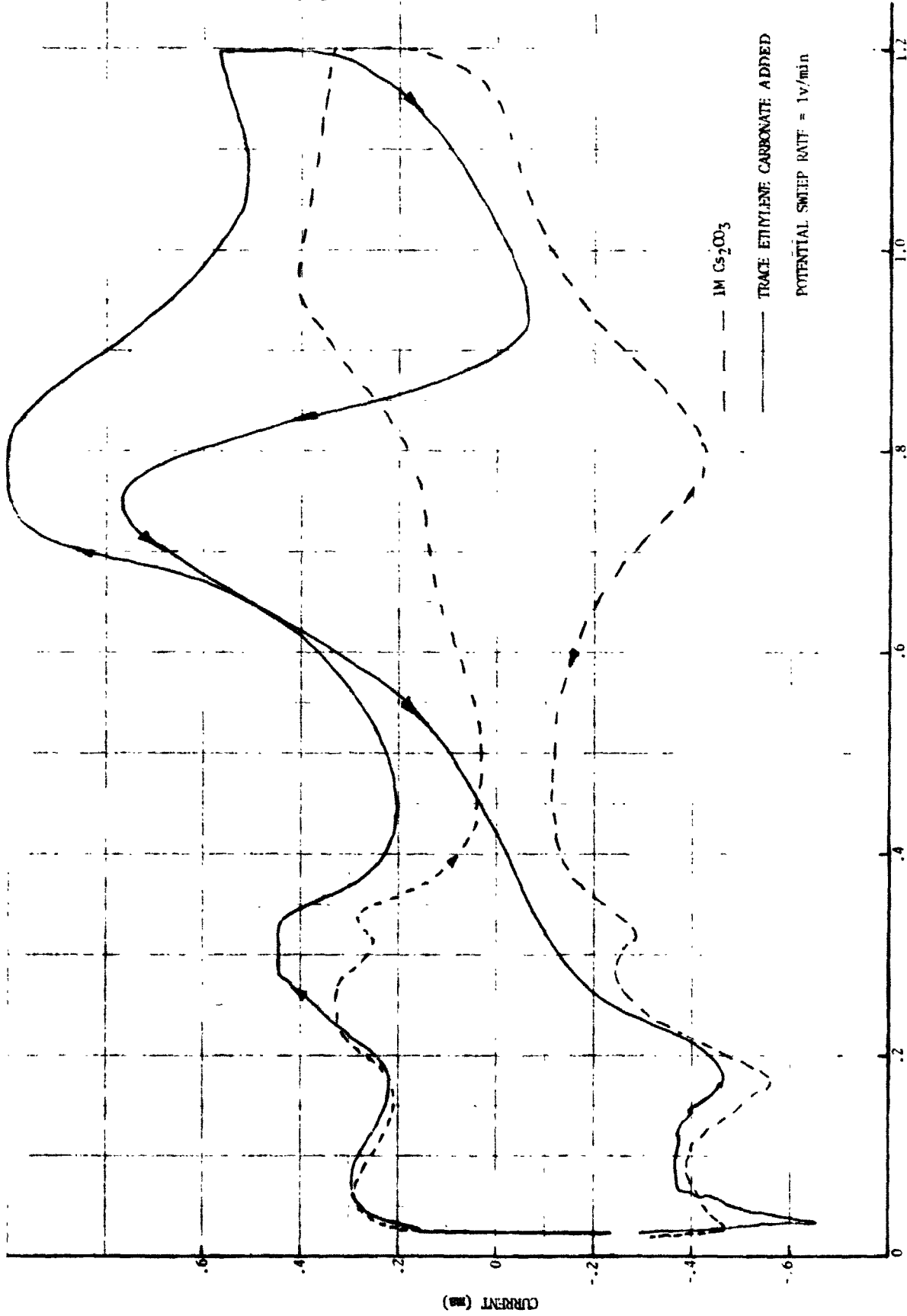
POTENTIAL (VOLTS) VERSUS HYDROGEN ELECTRODE IN SAME SOLUTION

FIGURE 32. POTENTIAL SWEEP FOR ETHYLENE CARBONATE

CURRENT (ma)

SPECIAL TESTS

In addition to the HDC tests discussed in the previous sections, three special investigations were made involving the HDC matrix compression, matrix set, and the hydrogen flow rate through the cell.

Matrix Compression

The purpose of the matrix compression tests was to determine the tortuosity and optimum matrix compression for asbestos, Neoprene asbestos, and Tissuquartz. The general procedure (reference Appendix) Master Test Plan, Section IV) was to measure electrolyte resistance (R) with and without matrix as a function of electrode distance (d) using the special micrometer cell shown in figure 33.

The resistance should be given by

$$R = \rho \frac{d}{A} \bullet \frac{d \ \tau}{d - d' \ (1 - m)} = \frac{\rho d \ f\tau}{A}$$

where ρ is the electrolyte resistivity, A the electrode geometric area, τ the tortuosity, d' the uncompressed matrix thickness (dry) and m is the matrix void volume. The dry uncompressed matrix thickness and the maximum compressed thickness, $d^O = d'$ (1 - m), were measured with a micrometer to $\pm$ 2 mils and $\pm$ 0.5 mils, respectively. Without matrix, the tortuosity and matrix factor (f $= d/d-d^O$) are both equal to one and the above equation reduces to R = ρ d/A. For a given electrolyte ρ /A is found by plotting R versus d and extrapolating to d = 0 to correct for the cell electronic resistance. The tortuosity is found from a plot of R versus fd ρ/A and if f is not a function of d the optimum compression should be at d = $2d^O$.

To determine the resistance a constant voltage ($\pm$ 50 mV, 1 K Hz) square wave was applied to the cell and the resulting current was extrapolated to "zero" time ($\pm 2 \mu$ sec) to eliminate capacitance and polarization effects. The electrolyte was 1 gram KCl per liter to give a resistance ($\sim$50 ohms at 25 mils) compatible with the instrumentation. To obtain reproducible results it was necessary to keep the cell in a 100 percent relative humidity chamber to prevent water evaporation from the electrolyte. Also, as the electrode spacing was decreased. the excess electrolyte was removed by blotting with electrolyte dampened paper tissue. It was found that the use of dry tissue had a tendency to remove too much electrolyte from the matrix material.

For each matrix material studied, a plot of R versus d (figure -a) and R versus fdp/A (figure-b) is given. The R versus d plot also shows the electrolyte resistance without matrix and the calculated resistance for a tortuosity value of one. The values for the dry uncompressed matrix thickness and the thickness at a maximum compression are shown as d' and d^O, respectively. Except where noted, two layers of matrix were used.

1 7/8 - 2 7/8 INCH MICROMETER

PLEXIGLAS
INSULATION

STOP OFF LACQUER

Pt — Pt ON FACE

ELECTRICAL
LEADS

COPPER RODS. GOLD
PLATED (1 MIL.) &
RHODIUM PLATED (1 MIL.)
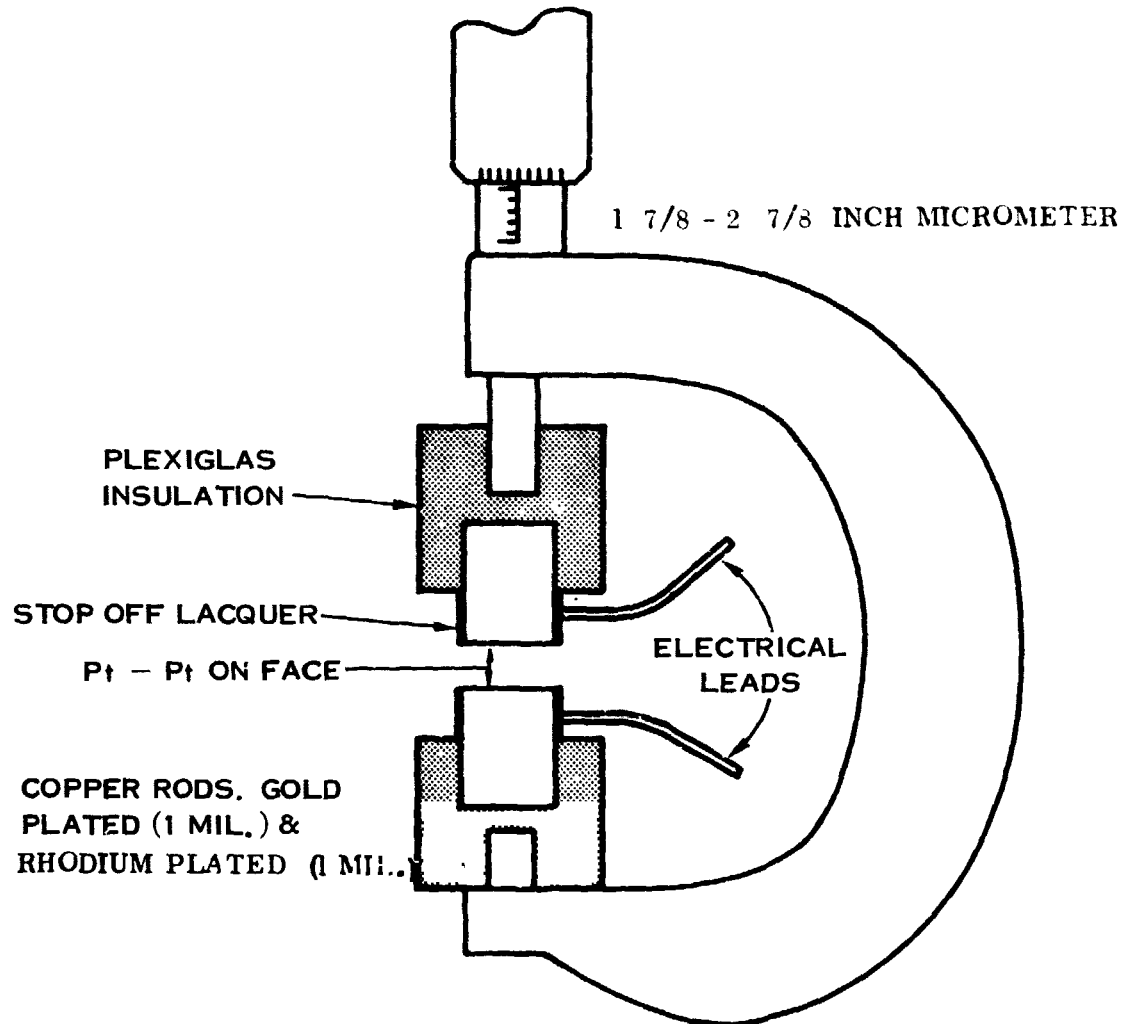
FIGURE 33. MATRIX CONDUCTIVITY FIXTURE

The data for fuel cell asbestos are shown in figures 34a and 34b. The tortuosity factor is 1.53 for d $>$ 35 mils but decreases for d values less than 35 mils. This behavior may be due to edge effects where a small fraction of the matrix remains uncompressed resulting in a constant (with d) parallel resistance so that the measured value is less than the true resistance between the electrode faces at low values of d as reflected in figure 32. The minimum resistance occurs at close to the calculated value of $2d^\circ$ = 24 mils.

The data for Tissuquartz and Neoprene asbestos are given in figures 35 and 36 respectively. Both of these materials, having an uncompressed void volume greater than 90 percent, gave unexpected results. As can be seen from figures 35 and 36, the R versus fd ρ/A plot is a straight line with a slope of one (T = 1) but does not extrapolate to zero. Unless it is assumed that the tortuosity increases continuously with increasing matrix compression, it appears that the equation for resistance should contain an added factor which is independent of d.

In view of the very high void volume, a tortuosity factor of one would be expected for all but very small values of d so that an additional term to the resistance equation seems more reasonable, although it is somewhat difficult to justify a priori. A possible explanation could be attributed to double layer effects associated with the large matrix – solution interfacial area. This interfacial area would be independent of the compressed matrix thickness d. Also, from microsco c examination of the three matrix materials it appears that the P&WA fuel cell asbestos has a much smaller surface area than the other two materials so that double layer effects would be much less pronounced with the fuel cell asbestos.

To minimize any electroosmotic effect a saturated solution of KCl was used as the electrolyte in one experiment with Tissuquartz. This experiment required special instrumentation because the measured resistance was in the order of 1/2 ohm. A Hewlett Packard model 606B signal generator supplied a 98 μ amp rms 100K Hz sine wave and the voltage was fed into a Tektonix 1A7A applifier with a Fluke model 873A AC/DC differential voltmeter as readout. With this equipment the measurements could be made to 0.1 micro volt. The results corrected for an electronic resistance of 0.345 ohms are shown in figure 37. The line for Tissuquartz is parallel to the plain electrolyte line but displaced by 0.075 ohms which would indicate again that the resistance equation should contain an additional term independent of d but probably a function of ρ .

An equation of the form R = ρ/A [df T + α], where α is a constant for a given matrix material and electrolyte, allows a fit of the data. The dilute and saturated KCl solutions with Tissuquartz gave the same α value which would indicate that α is independent of electrolyte concentration, however, additional experiments would be necessary to definitely establish this as fact. Data obtained with Neoprene asbestos and dilute TMAC electrolyte also required the addition of the α term but its value was different than that for the KCl electrolyte.
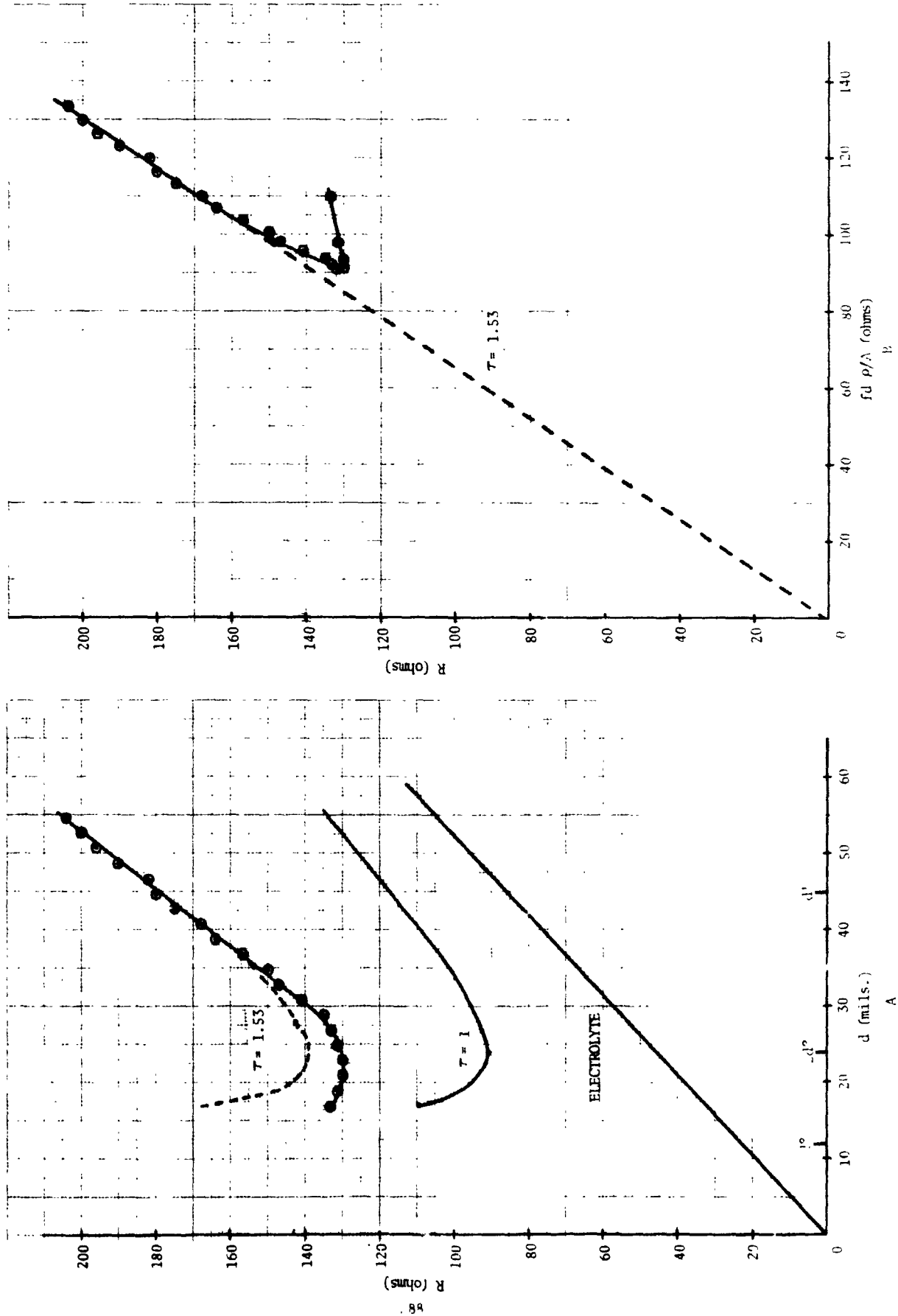
FIGURE 34. P&WA ASBESTOS - ELECTROLYTE RESISTANCE VERSUS THICKNESS
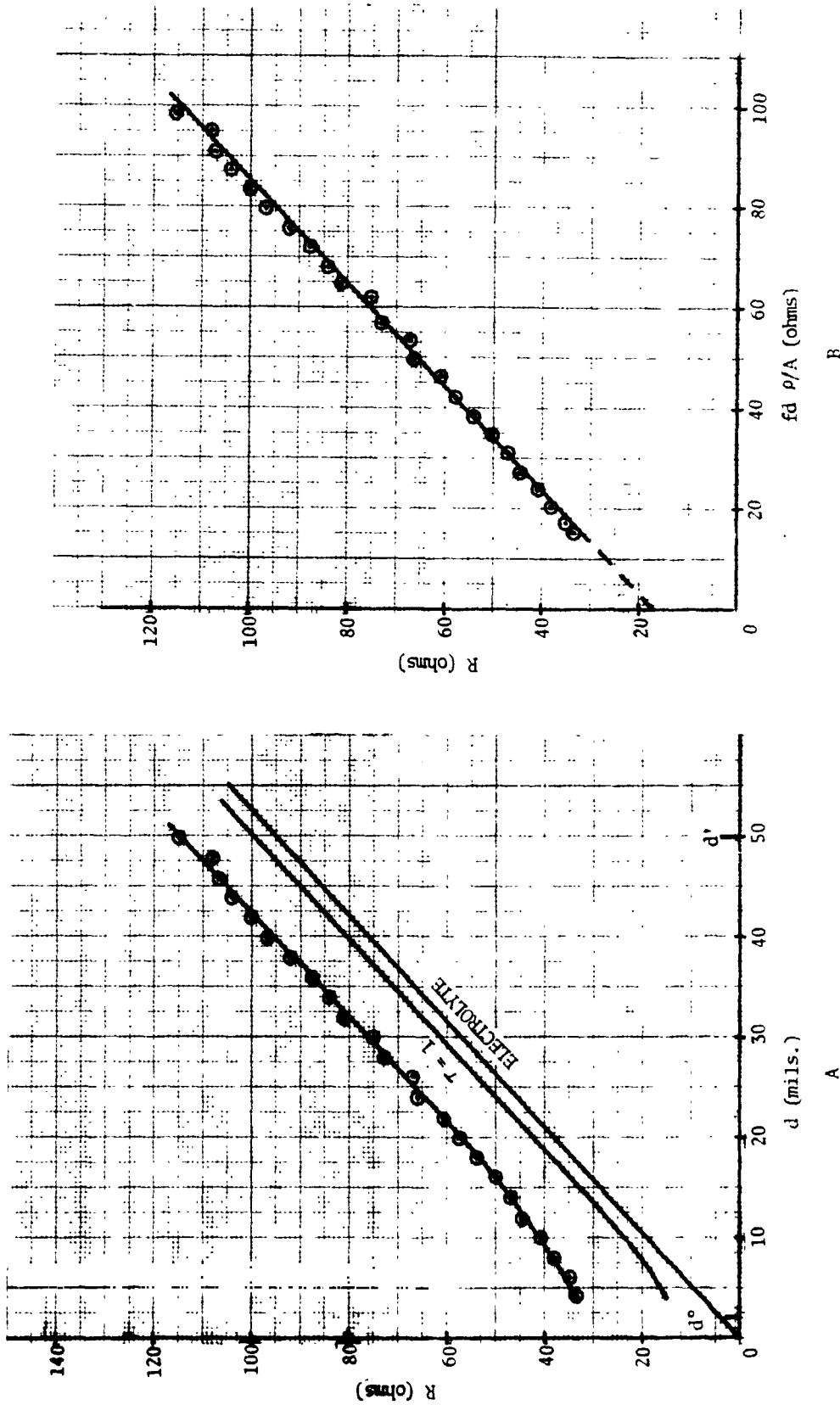
FIGURE 35. TISSUQUARTZ - ELECTROLYTE RESISTANCE VERSUS THICKNESS
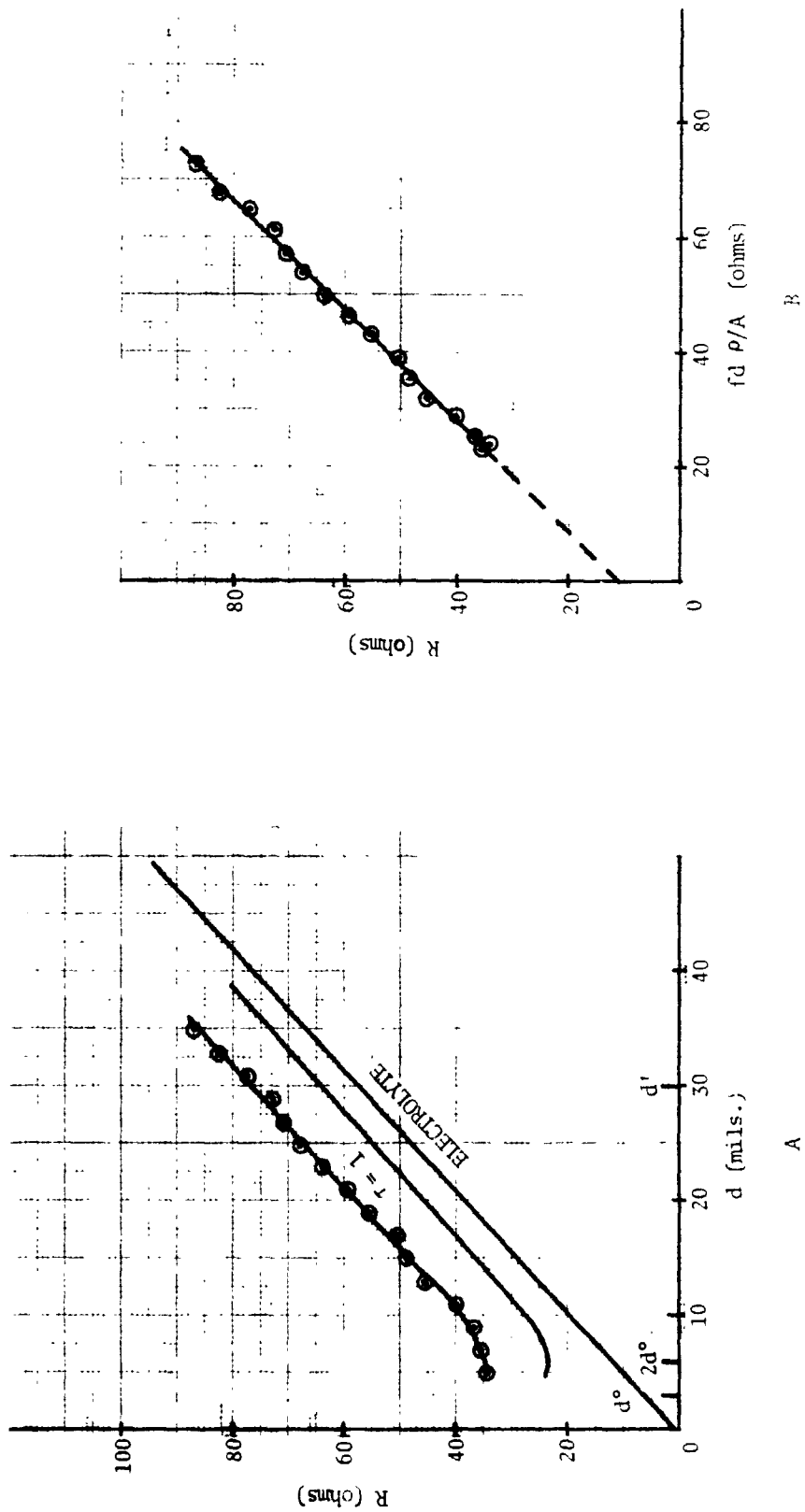
FIGURE 36. NEOPRENE ASBESTOS - ELECTROLYTE RESISTANCE VERSUS THICKNESS
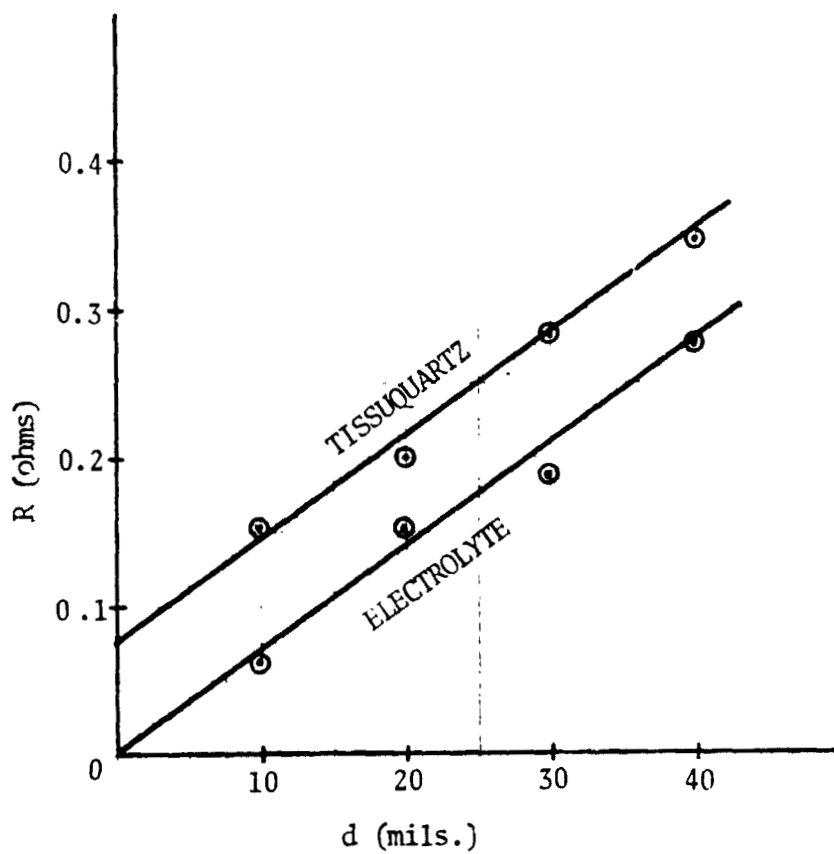
FIGURE 37. TISSUQUARTZ - (SATURATED KCl) RESISTANCE VERSUS THICKNESS

It is conceivable that the various interactions between the io~ 'ution and those immobilized in the double layer at the matrix – solution i face ld result in retardation of the mobile ions in solution. Such a decrease on mobi y would increase the matrix – electrolyte resistance by an additional fac'or.

Since the matrix for the WVE cell consists of one layer of Neoprene asbestos and two layers of Tissuquartz, data for this combination is shown in figure 38. The optimum compression is approximately 7 mils which corresponds to the value for $2d^O$. The simple equation would not apply to a combination of matrix material so an R versus fd P/A plot was not made.

Matrix Set

Part of the 'ask of investigating the HDC matrix characteristics required a compression relaxation and set test to be performed. The objective of this task was to determine if the final set of the matrix provided sufficient compression resistance (10 psi minimum) to maintain good contact between current collector, electrodes, and matrix. The testing established the pressure load versus matrix compression for various matrix configurations and for various durations up to six months. Tests were performed in accordance with the Master Test Plan, Section VIII, reference Appendix.

The comparison of loading versus deflection of three typical matrix configurations is presented in figure 39. This data shows that the addition of one sheet of Tissuquartz increases the loading very little, but one additional sheet of asbestos increases the loading by a factor of approximately 1.5. The slope of these curves, loading pressure per inch of deflection, is about the same.

The effects of compression cycling the matrix are shown in figure 40, which reveals the largest change in loading between the first and second cycles and very little change in the loading between the second through the fifth cycles.

The change in loading at a set matrix compression (0.022") is presented in figure 41, which shows about a 14 percent decrease in the matrix loading during the initial two hours of assembly.

Six samples were tested for the endurance portion of the matrix set investigation. Each sample, consisted of two sheets of 0.020 inch thick asbestos, separated by a 0.018 inch thick Tissuquartz, saturated with Cs_2CO_3 and compressed to 0.024 inch thickness and held. The test fixtures holding the compressed samples, were placed in a test chamber where a 52°F/45°F temperature/dew point and 2.5 mm Hg P_{CO_2} condition was maintained. After one, two, six, fifteen and twenty-five week durations, the samples were removed and compressed using a loading of 0 to 200 psi. The one and two week samples required a 70 psig loading before any indication of additional matrix compression was noted. Both of these samples, as shown in figure 42 had the same
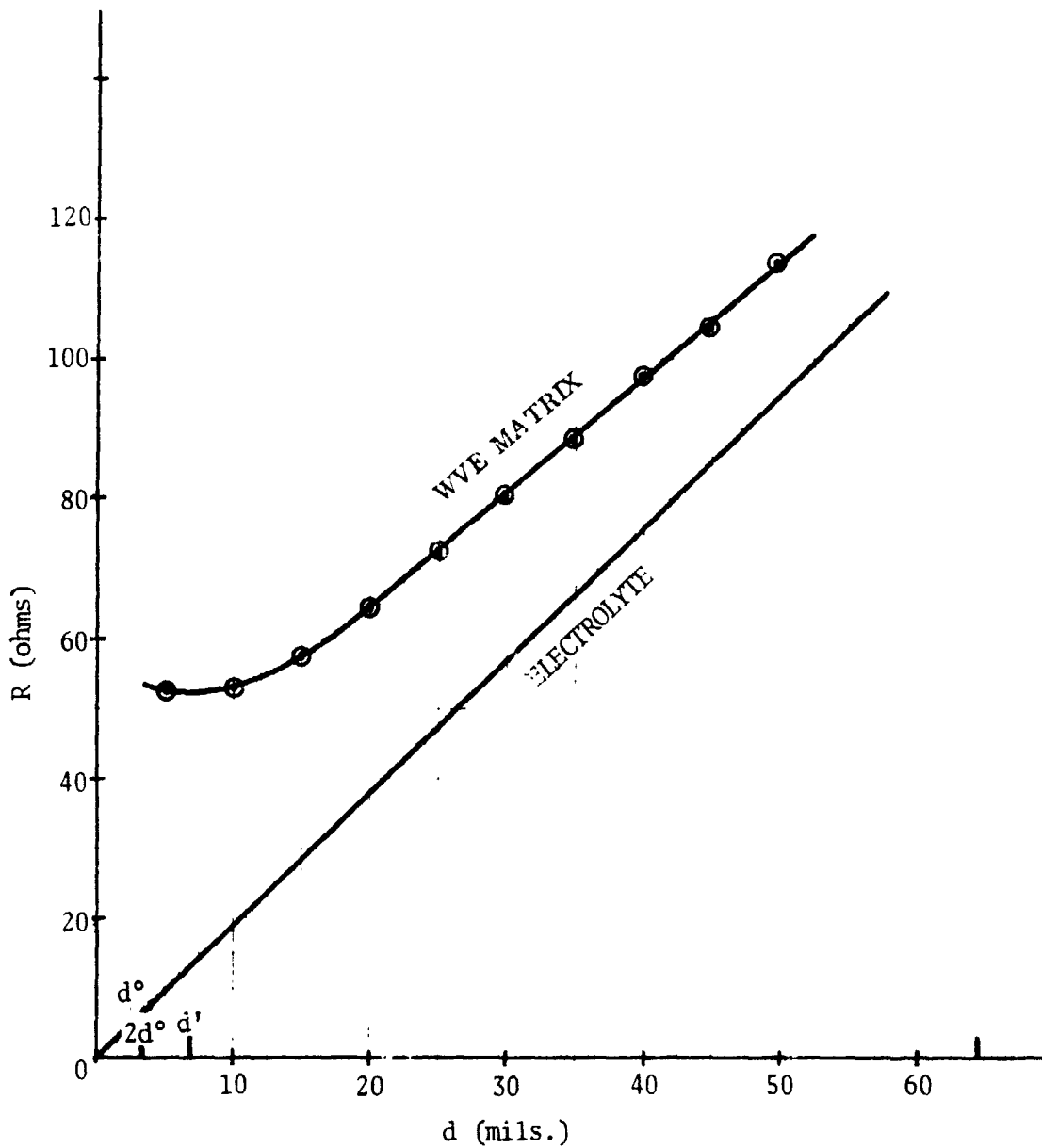
FIGURE 38. (WVE MATRIX) TISSUQUARTZ NEOPRENE ASBESTOS - ELECTROIYTE
RESISTANCE VERSUS THICKNESS

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MATRIX SET TEST
NAS 9-12920 MASTER TEST PLAN
SECTION VIII PARA. 4.0
0.020" P&WA SUPER CLEAN ASBESTOS

3 SHEETS OF WET ASBESTOS

2 SHEETS OF WET ASBESTOS & ONE .018 INCH SHEET TISSUQUARTZ

2 SHEETS OF WET ASBESTOS

LOADING (psig)

MATRIX THICKNESS (inches)
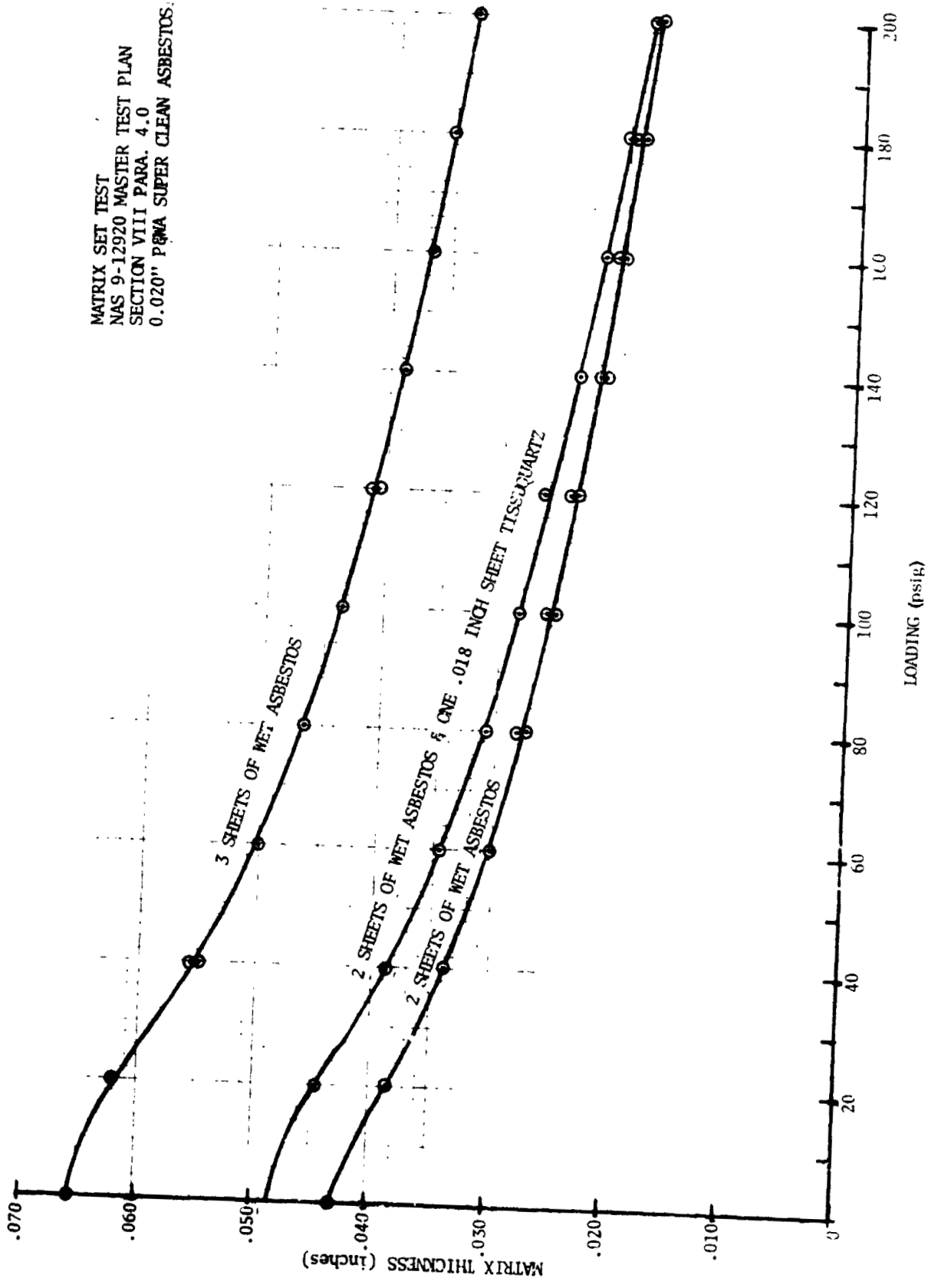
FIGURE 39. MATRIX COMPRESSION VERSUS LOADING

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A.

MATRIX SET TEST
NAS 9-12920 MASTER TEST PLAN
SECTION VII PARA. 4.0
2 SHEETS OF 0.020" P&WA
SUPER CLEAN ASBESTOS (WET)

NO. OF COMPRESSIVE CYCLES

1st
2nd
3rd
4th & 5th

MATRIX THICKNESS (inches)
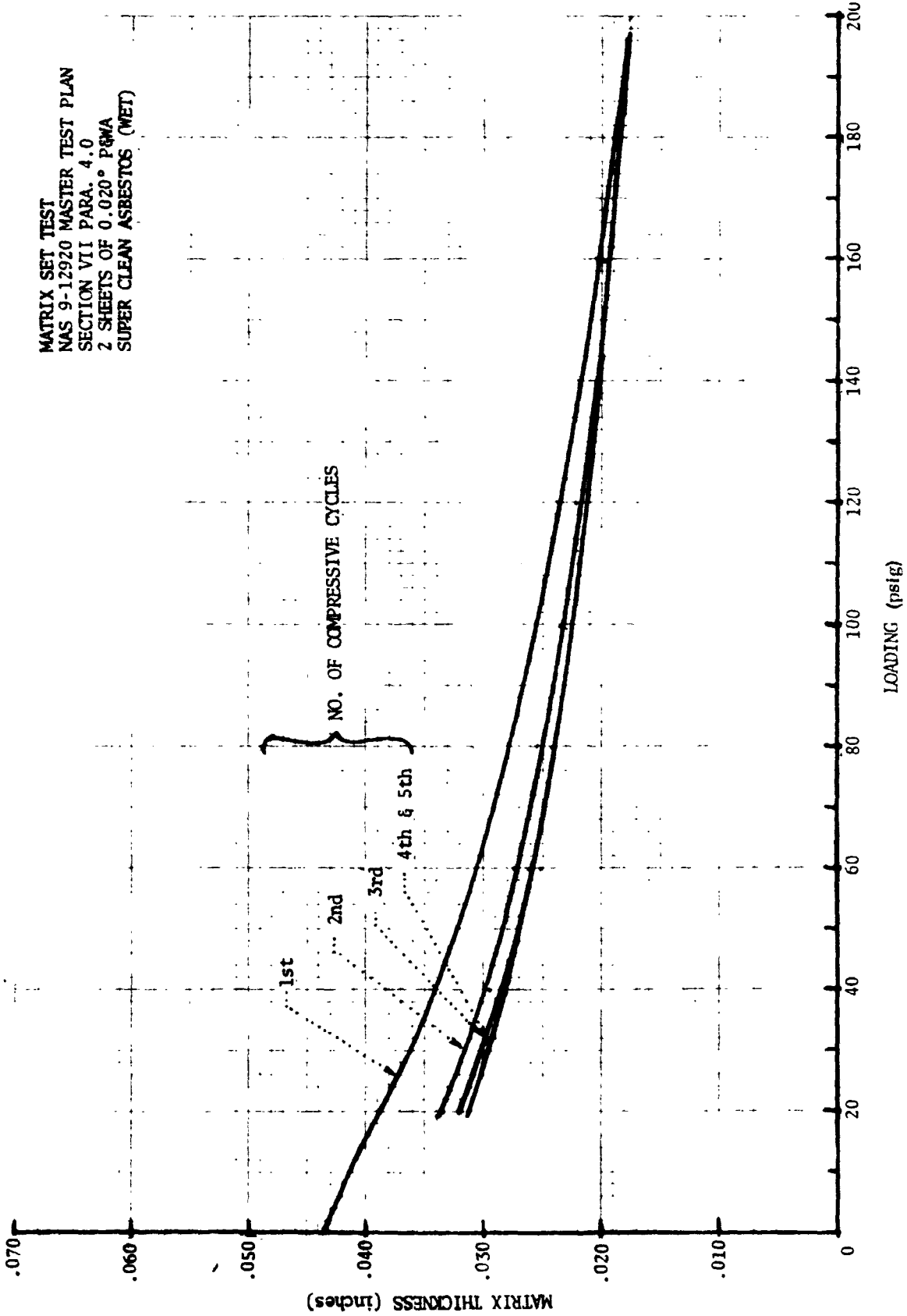
LOADING (psig)

FIGURE 40. MA1.IX CYCLIC COMPRESSION VERSUS LOADING
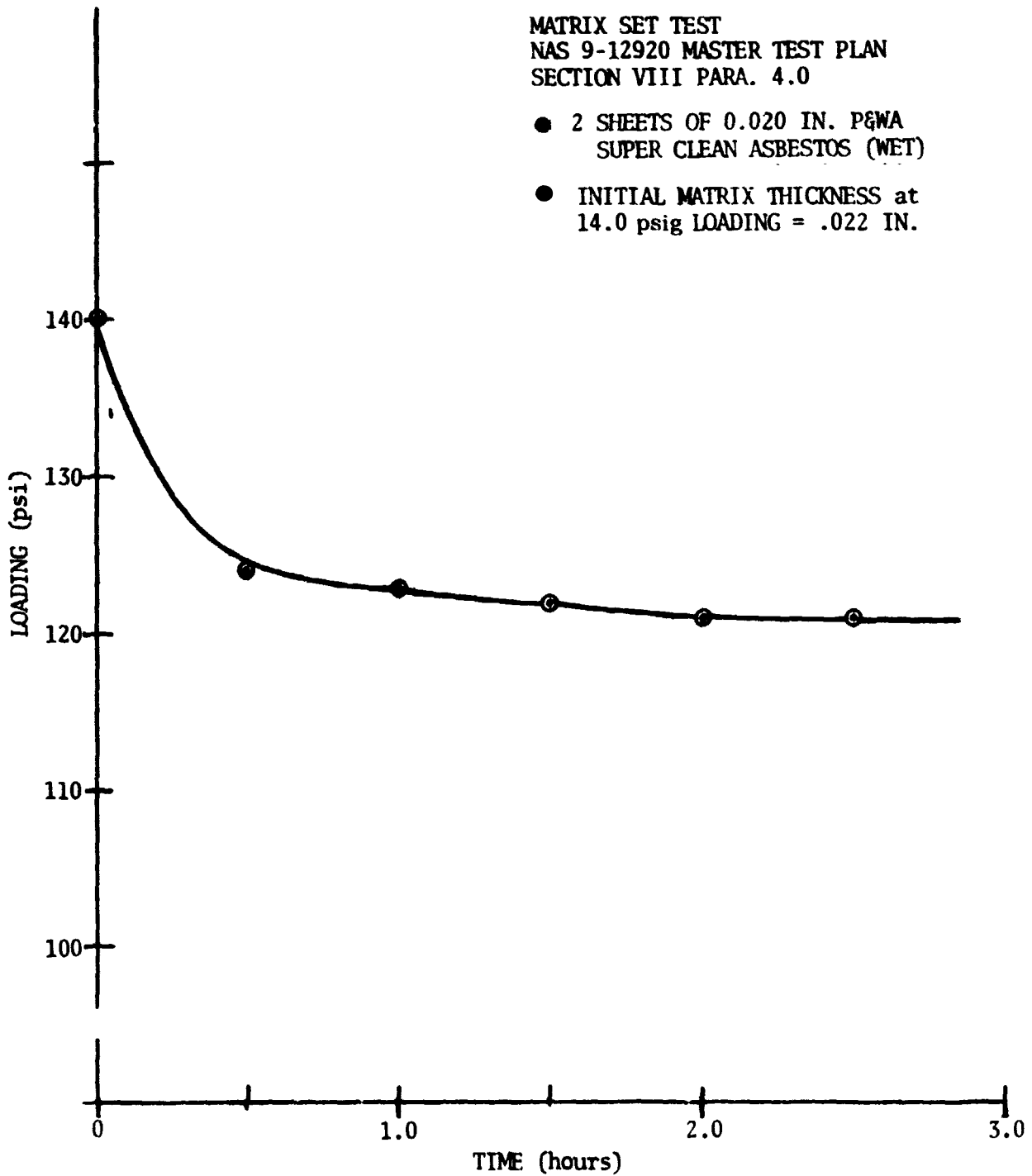
FIGURE 41.   MATRIX LOADING VS TIME
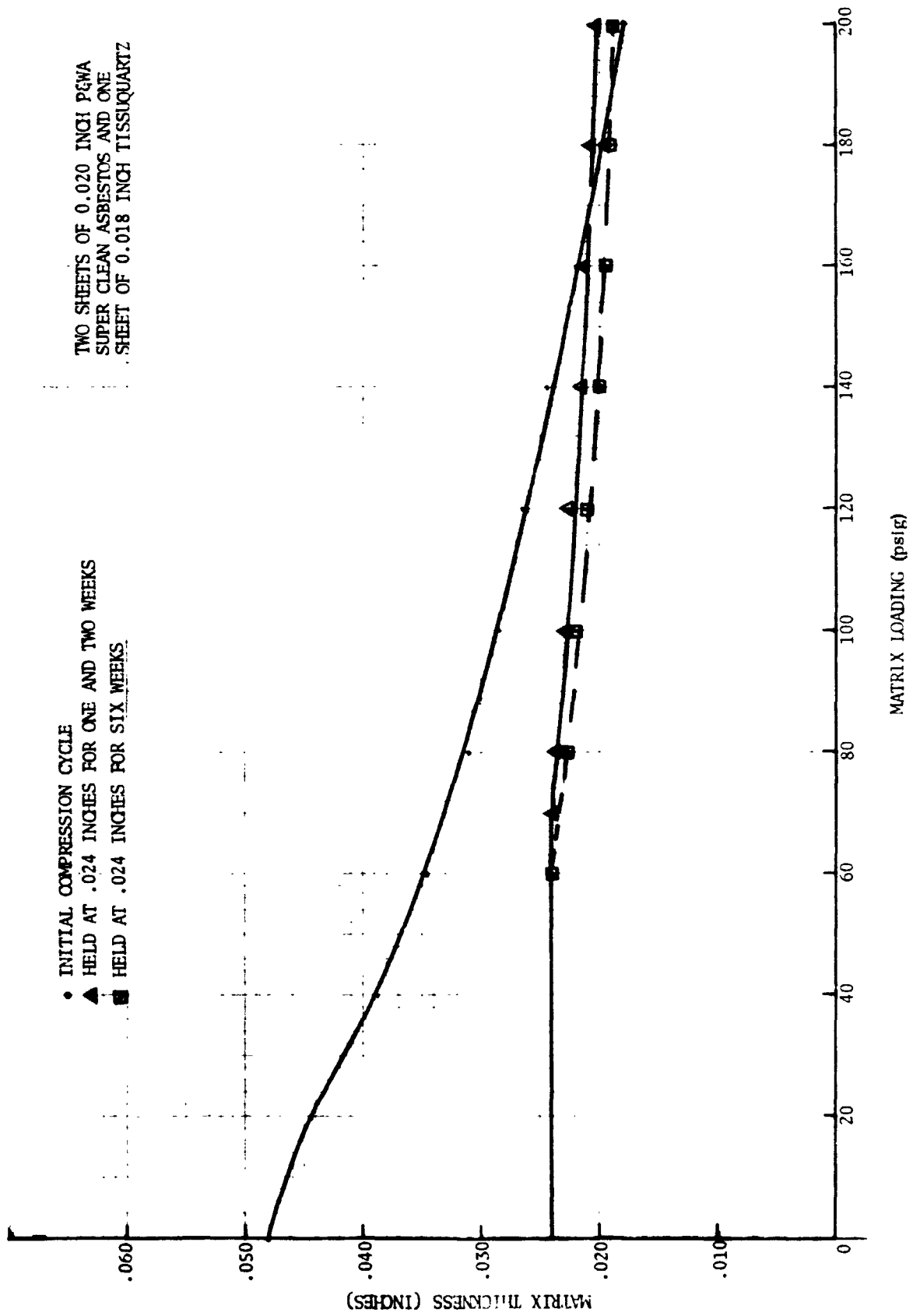
FIGURE 42. MATRIX SET TEST

INITIAL COMPRESSION CYCLE

HELD AT .024 INCHES FOR ONE AND TWO WEEKS

HELD AT .024 INCHES FOR SIX WEEKS

TWO SHEETS OF 0.020 INCH PGWA
SUPER CLEAN ASBESTOS AND ONE
SHEET OF 0.018 INCH TISSUQUARTZ

MATRIX LOADING (psig)

MATRIX THICKNESS (INCHES)

compression versus loading values. The six, fifteen and twenty-five week samples required a 60 psig loading before deflection was observed, but the slope of the com-pression/loading curve is the same as the earlier samples.

The six month endurance data reveals that the matrix maintains sufficient com-pression resistance (60 psig) to provide good interface contact between the matrix, electrodes and housings which should not cause any change in the cell's IR value.

HDC HYDROGEN FLOW IMPROVEMENT

The objective of testing the HDC at various inlet hydrogen flow rates was to determine the effect of this variation on cell performance. Testing was done per Master Test Plan, Section III, reference Appendix. The HDC cell pair which was used for this testing was cell pair S/N 011-3 which is a non-reservoir type cell using Cs_2CO_3 for the electrolyte. This cell pair previously had been used under the SSP HDC test program for approximately four months and was restarted for this test program on 8 September 1972. The inlet conditions to the cell were maintained at $68^\circ F$ dew point, $74^\circ F$ dry bulb, 3.00 mm Hg CO_2 partial pressure and 21 percent oxygen. The cell was operated at a constant current of 16 asf, except at the very low inlet H_2 flow rates where the current dropped off to a low of 14.2 asf. Nitrogen purging of the cell was done daily for approximately 5 minutes duration. The test data presented on figures 43 through 45 show that current efficiency (normalized) is not significantly affected by hydrogen flow rate. However, the power output of the HDC is reduced at the lower H_2 inflow rates below a stoichiometric flow of 3.0. This drop off in power below a stoichiometric flow of 3.0 will not present a problem to the WVE/HDC system since the WVE at 50 asf will produce sufficient H_2 to maintain a stoichiometric flow about 3.0.

HDC VERIFICATION TEST

The objective of this 90 day test program was to verify that the HDC cell pair modifications derived during the development phase of this program, would decrease the voltage decay rate and improve the CO_2 removal rate of the cell pair.

The cell pairs, reference figure 1, were of the following configuration:*

Item	Cell Pair S/N 021	Cell Pair S/N 022
Anode	PPF	Same
Cathode	DS 16-0	Same
Matrix	2 Sheets of (0.020 inches each) Fuel cell asbestos with 1 sheet of (0.018 inches Tissuquartz in the center	Same
Compressed Thickness Matrix	0.025 inches	Same
Electrolyte	Cs_2CO_3	TMAC
Outer Housings	Titanium Housings Platinum Plated (SVSK 83638 B) rolled to provide 0.060 inches concave curvature	Same

* Detail cell pair description and drawings are defined in reference (1).
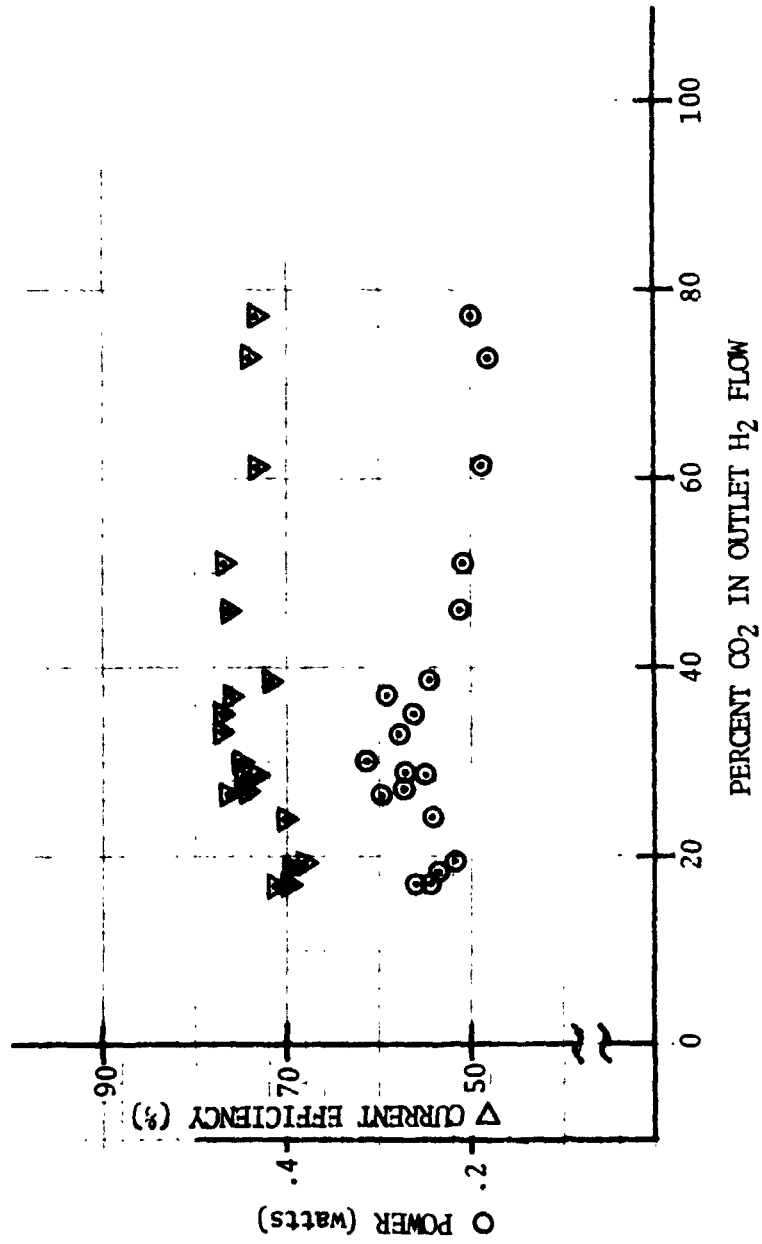
FIGURE 43. HDC HYDROGEN FLOW TEST RESULTS PERCENT CO_2 VERSUS
POWER AND EFFICIENCY
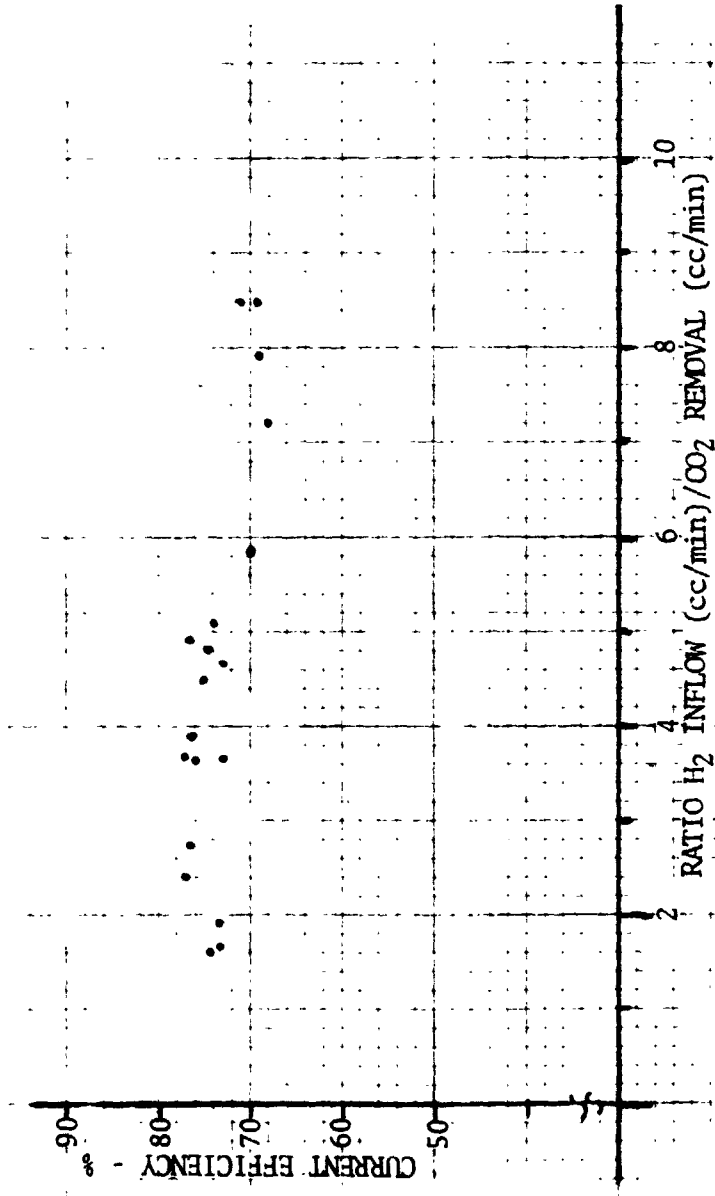
FIGURE 44.   HDC HYDROGEN FLOW TEST RESULTS H_2/CO_2 REMOVAL VERSUS EFFICIENCY
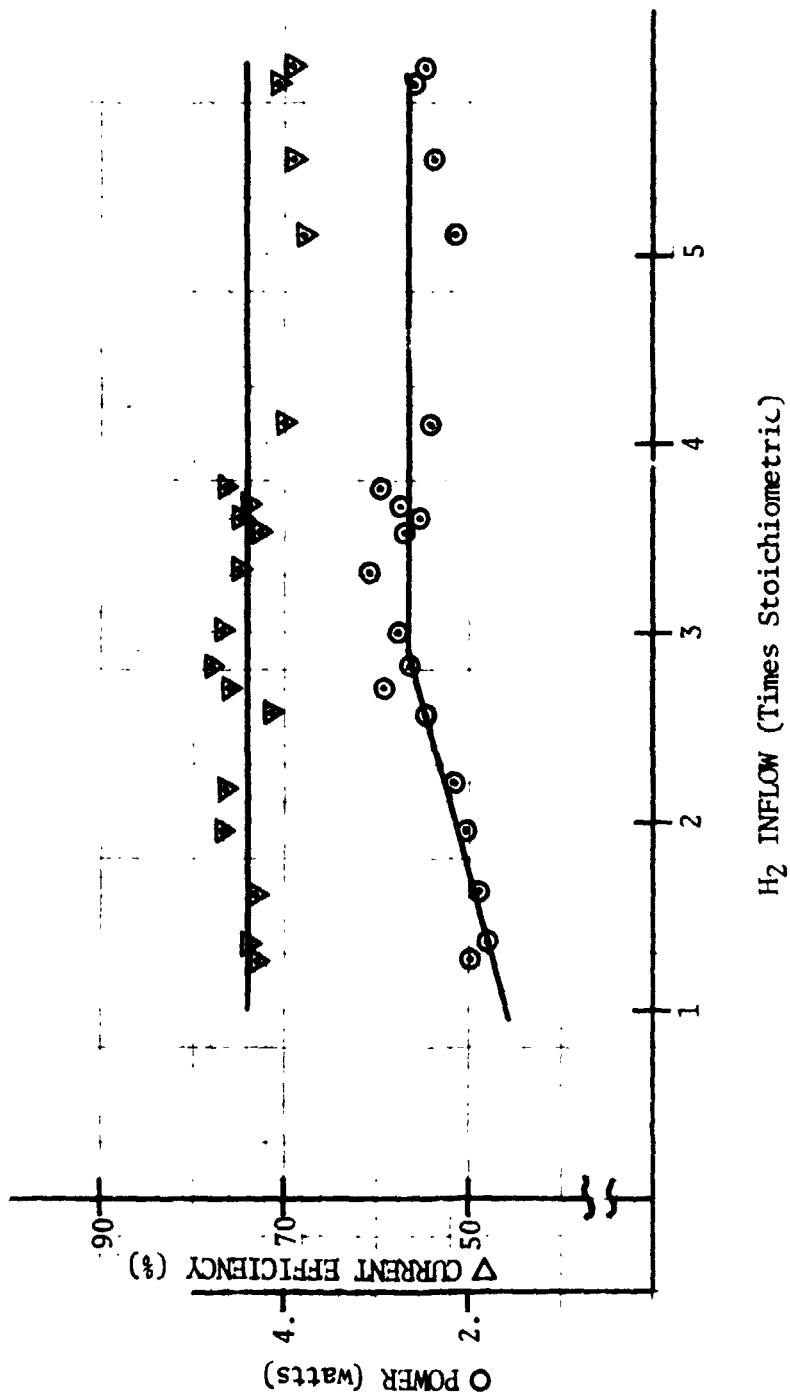
FIGURE 45. HDC HYDROGEN FLOW TEST RESULTS H₂ STOICHIOMETRIC FLOW
VERSUS POWER AND EFFICIENCY

Item	Cell Pair S/N 021	Cell Pair S/N 022
Center Housing	Titanium Housing Gold Plated (SVSK 83639 B) with a H_2 serpentine path of 0.060 inches width	Same

Testing of these cell pairs was done in individual test chambers as defined by figure 46 and schematically by figure 47. The facility consisted of separate plexiglass chambers, automatic data recorder, facility support equipment which maintained the chamber environment (P_{CO_2}, P_{O_2}, temperature and dew point) and cell pair current, and instrumentation for monitoring all the desired rig and cell parameters.

Verification testing of the two HDC cell pairs was conducted in accordance with the approved Master Test Plan, Test Number X (Appendix). The scope of the test plan was to establish parametric and endurar j test data on the two types of HDC electrolytes at various levels of P_{CO_2} and temperatures.

Cell Pair S/N 021 Testing

Cell pair S/N 021 operated satisfactorily for 40 days with an average CO_2 removal of 84% and an average voltage decay rate of 58 μ v/hr until a malfunction in the test facility caused the unit to flood with the loss of considerable electrolyte. During reconditioning at 52°/45°F a hydrogen leak developed and the cell was rebuilt with the same electrodes. At this time the electrode spacing was increased from the normal 25 mils to 35 mils in an attempt to increase the CO_2 transfer efficiency.

After this rebuild (test period 42nd-58th day) the cell power decayed rapidly as shown in figure 48. Potential decay curves of the anode and cathode of Cell Pair S/N 021 revealed a loss of active anode surface area but little change in the cathode. Just prior to shutdown for disassembly, attempts were made to restore cell power via the methods of open circuit, nitrogen purge, and vacuum on the hydrogen side, but the cell was not respondent to any of these treatments. Upon disassembly of the cell pair all components looked normal except the anode. Approximately 40 percent of the anode area contained a white precipitate, predominant on the hydrogen side but extending into the electrode structure as shown in figures 49a and 49b. This precipitate was found to be quite insoluble in water compared to cesium bicarbonate. Spectrographic analysis of the precipitate revealed silica as the major component with trace amounts of magnesium and calcium.

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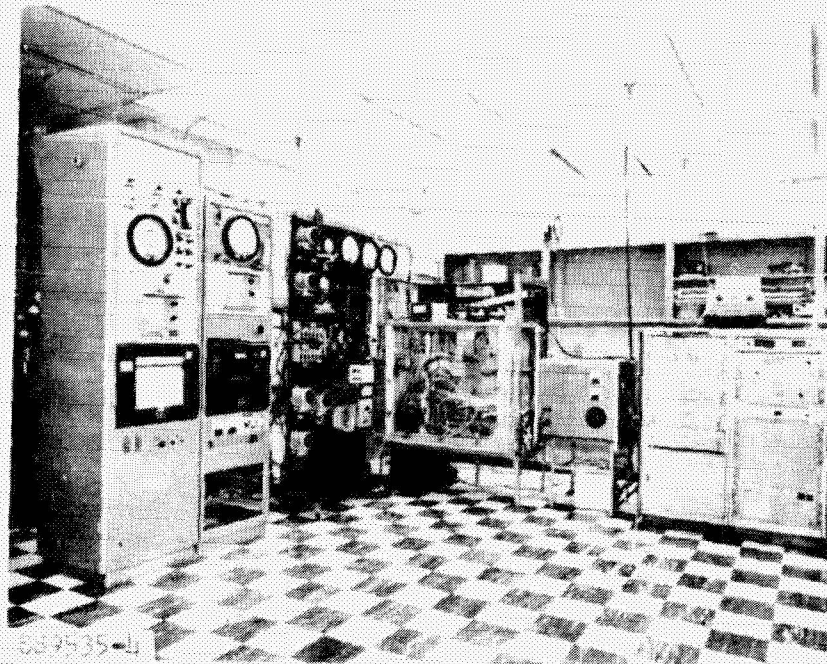
FIGURE 46. ELECTROCHEMICAL TEST FACILITIES
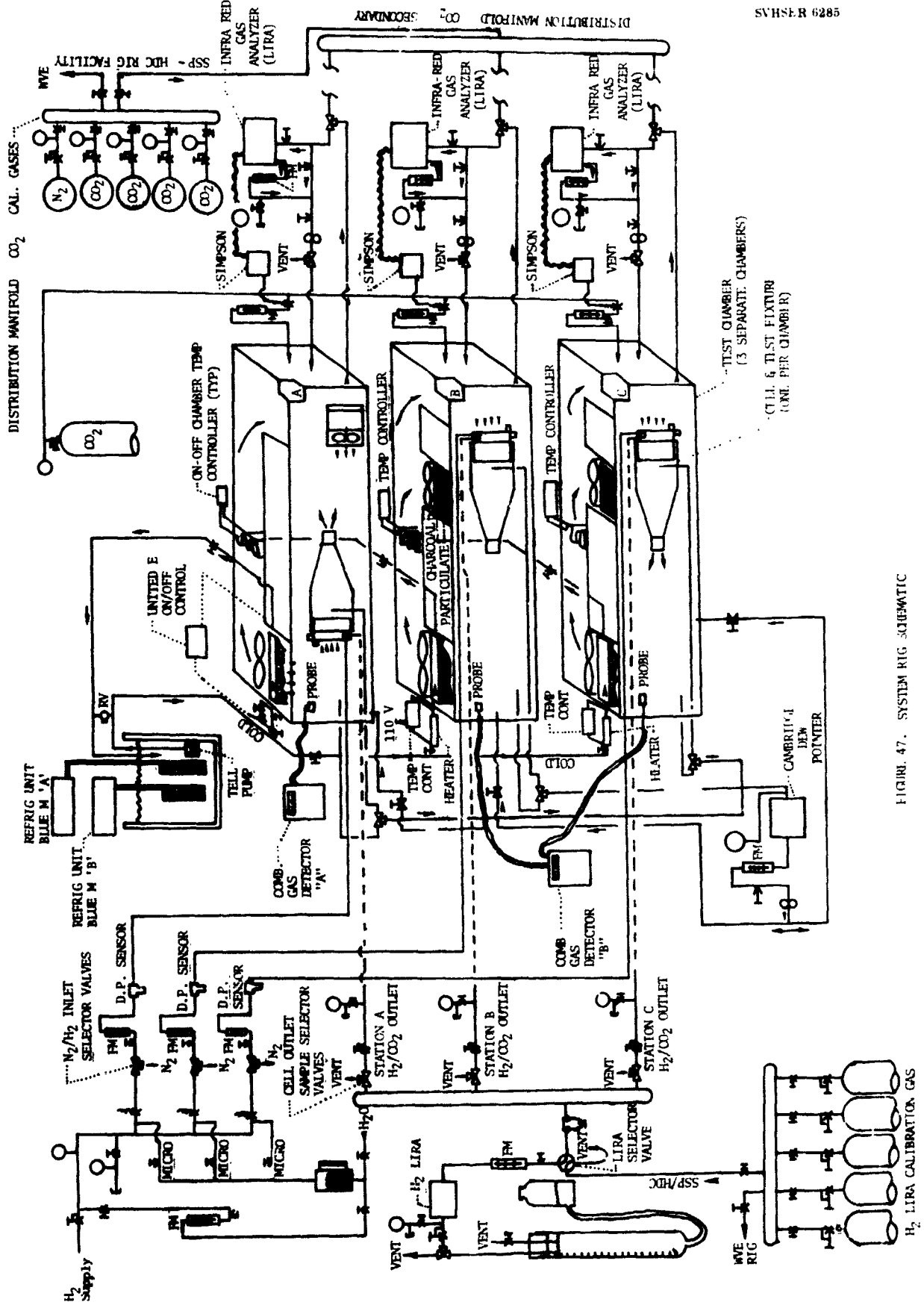
FIGURE 47. SYSTEM RIG SCHEMATIC

SVHSER 6285

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Standard
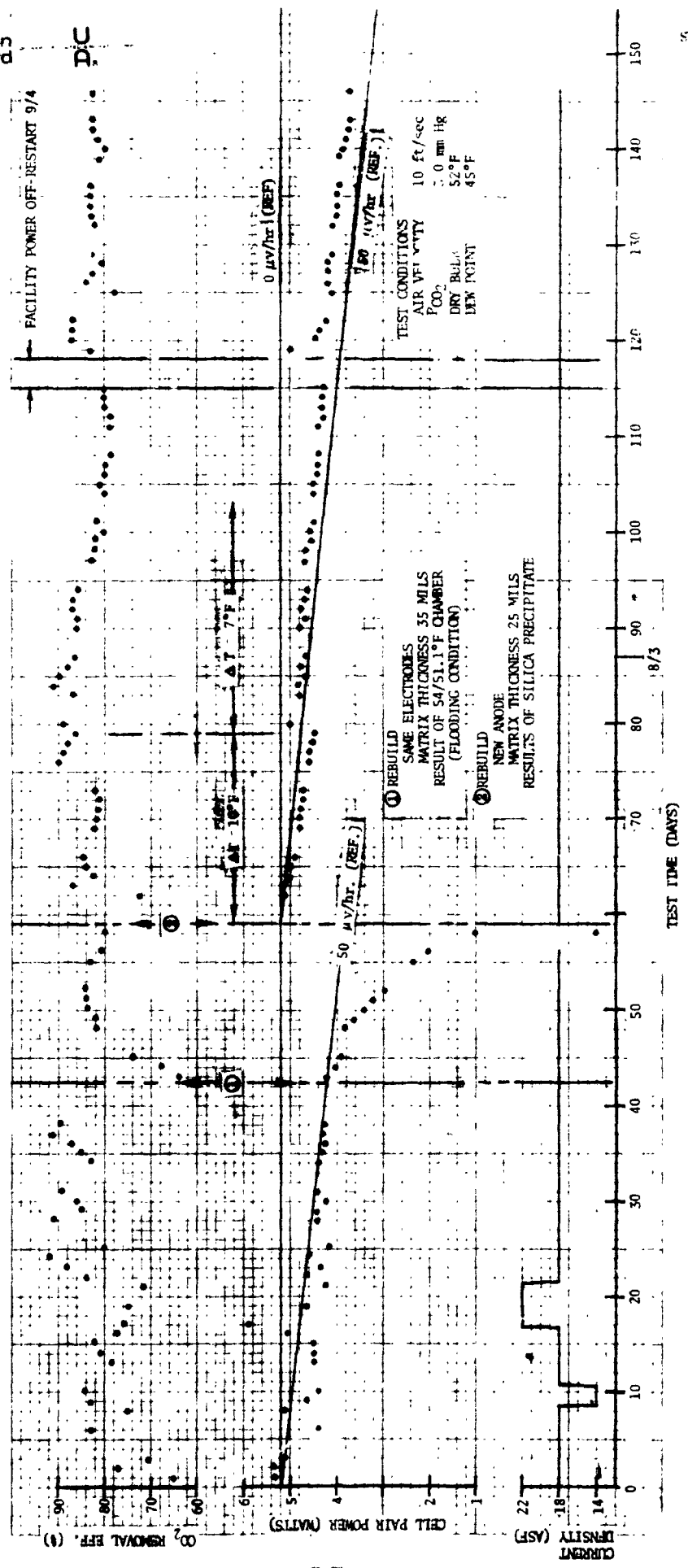
FIGURE 48. HDC CELL PAIR S/N 021 (Cs₂CO₃) PERFORMANCE VS TIME
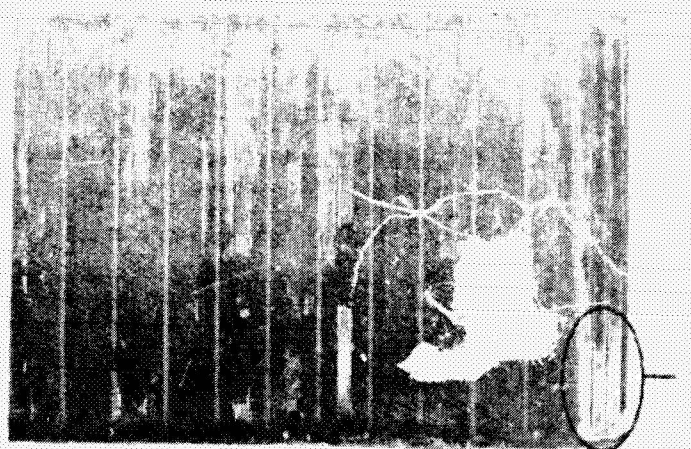
49 a   Mag = 0.4 x

MAGNIFIED VIEW OF SECTION A
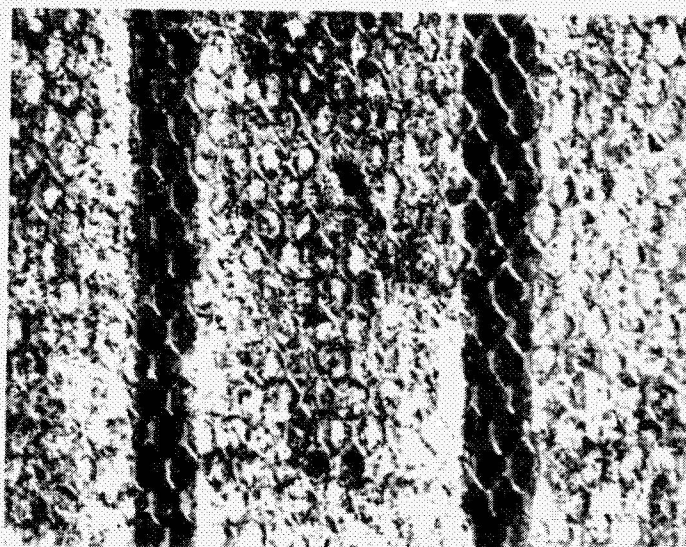
49 b   Mag = 6 x

FIGURE 49. CELL S/N 021 ANODE

This was the first time an insoluble precipitate had been found in the anode structure although some modification of the asbestos structure from fibrous to amorphous at the anode-matrix interface had been noted in the past. SEM photographs of a normal and a modified matrix from Cell Pair S/N 018 are shown in figures 50a and 50b, respectively. Figure 50a shows the normal fibrous structure from near the center of the cell matrix and figure 50b the modified structure at the anode-matrix interface. Apparently the anolyte pH is low enough to cause some attack of the asbestos at the interface, and this appears to be the reason for the matrix sticking to the anode.

The asbestos matrix is chrysotile ($Mg_6 (OH)_8 Si_4 O_{10}$), a basic magnesium silicate. Acid attack of chrysotile dissolves the magnesium leaving a silica structure. Apparently the anolyte pH of Cell Pair S/N 021 was low enough to dissolve the matrix, at a relatively rapid rate, and the silica with a negative charge migrated toward the hydrogen side of the anode.

The only difference in the Cell Pair S/N 021 build-up which could enhance matrix attack was the increase in matrix thickness from 25 to 35 mils which would result in a lower anolyte pH. The reason for the lower anolyte pH is that the cesium ion concentration decreases from cathode to anode because of the potential gradient, and at a fixed catholyte concentration the anolyte concentration would be significantly reduced in going to a 35 mil electrode separation (see figure 51). From thermodynamics it is known that the anolyte pH is a function of the cesium ion concentration and the equilibrium CO_2 partial pressure. As shown in figure 52 a reduction in the cesium ion concentration at constant P_{CO_2} results in a decrease in anolyte pH.* It is reasonable to assume that the dissolution rate of chrysotile is directly proportional to the hydrogen ion concentration, which would be equivalent to an exponential function of pH. Therefore a one unit decrease in pH would increase the dissolution rate by a factor of ten.

It can be rationalized that the increase in matrix thickness to 35 mils reduced the anolyte pH sufficiently to result in a relatively rapid matrix dissolution and subsequent blockage of the anode pores with silica. With TMAC electrolyte the problem of matrix attack is greatly reduced because the anolyte pH is higher as found from analytical cell testing. This also is supported by the fact that there is much less tendency for the asbestos matrix to bond to the anode surface in TMAC cells.

---

* The equilibrium partial pressure of CO_2 also has a large effect on anolyte pH; however, there is some evidence that the CO_2 evolution reaction is not at equilibrium, and if this is the case the CO_2 partial pressure in the hydrogen would have a much smaller effect on anolyte pH.

A
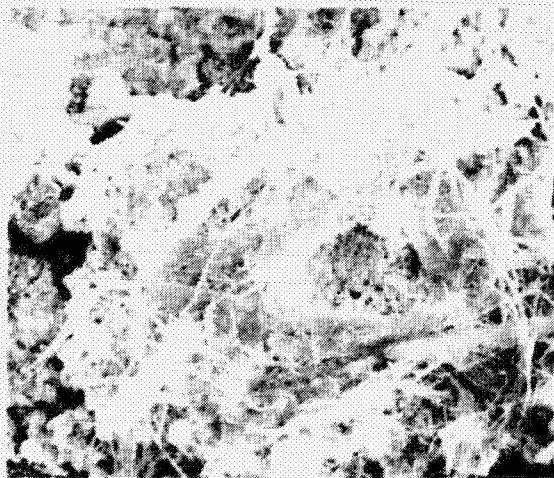
B

FIGURE 50. SEM PHOTOGRAP ... (A) AND MODIFIED (B) ASBESTOS MATRIX
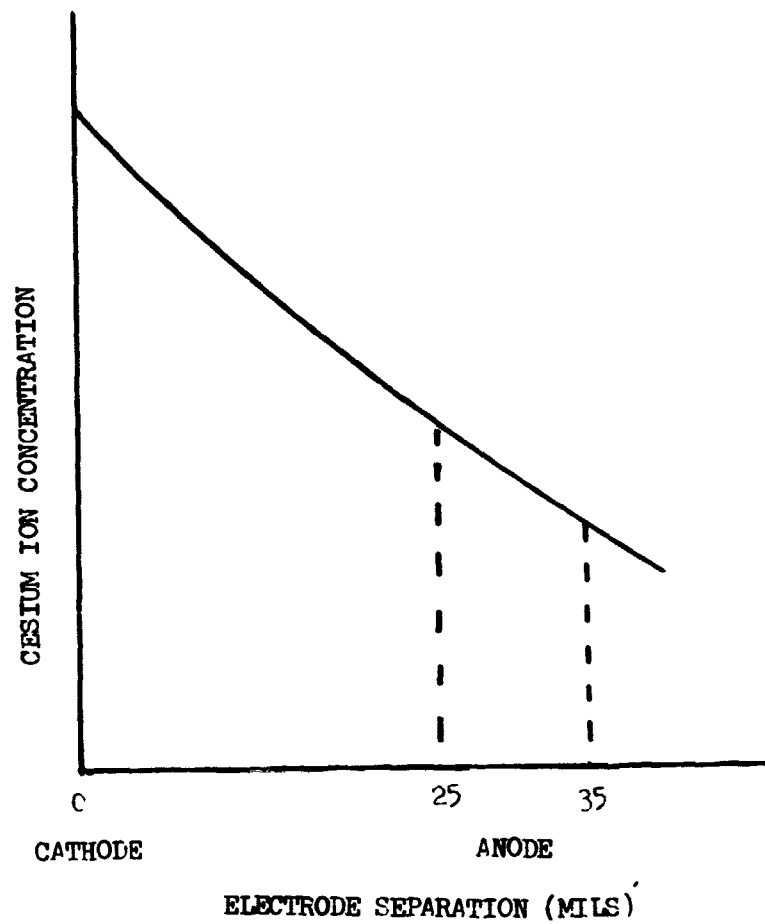
FIGURE 51. ILLUSTRATION OF CESIUM ION CONCENTRATION CHANGE WITH
ELECTRODE SEPARATION
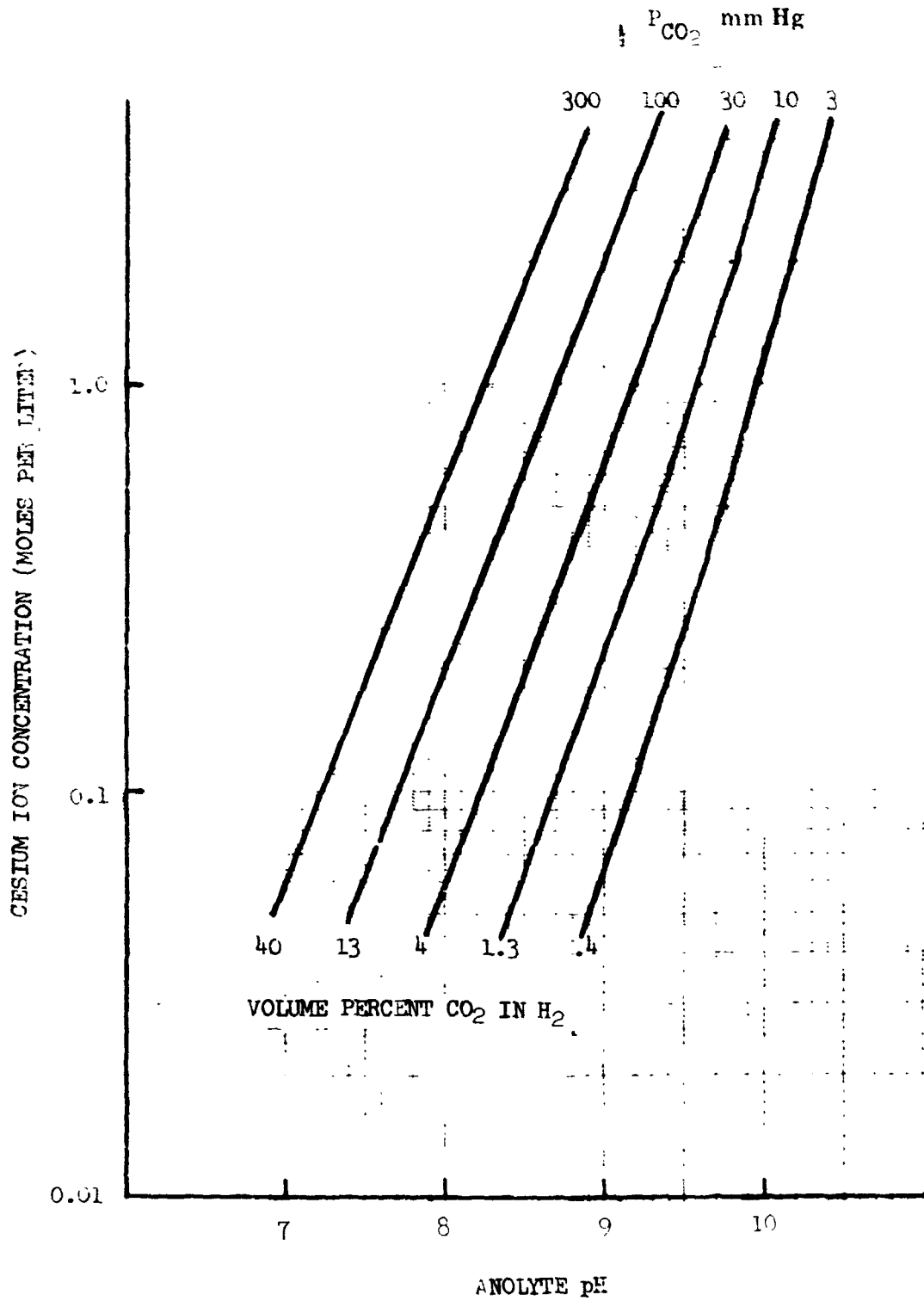
FIGURE 52. RELATIONSHIP BETWEEN CESIUM ION CONCENTRATION, ANOLYTE pH AND P_{CO_2}

A number of possibilities exist for preventing or minimizing the matrix dissolution problem in the future to achieve long term cell life.

- The electrode spacing could be reduced to 20 mils with a small sacrifice ($\approx 3\%$) in CO_2 transfer efficiency.

- A more acid resistant matrix material could be used (Neoprene asbestos).

- A dual-porosity sinter-type anode could be used to isolate the matrix from the low pH anolyte region.

The only one of these suggested changes which would eliminate the dissolution of the matrix would be the second one, a more acid resistant matrix. The suggested matrix material of Neoprene asbestos has been used in the WVE cell pair (H2SO4 electrolyte) very successfully and should be suitable for the HDC cell pair.

Cell Pair S/N 021 was reassembled on the 59th day using new electrodes and a matrix thickness of 0.025 inches. The performance of the rebuilt cell pair, at 18 asf during the remainder of the test program was a CO_2 removal efficiency of 83 percent (average) and power voltage decay of less than $50 \mu v/hour$. The noticeable decrease in power followed by a step increase during the 74th to 80th day test period resulted from first, a low chamber dew point (ΔT of 10^oF), which is on the borderline condition for forming cesium bicarbonate precipitate that caused a low cell power, and second, an increase in chamber dew point to obtain a temperature – dew point difference of 7^oF, causing an increase in power output (5.0 watt). The unit was removed from test after successfully completing 90 days of testing.

Cell Pair S/N 022 Testing

During the initial parametric testing (figure 53) of Cell Pair S/N 022 (TMAC unit) the test fixture fan failed causing the unit to operate without any cooling air which caused flooding of the unit and a permanent loss of electrolyte. For the next fifteen days of operation, after replacement of the fixture fan and addition of concentrated electrolyte to the cell pair reservoir, the unit slowly increased its output power, but not sufficiently to warrant continuation of the test. The unit was rebuilt, using the same electrodes but fresh matrix and electrolyte, and resumed testing. After the parametric testing at $52/45^oF$ (61st day) the inlet air conditions were changed to room air (temperature $\backsim 74^oF$, dew point $\backsim 60^oF$ and $P_{CO_2} \backsim 1/4$ mm Hg). The warmer inlet air and the low P_{CO_2} caused an initial increase in power, to a peak of 5 watts. After several days at this condition the cell power stabilized at 2.5 watts. The following 40 days of the testing was done at room air temperature and dew point but at 3.0 mm Hg P_{CO_3}. The average voltage decay rate of this unit for the entire test period was less than 50μ v/hour and provided and average CO_2 removal efficiency of $\backsim 72$ percent. The last portion of the test was conducted at room air conditions, including P_{CO_2}, which caused the power to increase and the CO_2 removal to decrease ($\backsim 16\%$

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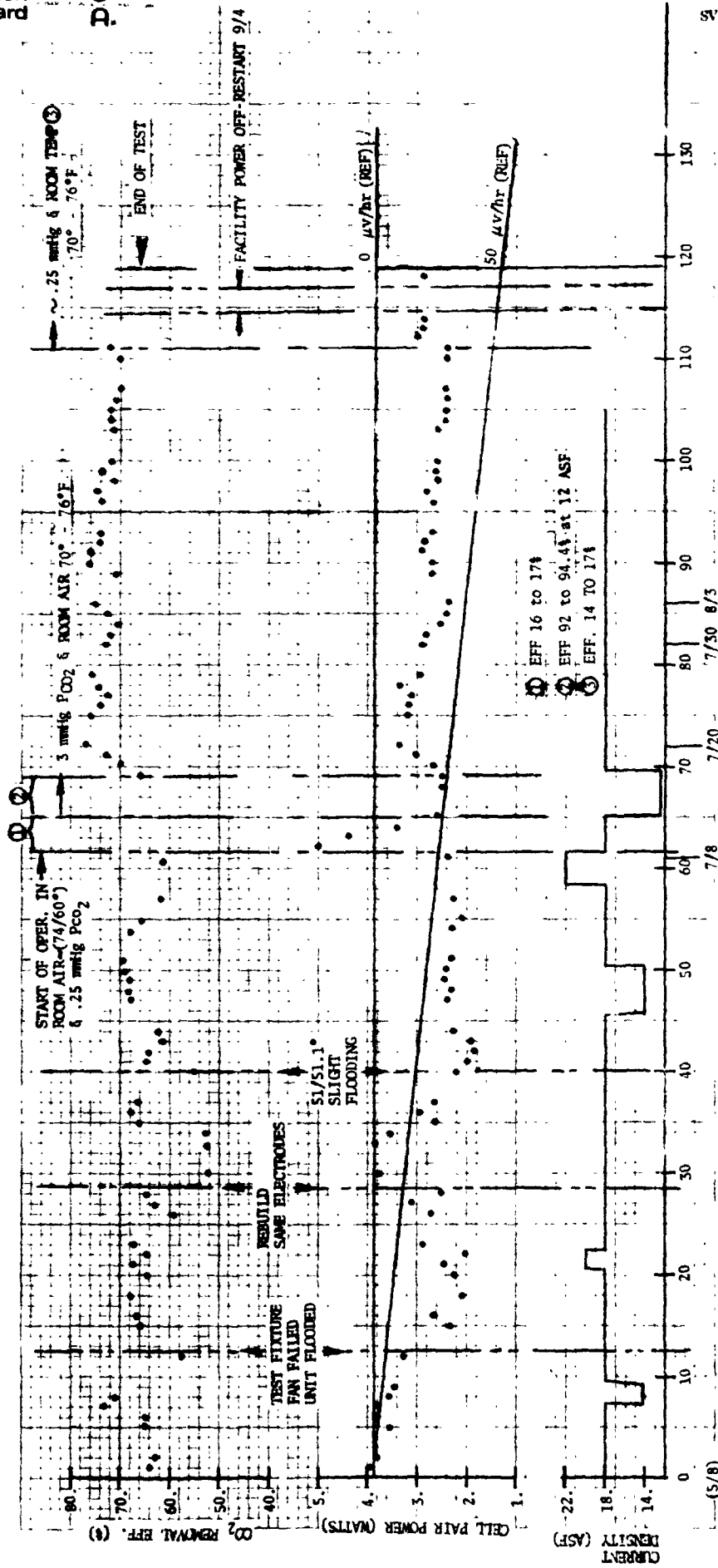
FIGURE 53. HDC CELL PAIR S/N 022 (TMAC) PERFORMANCE VS TIME

WVE CELL PAIR VIBRATION TEST

The WVE cell pair which had been assembled and tested for 3940 hours under the previous contract, NAS 9-11830, was restarted on August 30, 1972 to obtain a performance base line prior to vibration tests. The complete history of this WVE cell pair is given in Table XX. The performance prior to vibration, (figure 54) was 53 asf at 1.7 vdc for an inlet air condition of 68°F dew point and 75°F dry bulb. The unit was removed from test and vibrated in all three axes to the Apollo launch levels indicated in figures 55-57. The unit was not damaged structurally or electrochemically, did not leak, and did not release any free electrolyte from the cell. A very small amount of electrolyte (2-3 drops) was released from the reservoir area. This loss of electrolyte would not be expected in a flight type unit, with reservoir housings made of metal instead of plexiglass.

The initial performance of the WVE after vibration and 145 hours of non-operation was slightly lower than previous performance; approximately 47 asf at 1.7 v, however, the performance slowly increased as the cell pair continued to operate (figure 52). At the completion of the post vibration test, 5279 hours total operation time, the performance was 49 asf at 1.7 volts and the post vibration test was completed. This slight change in performance from the pre-vibration testing is considered acceptable.

Upon shutdown of the WVE, it was determined that the inlet to the cells hydrogen passage was blocked because the nitrogen purge gas was not purging out the hydrogen.

Visual examination of the WVE after vibration revealed some flaking of the gold plating on the outside surfaces of the center housing. The disassembly of the WVE cell pair, after the post vibration base line test, revealed that the gold plate of the titanium center housing internal to the cell also was flaking in certain areas (figure 58), and there was evidence of a slight titanium corrosion in these areas. A detail examination of the center plate by the Hamilton Standard Material Engineering group revealed that the gold plate was somewhat porous allowing gradual attack of the titanium by the sulfuric acid electrolyte which caused a weakening of the gold-titanium bond. The vibration may have contributed to the flaking in the weakened bond areas, making the problem more evident, but it certainly was not the original cause. A different type of plating procedure will be used in the future. This will provide a better gold-titanium bond and a less porous gold plate.

TABLE XX
WVE CELL PAIR HISTORY

Date	Event	NASA/MSC Contract
10/71	Hardware Manufacture	NAS 9-11830
11/18/71	Initial Assembly	NAS 9-11830
11/18-71- 12/2/71	Cell Was Operated (181 Hours)	NAS 9-11830
12/2/71	Final Assembly	NAS 9-11830
12/2/71- 5/12/72	Cell Was Operated (3940 Hours)	NAS 9-11830
5/12/72	Cell Was Removed From Test and Put In Storage	No Contract
8/30/72	Restart of Cell	NAS 9-12920
8/30/72- 10/12/72	Cell Was Operated (1028 Hours)	NAS 9-12920
10/12/72	Removed From Test For Vibration Test	NAS 9-12920
10/17/72	Cell Vibrated	NAS 9-12920
10/18/72- 10/31/72	Cell Operated (311 Hours) Post Vibration Baseline	NAS 9-12920
10/31/72	Cell Removed From Test and Disassembled	NAS 9-12920

Total Operating Time   5460 Hours

Total Time Assembled   11.5 Months

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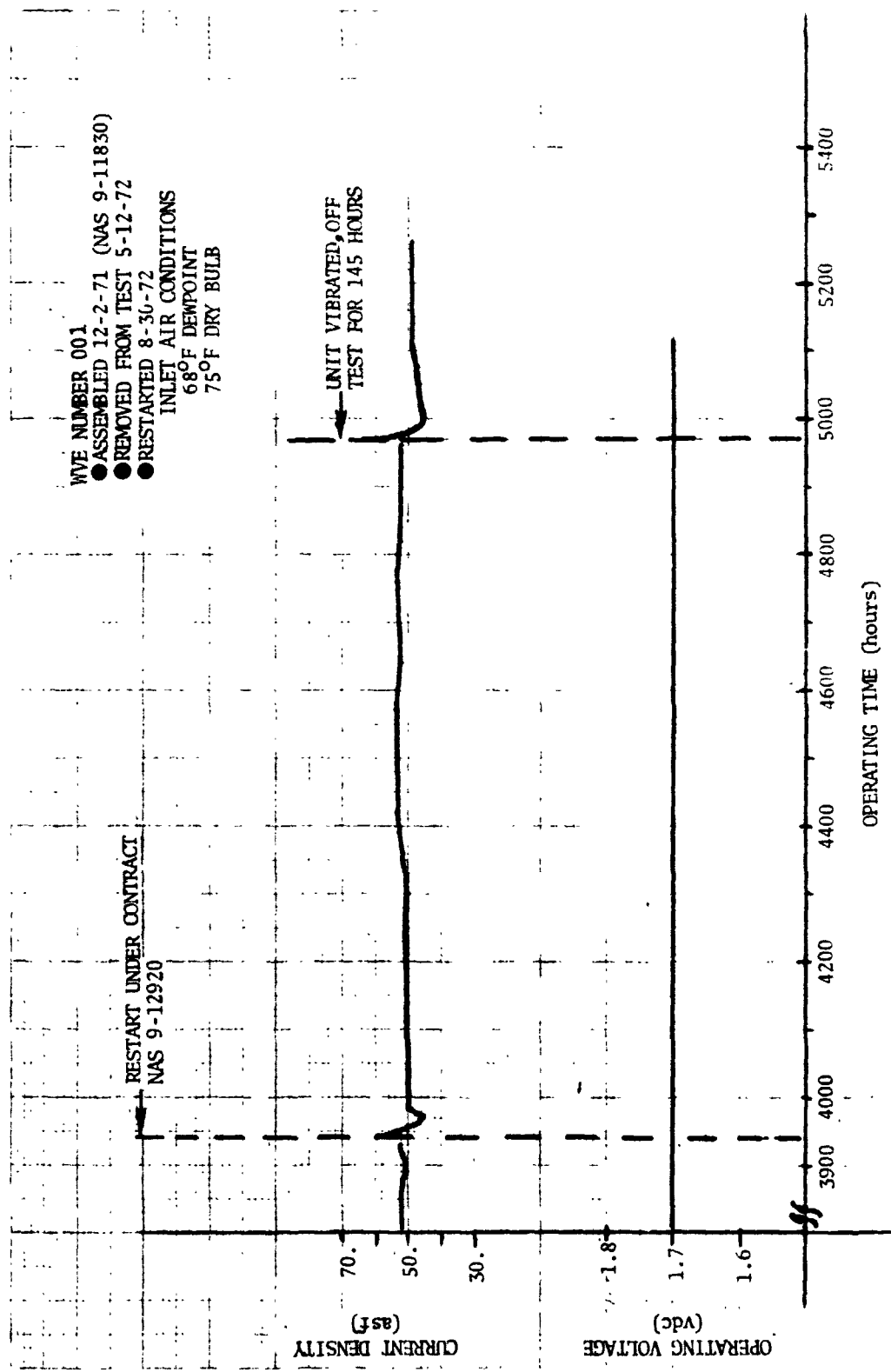
FIGURE 54. WVE PERFORMANCE DATA VERSUS OPERATING TIME

Hamilton Standard — Division of United Aircraft Corporation

HSF-1634A

RANDOM VIBRATION TEST
ANALYSIS METHOD A

PLOTTED BY: Muket
PROJECT: M6 9-12930
SPEC.: ECS-2128-6-036

CHECKED BY:
ITEM W36: Reservoir Cell Pair
PARA. PHASE: Random

TEST ENGINEER: BRADFORD
CODE: —

RIG NO.: 26
SERIAL NO.: 03
ACTION SHEET NO.: —
ATA NO.: —

WITNESS:
TYPE OF TEST: DVT
DATE: 10/17/72

WITNESS: —
TEST NO.: 49

EXCITATION ALONG X AXIS
GRMS INPUT: 11.25
NON-OPERATING
TEMP. °F: 75
PERIOD OF TEST: START ☑ END ☐
DURATION OF TEST: 2.5 MIN.
ACCEL. SERIAL NO.: WR11
ACCEL. SENSITIVITY: 2.996 MV RMS GP / COL GP
ACCEL. SENSING X AXIS
ACCEL. LOCATION: Ax
TAPE REEL NO.: 612 340
SPECIAL CONDITIONS:

REPORT NO.
SVHSER 6285

RESERVOIR
Z
X
Y
CELL PAIR

SPECTRAL DENSITY G²/HZ

FREQUENCY HZ
25 CPS BANDWIDTH

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PAGE NO.

FIGURE 55.   CELL PAIR VIBRATION TEST CURVE FOR X AXIS

Hamilton Standard
Division of United Aircraft Corporation

RANDOM VIBRATION TEST
ANALYSIS METHOD A

HSF-1634A

PLOTTED BY	CHECKED BY	TEST ENGINEER	RIG NO. 26	WITNESS
Mukesh		Mohmed		

PROJECT: NAS 9-13930
SPEC. EC6-2128-6-035

ITEM: WIE Reserve Cell Pair
CODE: —
PARA. —
PHASE: Random

SERIAL NO. 03
ACTION SHEET NO. —
ATA NO. —
TYPE OF TEST: DVT
DATE: 10/17/72

WITNESS: —

TEST NO. 6

EXCITATION ALONG Y AXIS
GRMS INPUT: 11.05
NON-OPERATING
TEMP. 75 °F
PERIOD OF TEST: ☑ START ☐ END
DURATION OF TEST: 2.5 MIN.
ACCEL. SERIAL NO. 4629
ACCEL. SENSITIVITY: MV RMS / GP — 2.617 COL / GP
ACCEL. SENSING Y AXIS
ACCEL. LOCATION: Ay
TAPE REEL NO. 612340
SPECIAL CONDITIONS

REPORT NO.
SVHSER 6285

SPECTRAL DENSITY G²/HZ

FREQUENCY HZ

25 CPS BANDWIDTH

VAR BW

10 100 150 500 1000 1500 2000

FIGURE 56.   CELL PAIR VIBRATION TEST CURVE FOR Y AXIS

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PAGE NO.

Hamilton Standard
DIVISION OF UNITED AIRCRAFT CORPORATION

RANDOM VIBRATION TEST
ANALYSIS METHOD A

HSF-1634A

PLOTTED BY	CHECKED BY	TEST ENGINEER	RIG NO.	WITNESS	
MICKET	S.M.K.	BRADFORD	2 G	—	WITNESS —
PROJECT	ITEM WVF RESERVOIR CELL PAIR	CODE —	SERIAL NO. 03	TYPE OF TEST DVT	
NAS9-12926	PARA. —	PHASE RANDOM	ACTION SHEET NO. —		
SPEC. ICC-2128-L-035			ATA NO. —	DATE 10-18-72	

SPECTRAL DENSITY G²/HZ

FREQUENCY HZ
25 CPS BANDWIDTH

VAR BW

PAGE NO.

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FIGURE 57. CELL PAIR VIBRATION TEST CURVE FOR Z AXIS

EXCITATION ALONG Z AXIS
GRMS INPUT 11.25
NON-OPERATING
TEMP. 75 °F
PERIOD OF TEST
☒ START ☐ END
DURATION OF TEST 2.5 MIN.
ACCEL. SERIAL NO. VL31
ACCEL. SENSITIVITY $\frac{MV\ RMS}{GP}$ ___ $\frac{COL}{GP}$ 2.648
ACCEL. SENSING Z AXIS
ACCEL. LOCATION A_Z

TAPE REEL NO. 012340
SPECIAL CONDITIONS

REPORT NO.
SVHSER 6285

Hamilton
Standard

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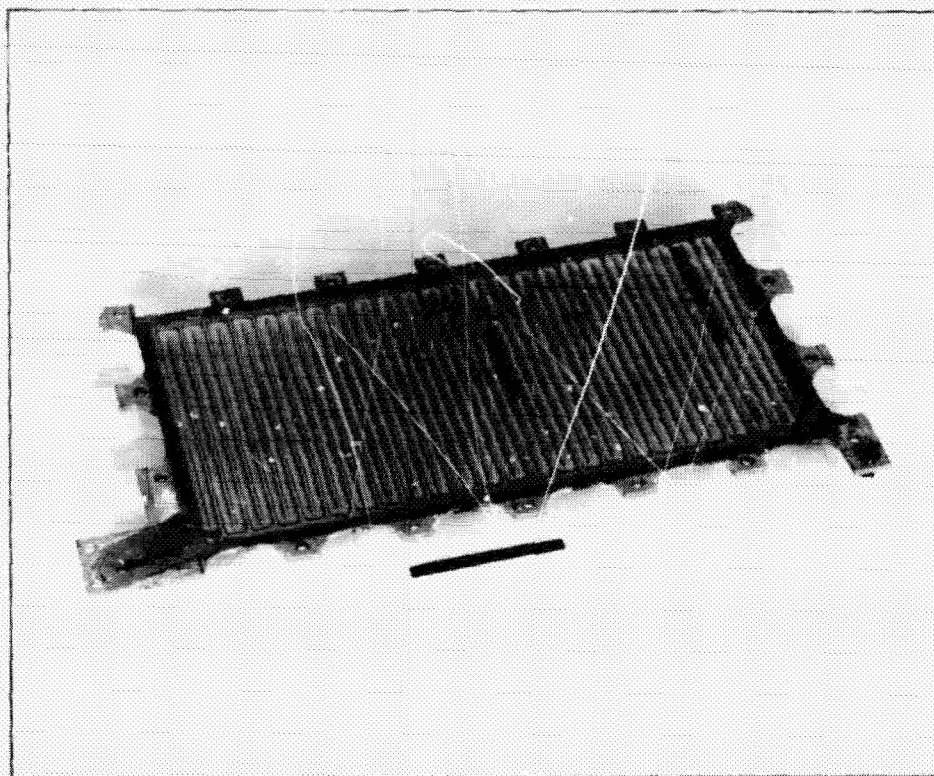
FIGURE 58. WVE CENTER HOUSING (ANODE SIDE)

WVE/HDC BREADBOARD FABRICATION

Tne Integrated WVE/HDC Breadboard unit illustrated schematically by figure 59, was designed under the previous WVE/HDC contract, NAS 9-11830, and manufactured and assembled during this program. The Integrated WVE/HDC Breadboard unit consisted of four cell pairs (2 WVE and 2 HDC), an air blower, miscellaneous valving and instrumentation incorporated into one package as shown by figure 60. This packaging provided parallel air flow through the cells, hydrogen gas manifolding between the cell pairs (HDC's in series and WVE's in parallel), provisions for isolating the HDC cell pairs, and means to monitor cell pair performance.

Testing of this unit was not conducted because of program redirection. The four cell pairs (2 WVE and 2 HDC) that were used for assembly and air flow checkout were not electrochemically functional.
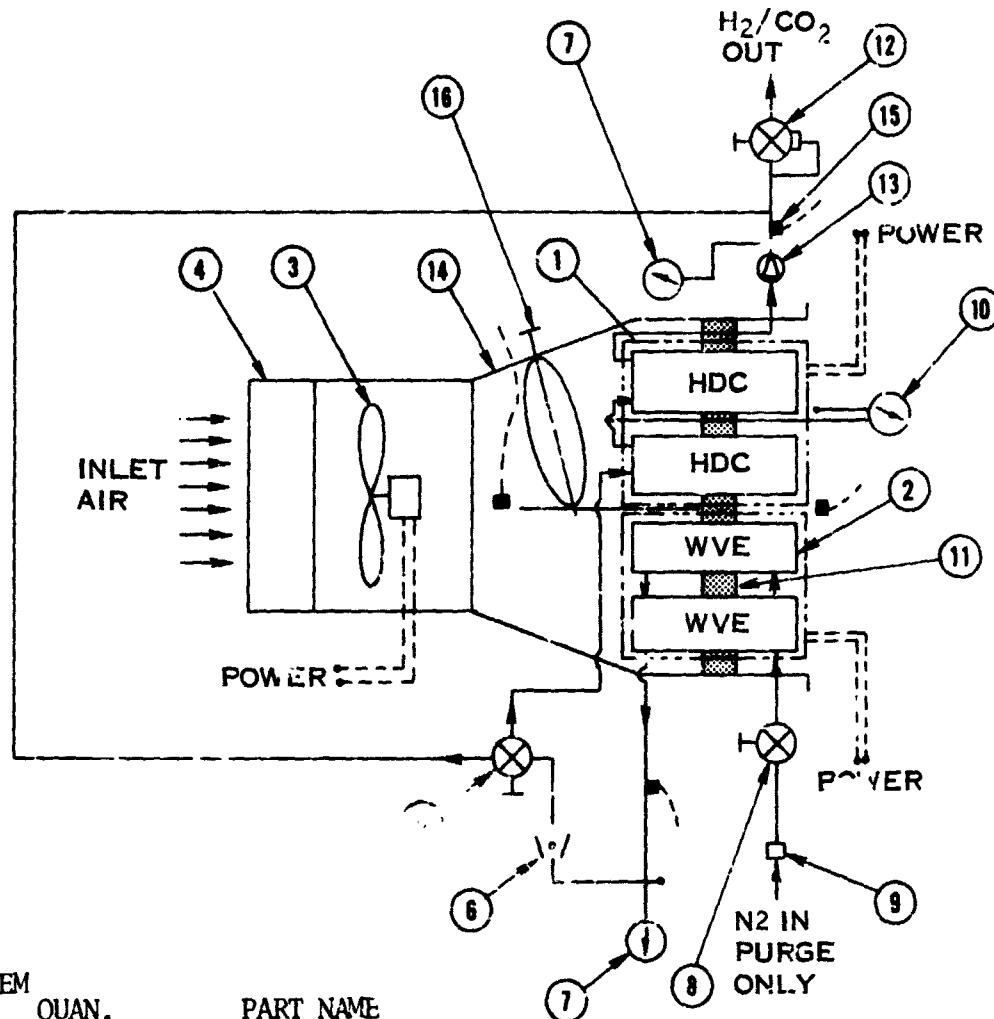
ITEM NO.	QUAN.	PART NAME
16	1	DUCT CONTROL VALVE
15	4	TEMPERATURE SENSOR
14	1	HOUSING
13	1	CHECK VALVE
12	1	BACK PRESSURE REGULATOR
11	AR	SPONGE RUBBER
10	1	DIFFERENTIAL PRESSURE GAGE
9	1	SWAGELOCK FITTING
8	1	SHUT-OFF VALVE
7	2	PRESSURE GAGE
6	1	FLOW METER
5		MANUAL VALVE 3 WAY
4	1	CHARCOAL FILTER
3	1	FAN
2	2	WATER VAPOR ELECTROLYSIS CELL
1	2	HYDROGEN DEPOLARIZED CELL

FIGURE 59. INTEGRATED WVE/HDC BREADBOARD SCHEMATIC (SVSK 83513)

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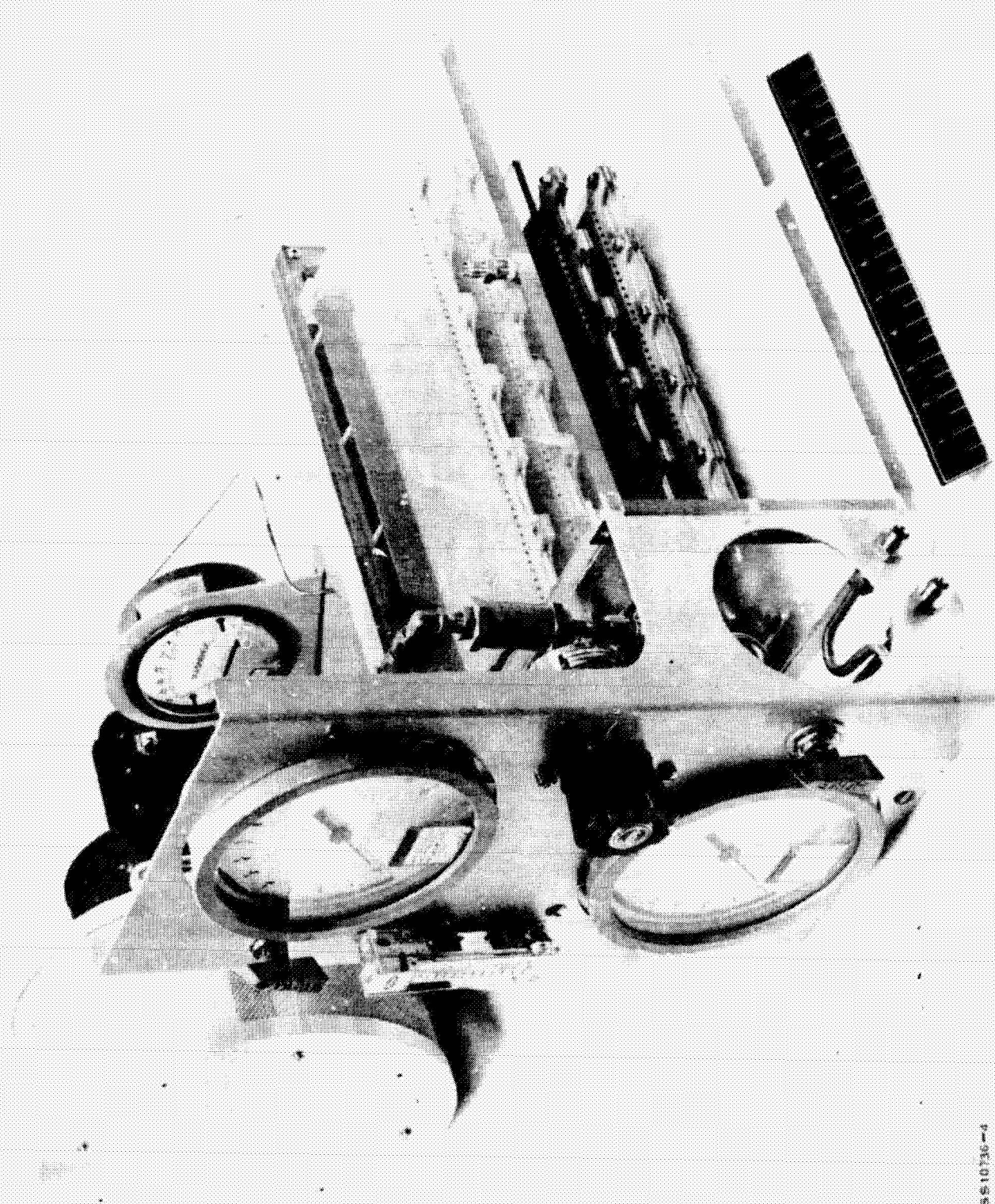
FIGURE 60. INTEGRATED WVE/HDC BREADBOARD SYSTEM

RELIABILITY/QUALITY ASSURANCE/SAFETY SUMMARY

The following is a summary of the effort performed during the various tasks of this program in the areas of Quality Assurance, Reliability and Safety.

- The successful demonstration of the WVE cell pair to withstand launch vibration loads verified that the cell pair design for both HDC and WVE systems is safe for flight use and will not release free electrolyte.

- The disassembly of the WVE cell pair (after approximately one year of assembly) revealed a poor gold plating of the center housing. Future plating of the cell pair housing will be done at Hamilton Standard where improved control can be applied to the various steps of the plating operation.

- In order to assure consistently high quality of electrodes for future cell pairs, Hamilton Standard will prepare electrode fabrication operation sheets and will use quality control surveillance during the fabrication of electrodes.

- All HDC cell pair malfunctions which occurred during testing were resolved and corrective action instituted to prevent the recurrence of each malfunction. As a result of this positive type failure analysis, the HDC has become a more reliable unit during this program.

REFERENCES

1. "Development of an Integrated Water Vapor Electrolysis Oxygen Generator and Hydrogen Depolarized Carbon Dioxide Concentrator" by J. C. Huddleston and Dr. J. R. Aylward, NASA-JSC Report No. CR-115575, Hamilton Standard, Division of United Aircraft Corporation, Windsor Locks, Connecticut, May 1972. (NASA Contract NAS9-11830)

2. "Hydrogen Depolarized Cell Pair Definition for Space Station Application" by C. R. Russell, Hamilton Standard, Division of United Aircraft Corporation, Windsor Locks, Connecticut, March 1973. (NASA Contract NAS 9-12920)

3. "A Simple Method for Measuring the Overvoltage of Hydrophobic Gas Diffusion Electrodes" by J. Giner and S. Smith, Electrochem. Tec. $\underline{5}$, 59 (1967).

APPENDIX

MASTER TEST PLAN

MASTER TEST PLAN

IMPROVED MULTI-CELLED BREADBOARD
ELECTROCHEMICAL OXYGEN-GENERATION
AND CARBON DIOXIDE CONTROL SYSTEM

PREPARED UNDER CONTRACT NAS 9-12920

by

HAMILTON STANDARD

DIVISION OF UNITED AIRCRAFT CORPORATION

WINDSOR LOCKS, CONNECTICUT

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

MANNED SPACECRAFT CENTER

HOUSTON, TEXAS

AUGUST 1972

Prepared by: _____
J. C. Huddleston
Program Engineer
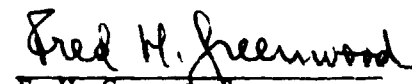
Approved by: _____
F. H. Greenwood
Program Manager

TABLE OF CONTENTS

1.0   <u>GENERAL INFORMATION</u>

1.1   <u>Scope</u>

The purpose of this Master Test Plan is to define the test
methods and equipment which will be utilized to perform the
various Water Vapor Electrolysis (WVE) and Hydrogen Depolar-
ized CO_2 Concentration (HDC) cell pair, ~~breadboard~~ and | B
electrolyte tests required under NASA/MSC contract NAS 9-12920.
This test program will be conducted at Hamilton Standard, Space
Systems Department, test facilities.

1.2   <u>Applicable Documents</u>

NASA/MSC contract NAS 9-12920
Hamilton Standard Program Operating Plan for contract NAS 9-12920
~~WVE/HDC Cell Pair Breadboard Unit, SVSK 83513~~ | B

1.3   <u>Functional Requirements</u>

Unless otherwise specified, the following operating conditions* and | B
requirements will apply to the testing of the WVE and HDC cell
pairs.

Standard operating conditions:
    Air flow through cell pair   10 SCFM
    Inlet air dry bulb temperature   70°F
    Inlet air dew point temperature   53°F
    Inlet Air CO_2 partial pressure   3 mmHg
    Oxygen concentration   20-21%

WVE operating requirements:
    Nominal current density   60 asf (min at 2.0 VDC)
    Maximum voltage   2.0 VDC
    H_2 production   Current x 7.48 scc/min
    O_2 production   Current x 3.74 scc/min

HDC operating requirements:
    Nominal current density   16 asf
    Min H_2 inflow   350 scc/min
    Current efficiency (design goal)
    @ 16 asf and 2.5 mmHg $75\% = \dfrac{CO_2 \text{ Removed (scc/min)}}{7.5 \times \text{current (asf)}}$ | B

* With exception to Table I of Test Number X, page 56. | B

A-1

1.4   Test Condition Tolerances

Unless otherwise specified, the following tolerances will
apply to the referenced test parameters:

Temperature	± 1°F
Pressure	± .1 psia
	± .02 inches of water
Dew Point	± 1°F
Voltage	± 0.01 volts
Current	+ 0.10 amps
CO_2 Level	± 2%
Flow Measurement	± 2.8%

2.0   TEST PLAN AND PROCEDURES

The test plans and procedures for the following tests are
detailed in the corresponding sections as noted:

	TEST	SECTION	
~~WVE/HDC Breadboard (WBS 1.0)~~		~~I~~	B
WVE Vibration Test (WBS 5.0)		II	
HDC Flow Test (WBS 6.0)		III	
Matrix Compression Test (WBS 3.3)		IV	
TMAC Electrolyte Analytical Tests (WBS 3.4)		V	
Trace Impurities (WBS 3.5)		VI	
Cs_2CO_3 Electrolyte Analytical Tests (WBS 3.6)		VII	
HDC Matrix Compression Set (WBS 9A)		VIII	
HDC Analytical Cell Tests (WBS 9A		IX	B
HDC Cell Pair Testing (WBS 9B)		X	

2.1   Test Facility and Instrumentation Readings

The schematic of the test set-up for the various electrolyte,
cell, and cell pair tests,   is defined in the detail test |B
procedure for each test. The general data acquisition for all
the testing is presented in Table I.

Data Point	Instrument	Range & Readout
Inlet & Outlet Air Temperature of Cell Pair (°F)	Thermocouple and Bristol Recorder	0° to 250°F (1°F inc.)
Inlet & Outlet Air Dew Point of Cell Pair (°F)	Cambridge 880 Dew Pointer	-40° to 120°F (2°F inc.)
WVE Hydrogen Outlet Temperature (°F)	Thermocouple & Bristol	0° to 250°F (1°F inc.)
WVE Hydrogen Outlet Dew Point (°F)	Cambridge 880 Dew Pointer	-40°F to 120°F (2°F inc.)
HDC H_2 & CO_2 Outlet Temperature (°F)	Thermocouple & Bristol	-30°F to 250°F (1°F inc.)
HDC CO_2 Inlet Temperature (°F)	Thermocouple & Bristol	-30°F to 250°F (1°F inc.)
HDC Hydrogen Inlet Temperature (°F)	Thermocouple & Bristol	-30°F to 250°F (1°F inc.)
Chamber Pressure (inches of H_2O)	Slant Water Manometer	0 to 2 in. of H_2O (0.02 inc.)
Item Fan ΔP (inches of H_2O)	Slant Water Manometer	0 to 4 in. of H_2O (0.01 inc.)
WVE Hydrogen Outlet Pressure (psia)	HISE Pressure Gauge	0 to 30 psia (0.1 psia inc.)
HDC Hydrogen Inlet Pressure (psia)	HISE Pressure Gauge	0 to 30 psia (0.1 psia inc.)
HDC CO_2 Inlet Pressure (psia)	HISE Pressure Gauge	0 to 50 psia (0.1 psia inc.)
HDC H_2/CO_2 Outlet Pressure (psia)	HISE Pressure Gauge	0 to 30 psia (0.1 psia inc.)
Cell Pair Voltage (Volts)	Digital Meter	0 to 4.0 volts (.0005 volts inc.)
Cell Pair Current (amps)	Digital Meter	0 to 100 amps (0.05 amp inc.)
HDC CO_2 Inlet Concentration (%)	Lira Gas Analyzer	0% to 1% (0.02% inc.)

DATA ACQUISITION

TABLE I

A-4

Data Point	Instrument	Range & Readout
HDC - CO_2 Outlet Concentration in H_2 stream (%)	Lira Gas Analyzer	0% to 100% (2% inc.)
WVE Outlet Hydrogen Flow (cc/min.)	Water Displacement Measurement	1.0 cc inc. and 0.001 min. inc.
HDC - H_2/CO_2 Outlet Flow (cc/min.)	Water Displacement Measurement	1.0 cc inc. and 0.001 min. inc.
HDC - Inlet CO_2 Flow (%)	Flowmeter	0 to 16% (.2 inc.)
HDC - Hydrogen Inlet Flow (%)	Flowmeter	0 to 20% (0.2 inc.)
Chamber Gas O_2 Concentration (%)	Beckman O_2 Analyzer	0 to 100% (2% inc.)

TABLE I (Continued)

~~SECTION I~~

(DELETED)

~~AVE/HDC BREADBOARD~~

TEST PLAN

~~(WBS 1.2)~~

(Pages 6-20 deleted with Revision B)

WVE/HDC BREADBOARD TESTS (WBS 1.2)

The test plan for the WVE/HDC Breadboard Unit is presented in two parts, the conditioning of the cell pairs, and the actual operation of the WVE/HDC breadboard.

SCHEDULE

Breadboard Assembly
and Rig Modifications

| Cell Pair | Breadboard | |
| Conditioning | Checkout | Breadboard 90 Day Endurance |

```
/  2  3  4  5  6     8    /0    /2    /4    /6      /8
```

WEEKS
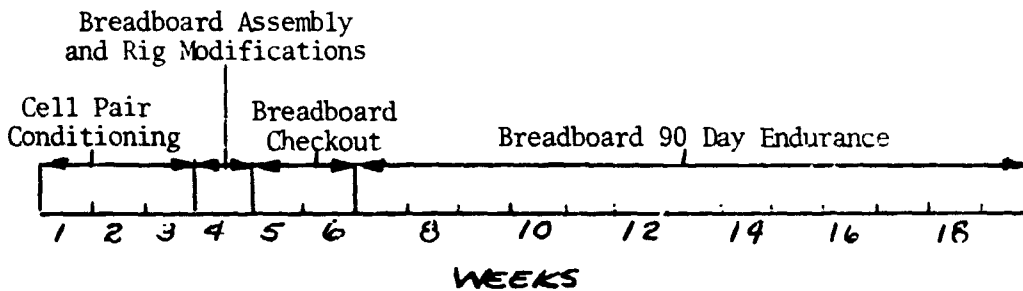
TEST FACILITIES

The test rig, as illustrated in figures 1 and 2, will be used for the cell pair and breadboard testing. The lira instrumentation of this test rig will be calibrated daily during the HDC cell pair and breadboard tests.

OVERALL TEST PHILOSOPHY

Any changes in the cell pair or breadboard configuration, and in the testing procedures or parameters will be reviewed with NASA/MSC prior to the change. In emergencies where changes must be made to protect the test unit, corrective action can be made without prior review with NASA/MSC. However, a full review of the incident will be made within 24 hours.

A-7

TEST FACILITY

FIGURE 1

Hamilton
Standard DIVISION OF UNITED AIRCRAFT CORPORATION

U
Ae
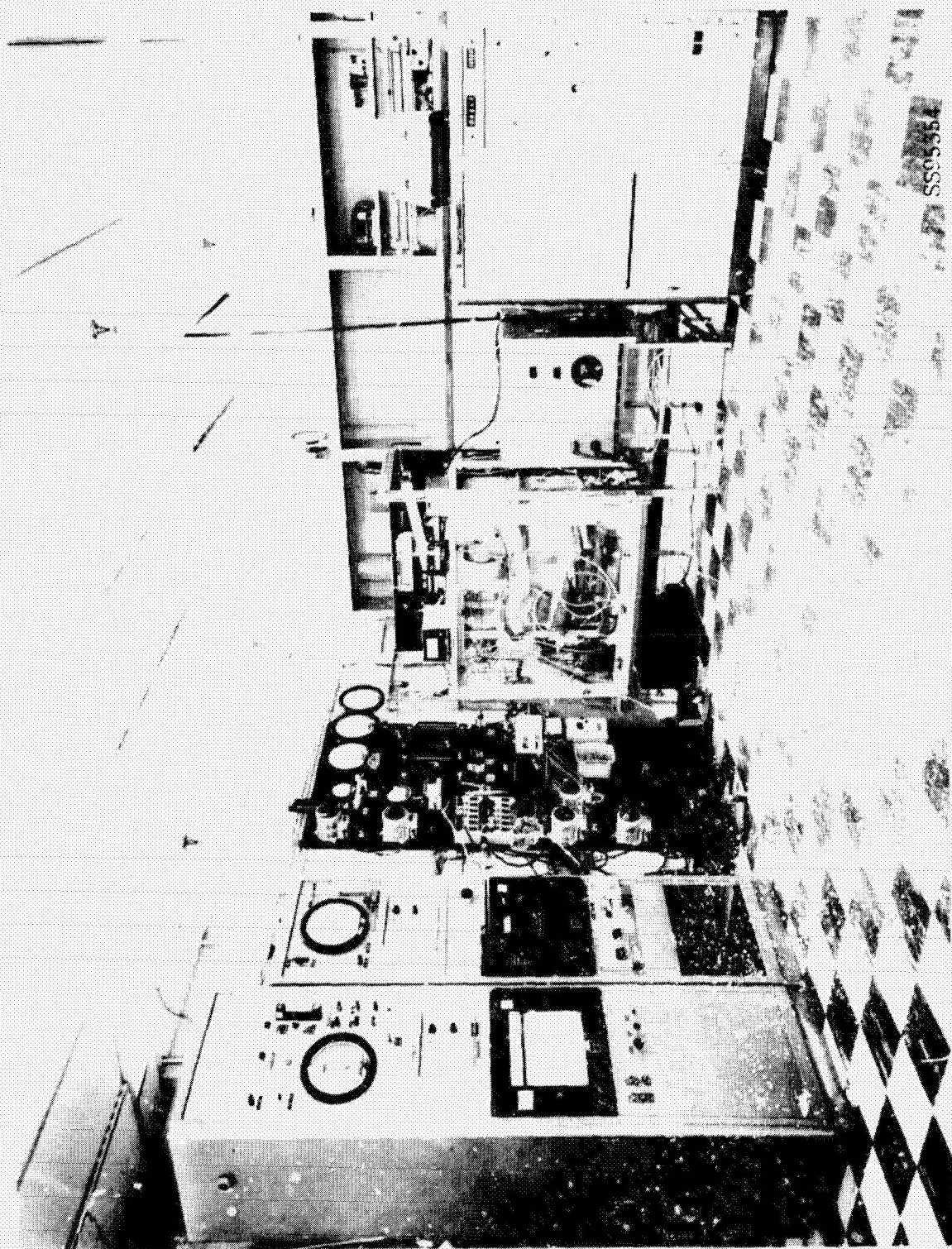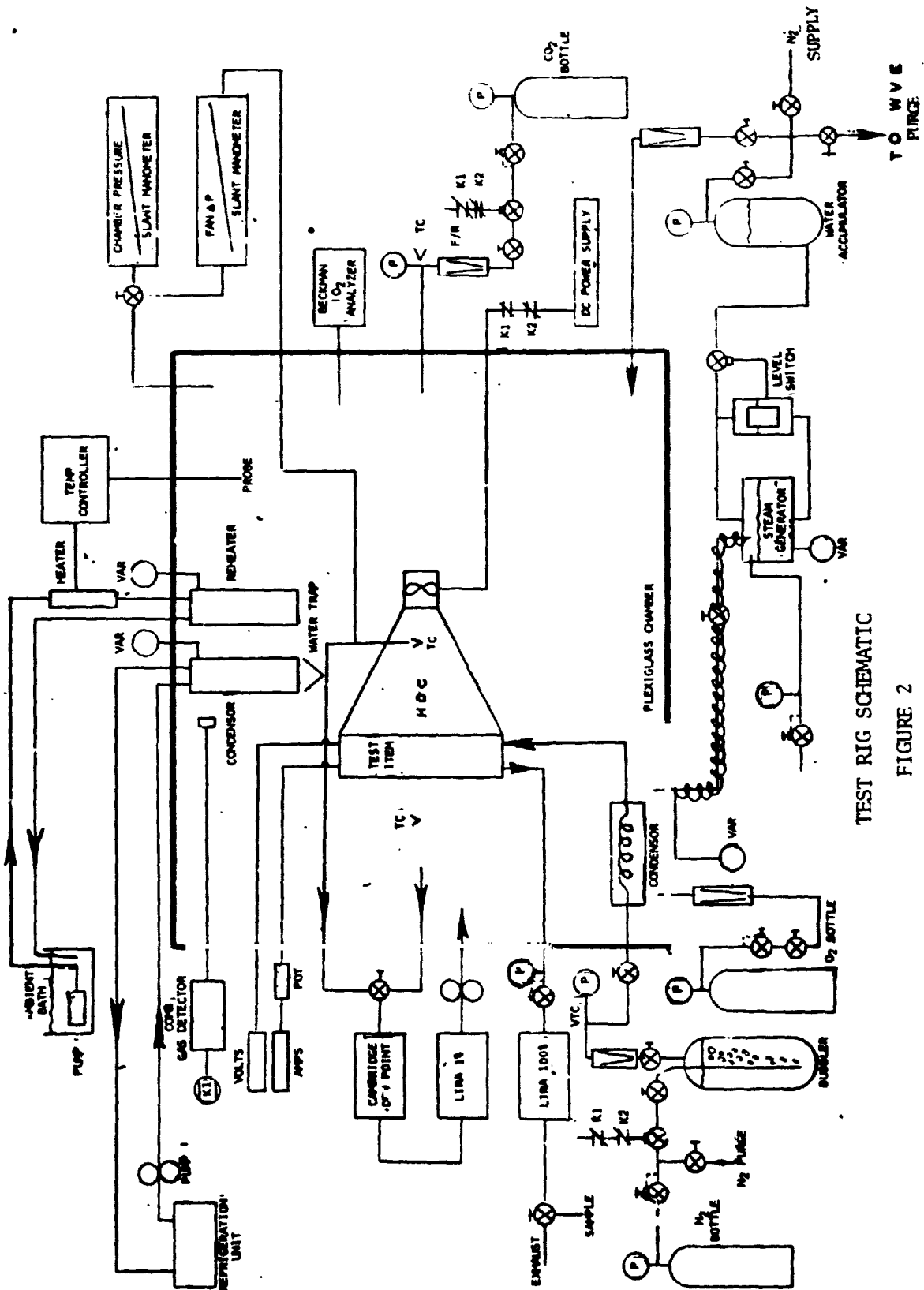
TEST RIG SCHEMATIC

FIGURE 2
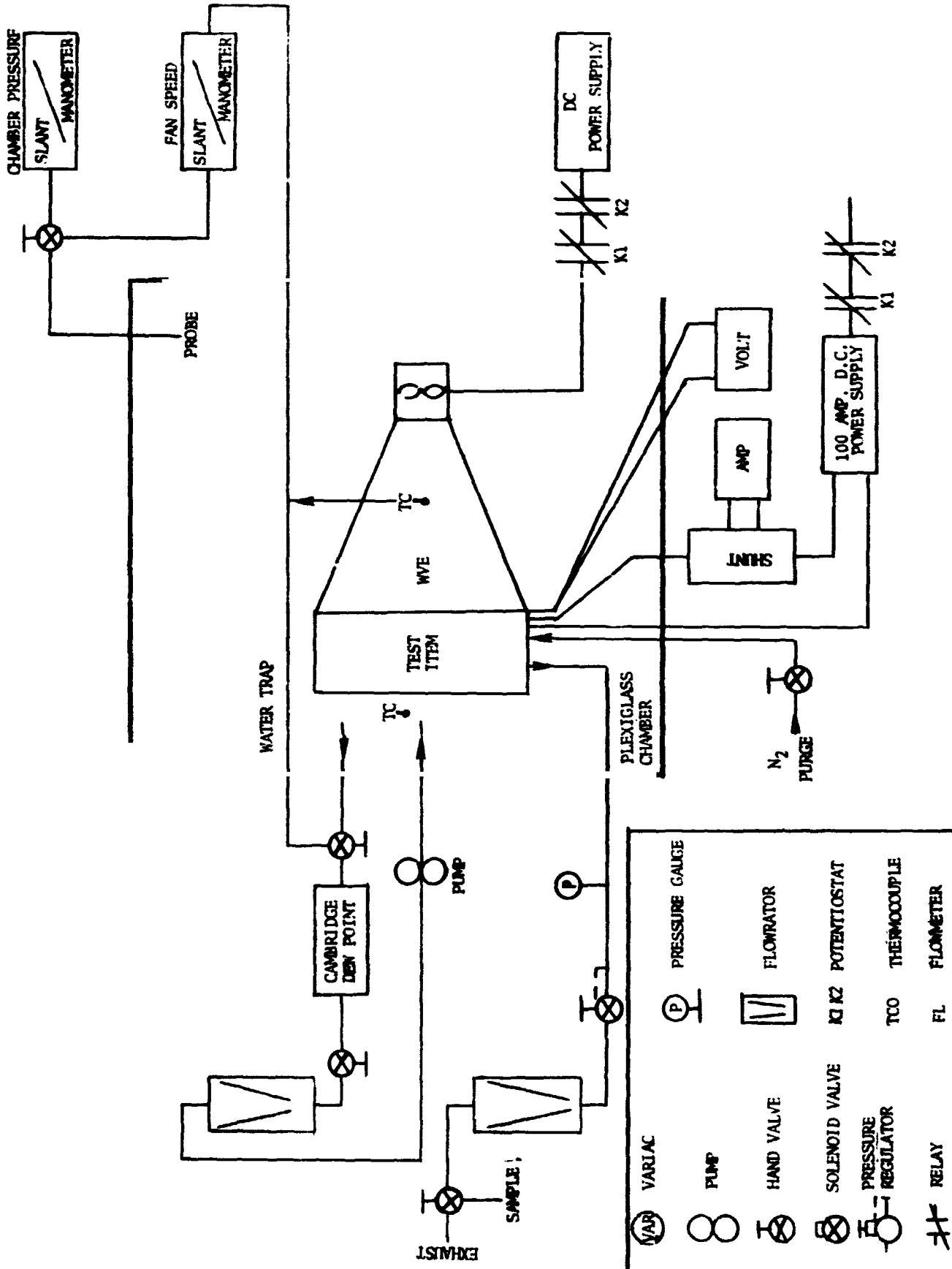
**Hamilton
Standard**

FIGURE 2 (Continued)

HS F-927 6/56

TEST NO. _____I-A_____   HAMILTON STANDARD   PAGE 1 OF _____

PLAN OF TEST

JOB: __WVE and HDC Cell Pair Conditioning Tests__   PLAN PREPARED BY: __J. Huddleston__

PROJECT & ORDER: __NAS 9-12920__   APPROVED BY: _____

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. ITEM BEING TESTED: The WVE and HDC cell pairs including the electrolyte
reservoir (reference figure 1). This testing will be done on each |A
cell pair.

2. PURPOSE OF TEST: The conditioning testing of the cell pairs establishes
the maximum relative humidity which the unit can operate under. The
excess electrolyte is also removed from the air passageways of the cell
during this period.

3. TEST PROCEDURE: The tests will be conducted in the WVE/HDC test rig,
using the cell pair test fixture (reference figure 2) under nominal cell
operating mode and inlet air conditions as presented in Table I. Data
will be taken three times daily and presented on log sheets per figures
3 and 4.

4. TEST RUNS: The duration of this test will be approximately one week or
until the unit shows that i_L has stabilized. N_2 purging of the H_2 passages |A
will be investigated during the cell pair testing.

5. SPECIAL INSTRUCTIONS: None.

6. PRESENTATION OF DATA: The performance will be graphically presented vs. time.

ELECTRICAL TERMINALS

RESERVOIR

AIR INLET

CELL PAIR

AIR OUTLET

SS10110-4

TYPICAL WVE OR HDC CELL PAIR

FIGURE 1

A-12
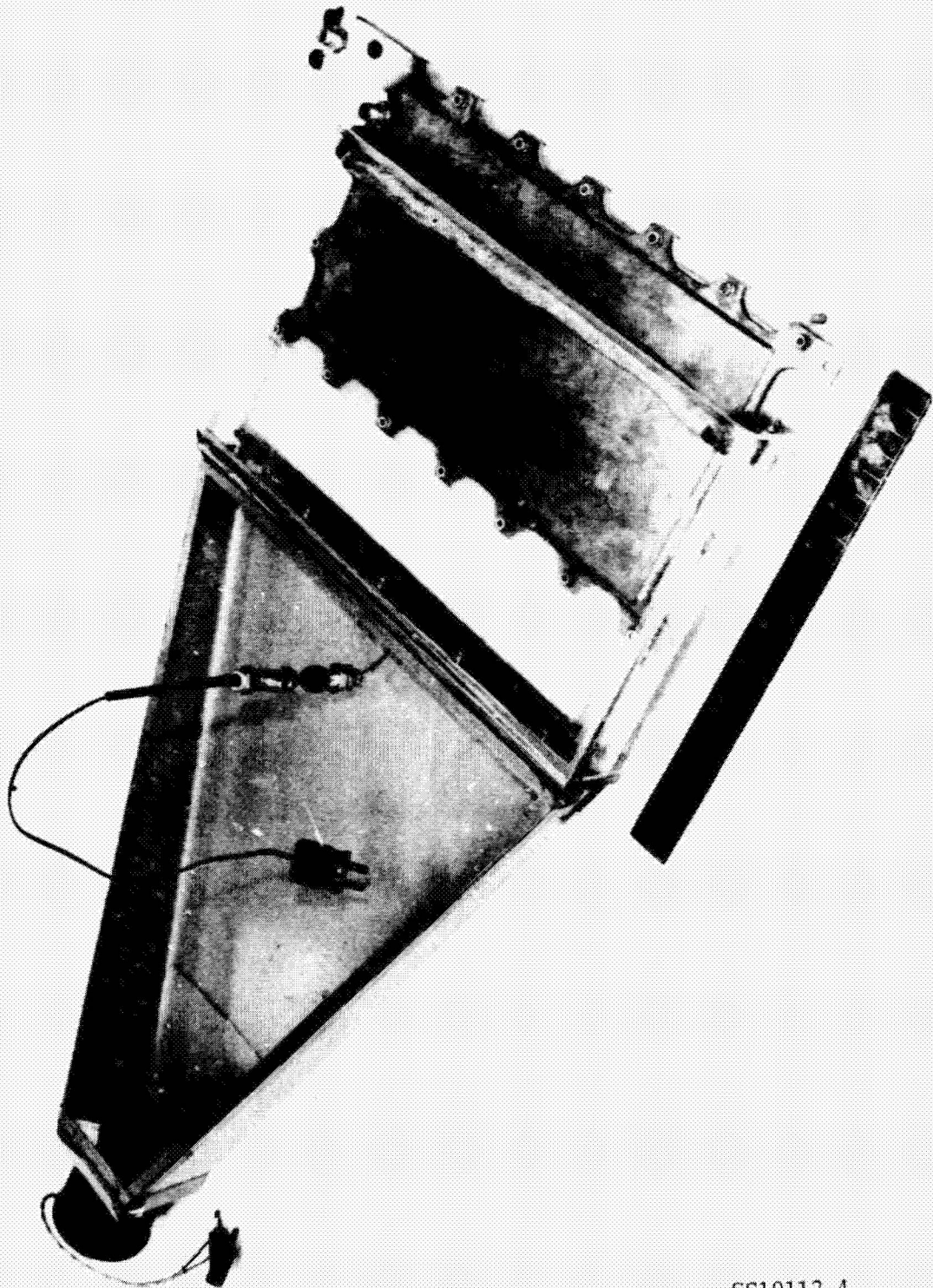
SS10112-4

CELL PAIR AND TEST FIXTURE

FIGURE 2

A-13

No.	Test Conditions	WVE Cell Pair	HDC Cell Pair
	Set Conditions		
1	Inlet Air Temperature	70°F	70°F
2	Inlet Air Dewpoint	65°F	65°F
3	Inlet Air CO_2 Partial Pressure	---	3mmHg
4	Inlet Air O_2 Concentration	20-21%	20-21%
5	Air Flow Through Cell	10 scfm	10 scfm
6	▲P Across Matrix	1.0 psi	1.0 psi
7	Voltage	1.7 VDC	---
8	Current	---	16 ASF
9	H_2 Inflow	---	320 cc/min
	Monitored Parameters		
	All of the Set Conditions	x	x
	H_2 Out Flow	x	-
	H_2 + CO_2 Out Flow	-	x
	CO_2 Removal Rate	-	x
	Current Efficiency	-	x
	Current	x	x
	Voltage	x	x
	Chamber Pressure	x	x
	N_2 Inflow (Diluent)	x	-
	H_2 Temperature (out)	x	-

A

TEST CONDITIONS

TABLE I

A-14

8001

Hamilton Standard
DIVISION OF UNITED AIRCRAFT CORPORATION
WINDSOR LOCKS, CONNECTICUT 06096

SPACE & LIFE SYSTEMS LABORATORY

LOG OF TEST

WVG PERFORMANCE

TEST PLAN NO.

TEST ENGINEER

MODEL NO.

NAME OF RIG

PART NO.

SERIAL NO.

PROJECT & ENG. ORDER NO. NAS9-12920

OPERATORS

DATE	ACT TIME	ACC TIME HRS	D.P. IN °F	T IN °F	T OUT °F	T H₂ °F	D.P. H₂ °F	CHMB PRESS ΔP "H₂O	FAN ΔP "H₂O	H₂ BACK PRESS PSIG	H₂ FLOW C/MIN	N₂ DILUENT FLOW %	O₂ PURCHM GANGER %	VOLTAGE VDC	CURRENT ASF					

Handwritten column descriptions:
- ACCUMULATIVE RUNNING TIME
- DEWPOINT OF INLET AIR
- DRYBULB TEMP. OF INLET AIR
- DRYBULB TEMP. OF OUTLET AIR
- DRYBULB TEMP. OF OUTLET HYDROGEN
- DEWPOINT OF OUTLET HYDROGEN
- TEST CHAMBER PRESSURE
- ΔP ACROSS CELL PACK - USING THIS VALUE WITH A ΔP VS FLOW CALIBRATION CURVE GIVES AIR FLOW
- HYDROGEN BACK PRESSURE ON C.L.
- HYDROGEN FLOW
- N₂ DILUENT GAS INFLOW
- OXYGEN CONCENTRATION (OXYGEN GANGER)
- INPUT VOLTAGE
- INPUT CURRENT DENSITY

REMARKS:

FIGURE 3

A-15

Hamilton Standard DIVISION OF UNITED AIRCRAFT CORPORATION
WINDSOR LOCKS, CONNECTICUT 06096

SPACE & LIFE SYSTEMS LABORATORY

LOG OF TEST

TYPE OF TEST: HDC PERFORMANCE

TEST ENGINEER

NAME OF RIG

PROJECT & ENG. ORDER NO. NAS 9-12920

SHEET ___ OF ___
TEST PLAN NO.
MODEL NO.
PART NO.
SERIAL NO.
OPERATORS

CURRENT EFFICIENCY = $\dfrac{CO_2 \ REMOVAL \ (Cc/min)}{7.5 \ | \ CURRENT \ (ASF)}$

CO₂ REMOVAL RATE

% CO₂ IN H₂+CO₂ OUTLET GAS

LIRA READING OF OUTLET H₂+CO₂ GAS FLOW

CELL PAIR POWER

CELL CURRENT DENSITY

CELL VOLTAGE

LIRA READING OF CHAMBER AIR

H₂+CO₂ OUTLET FLOW RATE

H₂ INFLOW TO CELL

HYDROGEN BACK PRESSURE ON CELL

DATE	ACT TIME	OO TIME	D.P. I.O	T IN	T OUT	T H₂	CHAMB PRESS	H₂ BACK PRESS	H₂ INFLOW	H₂+CO₂ FLOW	LIRA CHAMB	VOLTAGE	CURRENT	POWER	H₂ W.R.i	% CO₂	CO₂ REMOV	EFF
	MRS	°F	°F	°F	°F	"H₂O	"H₂O	PSIA	C#/min	C#/min	%	VDC	ASF	WATTS	%	%	Cc/min	%

* Note when lira calibration (date & time) is made.

FIGURE 4

8001

MS F-927 6/96

TEST NO. **I-B**

HAMILTON STANDARD

PLAN OF TEST

JOB: __WVE/HDC Integrated Breadboard Testing__   PLAN PREPARED BY: __J. Huddleston__

PROJECT & ORDER: __NAS 9-12920__   APPROVED BY: _____

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.

<u>NUMBER ENTRY AS LISTED ABOVE AND DESCRIBE BELOW</u>

1. ITEM BEING TESTED: Integrated WVE/HDC Breadboard Assembly, SVSK 83513 (reference schematic figure 1).

2. PURPOSE OF TEST: To obtain design data of an Integrated WVE/HDC Multi-Celled Unit which will be used in the design of large multi-celled assemblies.

3. TEST PROCEDURES: The breadboard unit will be tested in the WVE/HDC test rig. Testing will consist of a two week checkout followed by a 90 day endurance test. The test parameters for these two test periods are outlined in Table I. The procedure for nitrogen purge of the HDC cell pairs, established during cell pair conditioning, will be incorporated into this test program. Data will be taken three times daily and presented on log sheets per figure 2.

4. TEST RUNS: The duration of each test is specified on Table 1.

5. SPECIAL INSTRUCTIONS: None.

6. PRESENTATION OF DATA: The performance of the WVE and HDC units will be graphically presented.
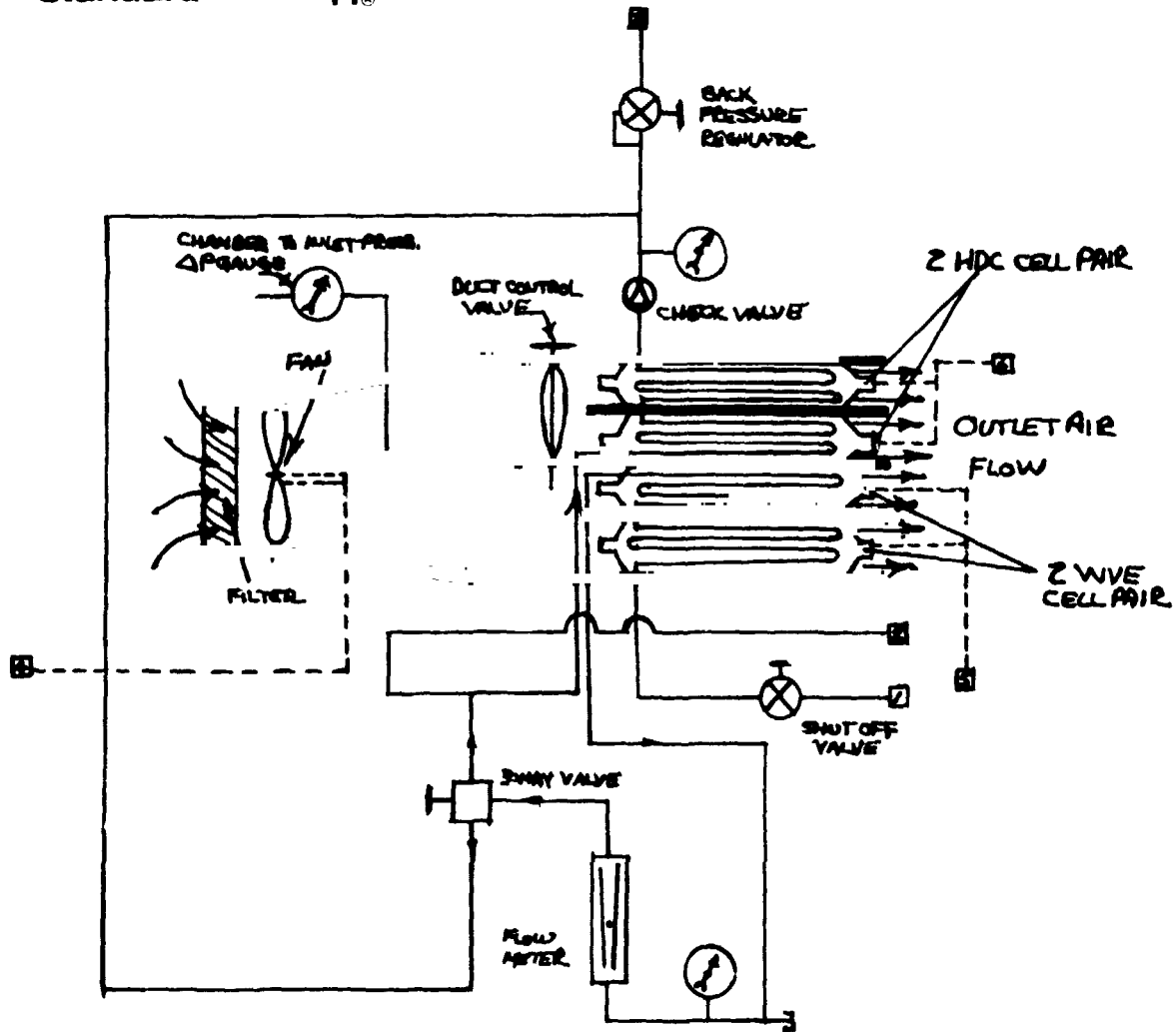
Rig to Breadboard Interface:

[1] N2 Purge Control Pressure Supply
[2] N2 Purge Control Pressure Supply
[3] To Lira and Total Flow Measuring
[4] Controlled AC Power to Fan
[5] DC Power Input to WVE Cell Pairs
[6] DC Power Output from HDC to Control Source in Rig

MULTI-CELLED WVE/HDC BREADBOARD UNIT

FIGURE 1

A-18

Hamilton Standard
DIVISION OF UNITED AIRCRAFT CORPORATION
WINDSOR LOCKS, CONNECTICUT 06096

SPACE & LIFE SYSTEMS LABORATORY
LOG OF TEST

TEST ENGINEER

MODEL NO

PART NO

SERIAL NO

OPERATORS

NAME OF RIG

PROJECT & ENG. ORDER NO. NAS9-12920

REMARKS:

DATE AST TIME	Gvo TIME	D.P. IN	T_{IN}	T_{OUT}	TH_3 VLVE	CHMB LEA	CHMB PRESS.	O_2 CONC.	FAN ΔP	H_2 FLOW VLVE	$H_2T CO_2$ FLOW HDC	H_2 PRESS VLVE	H_2 PRESS HDC	PUMP VOLTAGE	PUMP CURRENT	HDC VOLTAGE	HDC CURRENT	HDC POWER	H_2 USA	% CC1	CO_2 REMOV	EFF.
HRS'	°F	IN	°F	°F	°F	%	'H_2O	%	"H_2O	%	c/min	PSA	PSA	VDC	ASF	VDC	ASF	WATTS	%	%c	c/min	%

FIGURE 2

Hamilton Standard U A® DIVISION OF UNITED AIRCRAFT CORPORATION

NOMINAL CELL PAIR OPERATING CONDITIONS:

WVE	Constant Voltage	1.7 VDC
	Air Flow	10 scfm
	H_2 Back Pressure	1.0 psig

HDC	Constant Current	16 ASF
	Air Flow	10 scfm
	P Across Matrix	1.0 psi
	H_2 Inflow	320 cc/min
	CO_2 Partial Pressure	3mmHg

Test No.	Inlet Temp. °F	Inlet Dew Point °F	Change to nominal cell pair operation HDC	WVE	Duration of Test
Checkout	70	53	Basically nominal operation - however all adjustable parameters will be checked out during this test period.		
Endurance					
A	70	41	Nominal	Nominal	1 wk.
B_1	70	61	Nominal		1 wk.
B_2			P_{CO_2} = 2.0 mmHg		
B_3			P_{CO_2} = 1.0		
B_4			P_{CO_2} = 0.5		
B_5			P_{CO_2} = 0.2		2 days per
B_6			P_{CO_2} = 1.0		P_{CO_2} change
B_7			P_{CO_2} = 2.0		
B_8			P_{CO_2} = 3.0		
C_1			System H_2 Back Pressure	2.5 psi	
C_2				5.0 psi	4 days per
C_3				2.5 psi	press. change
C_4				1.0 psi	
D	70	41	Nominal	Nominal	1 wk.
D_1			P_{CO_2} = 2.0 mmHg		2 days
D_2			P_{CO_2} = 1.0 mmHg		2 days
E	70	61	Nominal	Nominal	1 wk.
F	70	61	Nominal/Cyclic*	Nominal/Cyclic*	1 wk.
G	70	61	Nominal	Nominal	3 days
H			(Conditions TBD after review of data through test F.)		2½ wks.

* Cyclic operation: during this mode, the power to the WVE will be removed and the WVE cells closed circuited. The air flow through the cells will be left on and the HDC left closed circuited. If the H_2 back pressure decays, nitrogen will be used to maintain the back pressure. Cycle time will be 60 min on, 30 min off.

TEST CONDITIONS

TABLE I

SECTION II

WVE VIBRATION

TEST PLAN

(WBS 5.1 and 5.2)

as F-027 6/50

TEST NO. ___II___   **HAMILTON STANDARD**   PAGE 1 OF _____

PLAN OF TEST

JOB: ___WVE Vibration Test (WBS 5.1)___   PLAN PREPARED BY: __J. Huddleston__

PROJECT & ORDER: __NAS 9-12920__   APPROVED BY: _____
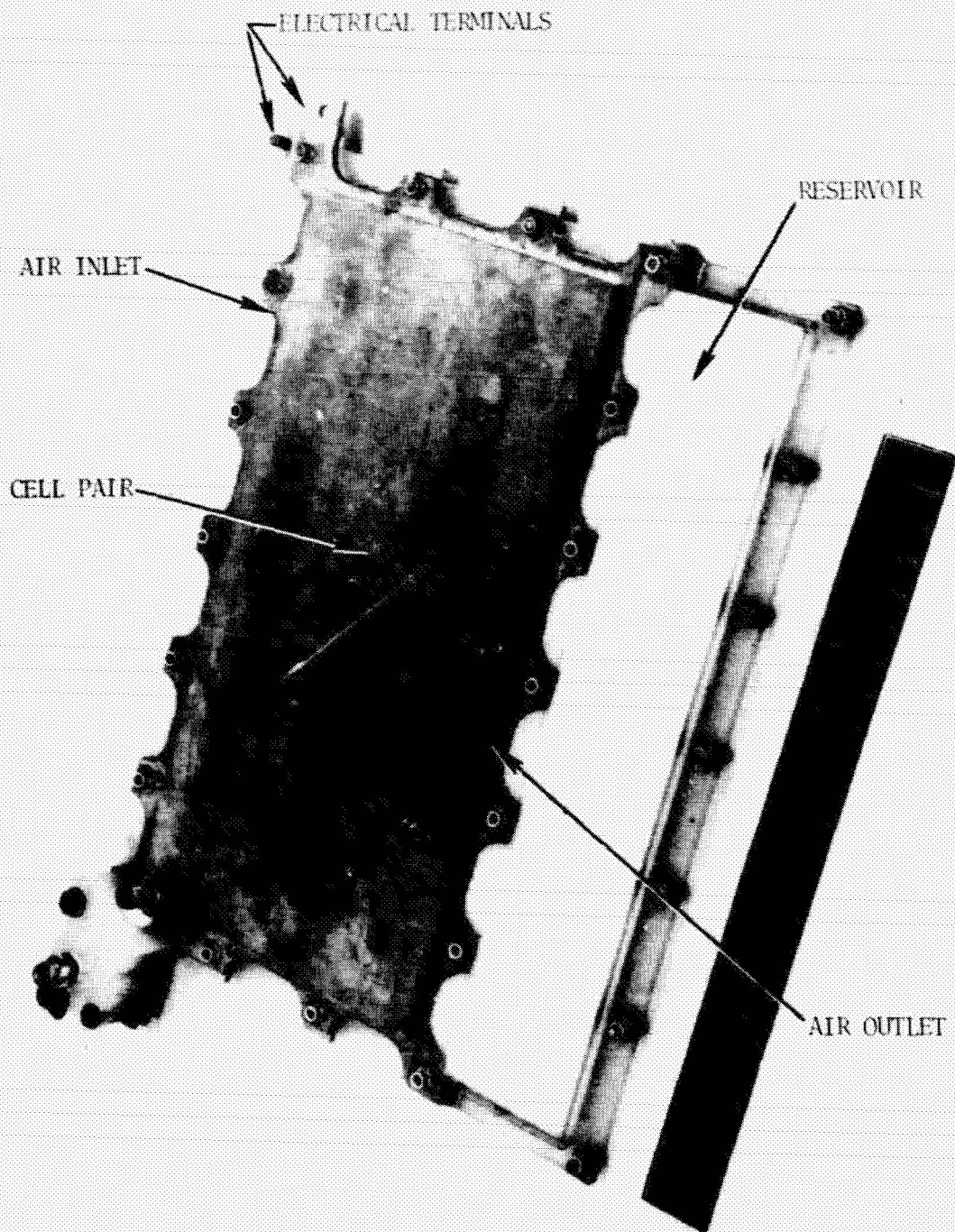
INSTRUCTION: _____   TEST ENGINEER: _____

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: WVE Reservoir Cell Pair

2. Purpose of Test: To determine the effect of vibration on the Reservoir Cell Pair Design (SVSK 81239 Concept).

3. Test Procedure: Testing will consist of one week of WVE baseline testing per Section I (WVE Conditioning Test), random vibration per Table I and a Post Baseline Test of one week.

Setup for Vibration Test: The cell pair will be mounted in a test fixture to simulate a rack mount. This fixture will provide additional protection to the Plexiglas Reservoir Housing.

The cell pair will be bagged to prevent any contamination of the cells and to protect the vibration equipment in case electrolyte (H_2SO_4) is vibrated loose from the matrix.

4. Test Runs: Baseline test 1 week; vibration in each of three mutually perpendicular planes; examination of cell after each plane of vibration; and one week of post baseline test.

5. Special Instructions: If the unit has been structually damaged during vibration testing Hamilton Standard and NASA/MSC will determine the remaining

WVE Vibration Test (WSS 5.1) (Continued)

program activity for this task.

6. Test Data: Performance and vibration test data will be presented graphically.

Random Vibration Levels - 2.5 minutes duration in each of the three (3) mutually perpendicular axes (x, y, z).

  20   to   80 H_z at +3 db/octave

  80   to   180 H_z at 0.06 g^2/H_z

 180   to   200 H_z at +12 db/octave

 200   to   400 H_z at 0.10 g^2/H_z

 400   to   450 H_z at -12 db/octave

 450   to 2000 H_z at 0.06 g^2/H_z

A minimum of six (6) accelerometers will be mounted, observed and recorded during this test, three (3) will be mounted on the fixture at the item/fixture interface as close to the mounting point as possible, sensing the vibration in each of the orthogonal directions of the item; and three (3) accelerometers located on the item to sense the vibration motion in the same direction as those accelerometers on the fixture.

Each of the accelerometers on the fixture will be used to control the vibration level during that portion of the test when it is primary sensor.

During the random testing all the accelerometers signals will be continuously recorded on magnetic tape. This tape will be retained for the remainder of the NAS 9-12920 program.

                                    -

RANDOM VIBRATION

TABLE I

SECTION III

HDC FLOW IMPROVEMENT

TEST PLAN

(WBS 6.1)

TEST NO. ___III___

HAMILTON STANDARD

PLAN OF TEST

JOB: _____HDC Hydrogen Flow Test (WBS 6.1)_____   PLAN PREPARED BY: _J. Huddleston_

PROJECT & ORDER: _____NAS 9-12920_____   APPROVED BY: _____

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: HDC Non-Reservoir Cell Pair

2. Purpose of Test: To determine the effect of various H_2 inflow rates on cell current efficiency.

3. Test Procedure: The HDC will b   sted per Section I, HDC Conditioning Tests, with the following excepti   s in the H_2 inflow rates: 500, 350, 200, and 150 cc/min.

4. Test Runs: Each H_2 flow rate will be maintained for approximately 24 hours with at least three readings taken during this time.

5. Special instructions: Prior to performing this series of tests the HDC Test Unit will be operated per conditioning requirements for one week.

6. Test Data: Test data will be presented as a curve of current efficiency vs the ratio of H_2 inflow/CO_2 transfer rate.

SECTION IV

MATRIX COMPRESSION

TEST PLAN

(WBS 3.3)

HS F-027 6/46

TEST NO. ___IV___

HAMILTON STANDARD

PAGE 1 OF _____

PLAN OF TEST

JOB: _____Matrix Compression (WBS 3.3)_____ PLAN PREPARED BY: __J. R. Aylward_____

PROJECT & ORDER: _NASA Contract - NAS 9-12920_____ APPROVED BY: _____

INSTRUCTION: _____ TEST ENGINEER: _____

TIME PERIOD: _____ TO _____

1. WHAT IS ITEM BEING TESTED?

2. WHY IS TEST BEING RUN? WHAT WILL RESULTS SHOW ON 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.

ℓ

NUMBER ENTRY AS LISTED ABOVE AND DESCRIBE BELOW

1. Test Item: Asbestos, Neoprene asbestos, Tissuquartz

2. Purpose of Test: To determine the matrix factor, tortuosity and optimum
   matrix compression for use in HDC computer program. |A

3. Test Procedure: Measure electrolyte resistance (R) with and without
   matrix as a function of electrode distance (d) using special micrometer
   cell (Figure 1) and suitable electrolyte. Determine |A
   uncompressed matrix thickness (d') and matrix void volume (m) with
   micrometer.

$$R = \frac{\rho}{A} d \times \frac{d}{d - d'(1-m)} \gamma = \frac{\rho}{A} d \times f \gamma$$

   (A = Area and ρ = Resistivity) |A

   Without matrix, tortuosity ($\gamma = 1$) and m = 1 so R = $\rho/A \times d$. Find ρ/A
   from plot of R vs d and correct for electronic resistance by extrapolation
   to d = o. Find γ from plot of R vs df ρ/A data with matrix (f = matrix
   factor). Determine if γ is function of d. If $\gamma \neq f(d)$ optimum compres-
   sion is at d = 2d' (1 = m).

4. Number of Runs: As required.

TEST NO. IV

PLEXIGLAS RETAINER & INSULATOR (2)

PT-PT ON FACE OF EACH ROD

ELECTRICAL LEADS

COPPER RODS, GOLD PLATED (IML) AND RHODIUM PLATED (1/10 MIL) (2)

1 7/8 - 2 7/8 OUTSIDE MICROMETER

MATRIX AREA

STOP-OFF LACQUER COATING (2 PLACES)

MATRIX CONDUCTIVITY FIXTURE

FIGURE 1

A-29

Matrix Compression (WVS 3.3) (Continued)

5. Special Instructions: None.

6. Test Results: The matrix factor, tortuosity and optimum compression
    for each matrix material will be tabulated.

Hamilton Standard
DIVISION OF UNITED AIRCRAFT CORPORATION

SECTION V

TMAC ELECTROLYTE ANALYTICAL TEST CELL

TEST PLAN

(WBS 3.4)

TEST NO. _____V_____

HAMILTON STANDARD

PLAN OF TEST

JOB: _____TMAC Analytical Cell Tests (WBS 3.4)_____   PLAN PREPARED BY: _J. R. Aylward_

PROJECT & ORDER: NASA Contract - NAS 9-12920_____   APPROVED BY: _____

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: Analytical HDC test cell using TMAC Electrolyte (Figure 1)

2. Purpose of Test: To obtain a more detailed knowledge of HDC cell
operation and evaluate new concepts to improve its performance.

3. Test Procedure: The analytical test cell will be operated in an environ-
ment provided by the test rig as illustrated in Figure 2. The electrical
operation of the cell will be controlled and monitored by a potentiostat
console pictured in Figure 3.

Each test condition will be run for approximately two days with at least
three data readings taken each day. The data and calculations which will
be made during each test condition are listed in Table I.

4. Itemized Runs: See Table II

5. Special Instructions: None

6. Test Results: Test data will be summarized in tabular form.

TITANIUM HSG.

THRU HOLES IN TITANIUM HSG (COVERED BY PLEXIGLAS COVER)

SPECIAL REFERENCE ELECTRODES

INLET AIR

(4 PASSAGE WAYS)

OUTLET AIR

6" AIR PASSAGE
(SAME AS FULL SIZE CELL AIR)

RESERVOIR

PLEXIGLAS COVER

MATRIX

ANODE

CATHODE

INLET AIR

OUTLET AIR

RESERVOIR

H₂ INLET

SPECIAL REFERENCE ELECTRODES

HDC ANALYTICAL TEST CELL

FIGURE 1

A-33

HDC ANALYTICAL TEST UNIT RIG
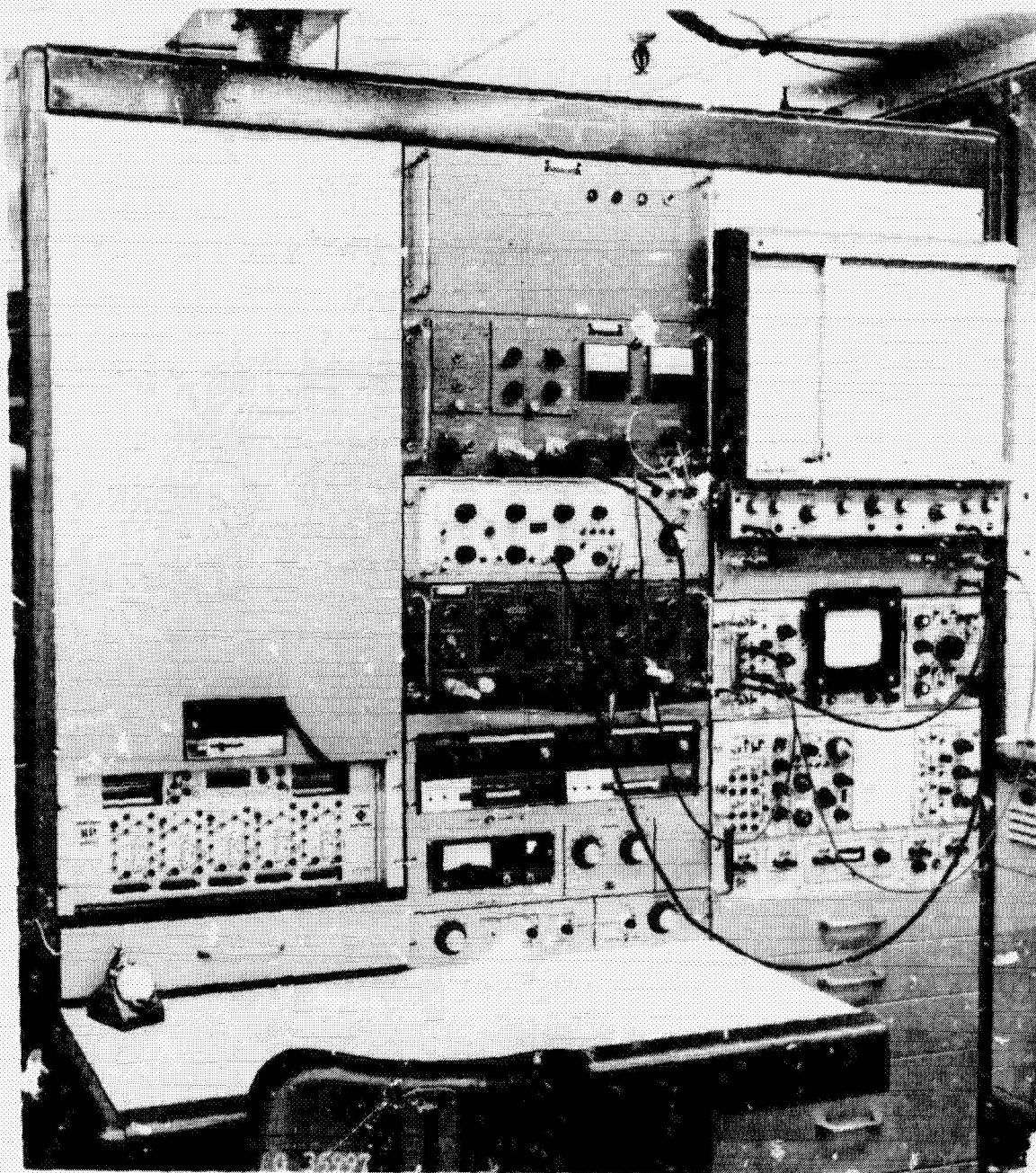
FIGURE 2

OPERATING CONSOLE FOR HDC ANALYTICAL TEST CELL

FIGURE 3

A-35

INSTRUMENTATION

Read-Outs	Calculations
Inlet Air Temperature	Inlet Air P_{CO_2}
Inlet Air Dewpoint	CO_2 Removed
Inlet CO_2 Lira	Current Efficiency
Outlet Air Temperature	Power
Air Flow ΔP Across Cell	Air Flow Vel.
H_2 Inflow meter	Current Density
H_2 Inlet Press.	H_2 inflow
H_2 Inlet Temperature	ΔP Across Matrix

$H_2 + CO_2$ Outlet Flow

CO_2 in H_2 Outlet (Lira)

Cell Voltage

Cell Current

O_2 Concentration

Special Readings

pH at Anode

pH at Cathode

Activation Overvoltage at Anode

Activation Overvoltage at Cathode

Concentration Overvoltage

IR Drop

TABLE I

TABLE II

TEST (CONDITIONS)	DEW POINT (°F) 40	50	60	PCO₂ (mmHg) 0.25		4	AIR FLOW RATE (FT/SEC) 5	10	20	MATRIX THICKNESS .020	.040	CURRENT DENSITY (asf) 6	12	24
Matrix Thickness & Base Line		✳			✳			✳			✳		✳	
Current		X			X			X		X		X	X	X
CO₂ Mass Transport in Air		X	X	X			X	X	X				X	
CO₂ Mass Transport in Air		X	X	X				X	X				X	
P_CO₂ & DP	X	X	X	X		X		X					X	
P_CO₂ & DP	X	X	X	X		X		X					X	
Inert Electrolyte		X	X		✳			✳					✳	
Catalyst for CO₂		X	X		X			X						X
NH₃		X	X		X			X					X	
Trace Impurities CO		X	X		X			X					X	
H₂S		X	X		X			X					X	
H₂ Flow and Backpressure		X			✳			X					✳	

a. H₂ flow rates and backpressure will be determined from tests of Series H-1 for cell design correlation.

SECTION VI

TRACE IMPURITIES

TEST PLAN

(NBS 3.5)

HAMILTON STANDARD

PLAN OF TEST

JOB: _____Trace Impurities (WBS 3.5)_____   PLAN PREPARED BY: J. R. Aylward _____

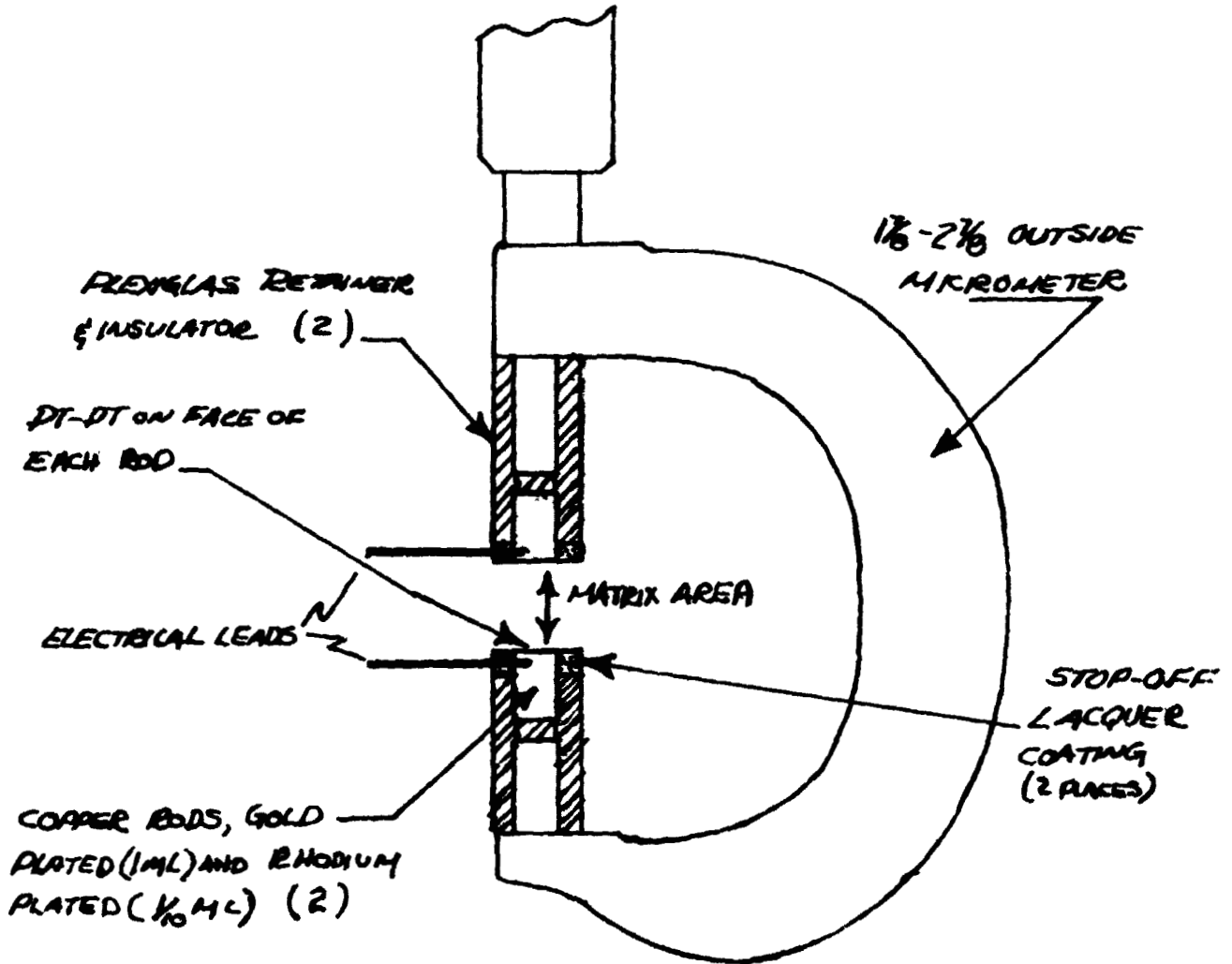PROJECT & ORDER: ___NASA Contract NAS 9-12920___   APPROVED BY: _____

INSTRUCTION: _____   TEST ENGINEER: _____

TIME PERIOD: _____ TO _____

1. WHAT IS ITEM BEING TESTED?

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

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

4. ITEMIZE MM'S TO BE MADE GIVING LENGTH OF EACH AND READINGS TO BE TAKEN.

5. SPECIAL INSTRUCTIONS: SAFETY PRECAUTIONS OR 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: Trace impurities CO, H_2S, NH_3, Freon.

2. Purpose of Test: Determine if the above trace impurities and their oxidation or reduction products have any adverse affects on the HDC cell and investigate possible methods of recovering from any such affects.

3. Test Procedure: Particular procedure to be worked out for each impurity. In general potentiostatic sweeps will be made to determine adsorption characteristics, electrochemical reactivity and main reaction products, as a function of electrode potential and pH. Floating electrode tests will be used to determine poisoning effects, and if necessary small cell tests will be conducted when the above results are not conclusive. The cathode studies will use CS_2CO_3 electrolyte (pH = 12) and the anode $CsHCO_3$ (pH = 8).

4. Number of Runs: As required.

5. Special Instructions: None.

6. Test Results: The test results will be presented in table form, showing the amount of decrease in H_2 and O_2 absorbtion.

SECTION VII

Cs_2CO_3 ELECTROLYTE ANALYTICAL TEST CELL

TEST PLAN

(WBS 3.6)

JOB: __CS$_2$CO$_3$ Analytical Cell Tests (WBS 3.6)__   PLAN PREPARED BY: __J. R. Aylward__

PROJECT & ORDER: __NASA Contract NAS 9-12920__   APPROVED BY: _____
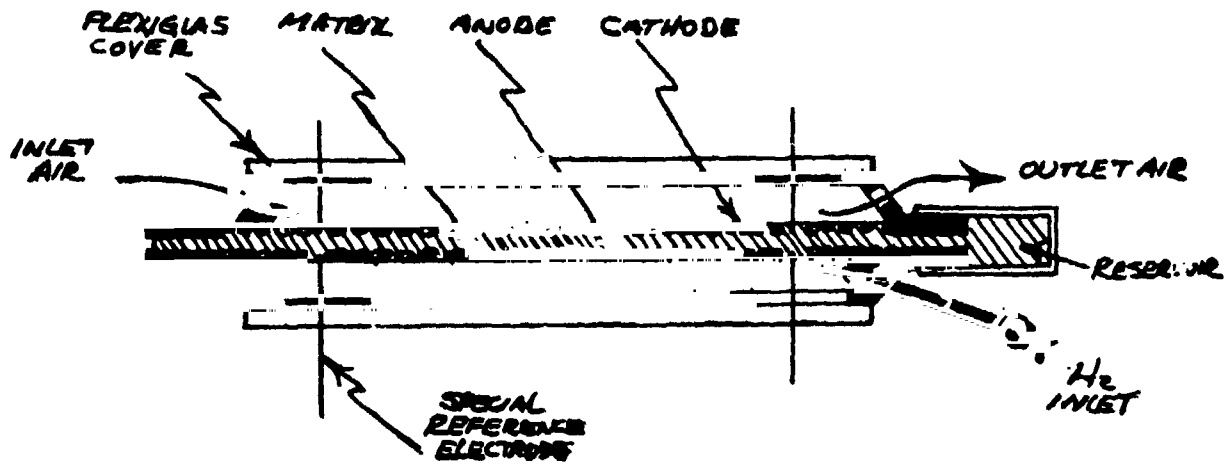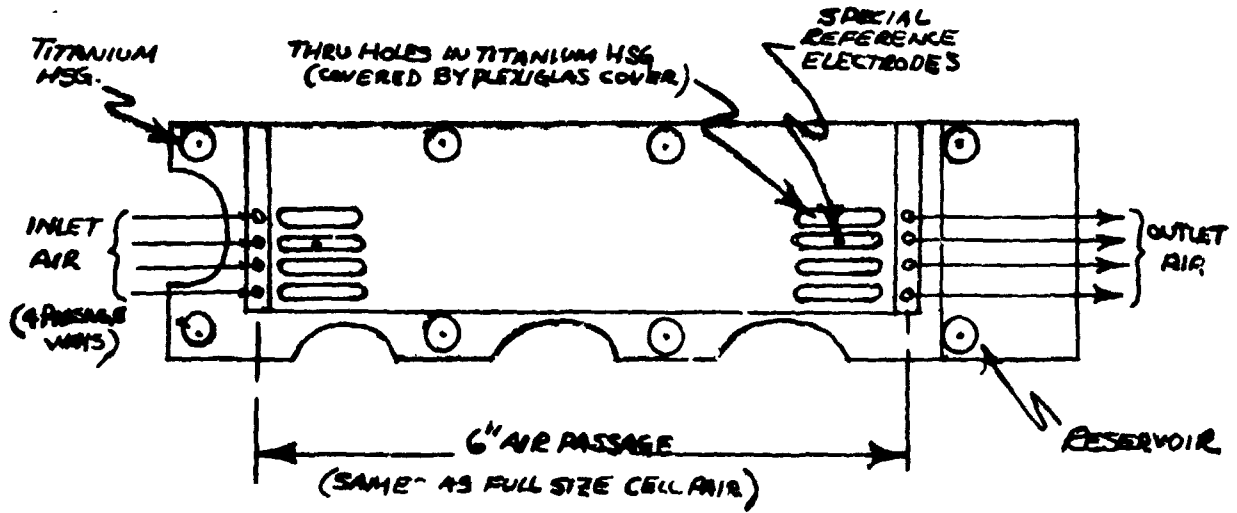
INSTRUCTION: _____   TEST ENGINEER: _____

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
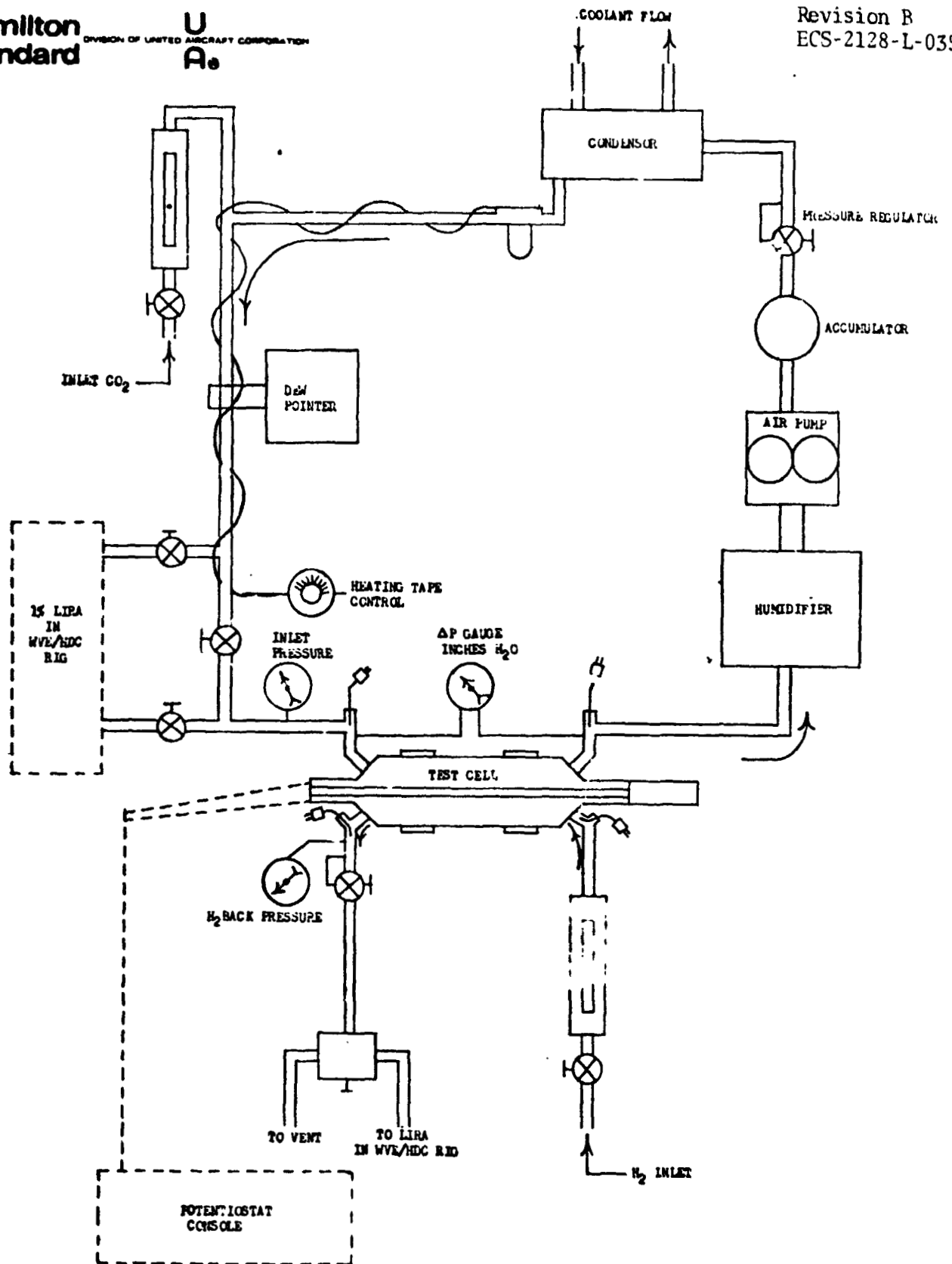   AS IT WILL BE FINALLY PRESENTED.

NUMBER ENTRY AS LISTED ABOVE AND DESCRIBE BELOW

1. Test Item: Analytical HDC Test Cell using Cs_2CO_3 Electrolyte
   Reference figure 1).

2. Purpose of Test: To determine effective CO_2 gas-phase mass transport
   coefficient and correlate with theoretical equation. Also to determine
   anolyte and catholyte pH variations over a range of operating conditions.

3. Test Procedure: The analytical test cell will be operated in an environ-
   ment provided by the test rig as illustrated in figure 2. The electrical
   operation of the cell will be controlled and monitored by a potentiostat
   console pictured in figure 3.
   Each test condition will be run for approximately two days with at least
   three data readings taken each day. The data and calculations which will
   be made during each test condition are listed in Table I.

4. Itemized Runs: Mass transport; P_{CO_2} (0.25, 0.5, 1.0 mmHg) air velocity
   (5, 10, 20 ft/sec). Electrolyte pH; current density (12 ASF, 24 ASF).
   P_{CO_2} (0.25, 1, 4 mmHg), air dew point (6.).   One test at nominal |B
   conditions, except for P_{CO_2} 3 mmHg, current density 5 ASF and air flow |A
   of 7 ft/sec.

5. Special Instructions: None.   A-41

CS_2CO_3 Analytical Cell Tests (WBS 3.6) (Continued)

6. Test Results: Test data will be presented in tabular form as follows:

   a. Mass transport coefficient for the various combinations of air velocity and CO_2 partial pressures.

   b. pH values of the cesium carbonate at the cathode and anode for various combinations of current density, CO_2 partial pressures and inlet air dew points.

HDC ANALYTICAL TEST CELL

FIGURE 1

HDC ANALYTICAL TEST UNIT RIG

FIGURE 2

OPERATING CONSOLE FOR HDC ANALYTICAL TEST CELL

FIGURE 5

HAMILTON STANDARD

PLAN OF TEST

JOB: __HDC MATRIX COMPRESSION SET (WBS 9A.2)__   PLAN PREPARED BY: __J. C. Huddles__

PROJECT & ORDER: __NASA Contract NAS 9-12920__   APPROVED BY: _____

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. Item Being Tested: HDC asbestos matrix and asbestos matrix/electrode combination.

2. Purpose of Test: To determine force versus deflection data - will determine thickness of insulating shims for HDC cell assembly.

3. Test Procedure: See attached sheet - tests will be conducted on the "Instron" compression test machine in the ASD Mechanical Laboratory (see figure 1). Fabricated hardware needed:

   A. Fixture for dynamic tests - cavity and plunger

      Area - 1 sq. inch. - see figure 2 - one req'd.

   B. Fixture for pre-set testing - cavity and plunger

      Area - 1 sq. inch, see figure 2, four req'd. - make 3 - use fixture from dynamic tests.

   C. Punch for test samples - one req'd.

      Dynamic tests - test samples

         Matrix (Asbestos) - 22 req'd, 10 spares

         Electrode - 2 req'd, 4 spares

      Pre-set tests - test samples

         Matrix (Asbestos) - 10 req'd, 10 spares

         Electrode - 2 req'd, 4 spares

ON RESET ZERO TO — COMPENSATE FOR TARE WEIGHT OF SAMPLE AND FIXTURE UP TO 5 LBS.

PEN

INSTRON

FEED SCREWS
FEED RATE
.02 OR .05
IN. PER MIN.

50 LBS.   0 LBS.

FULL SCALE LOAD 50
0 200

RECORDER

FIXTURE & SAMPLE

DIAL INDICATOR

FEED
ON
OFF

COMPRESSION
SENSOR HEAD

TEST SETUP

FIGURE 1

A-47

Hamilton Standard DIVISION OF UNITED AIRCRAFT CORPORATION U A®

1.128 DIA - MATRIX &
ELECTRODE TEST SAMPLE

CLAMPS USED
FOR PRE-SET
TESTING-TWO

MACHINE
HEAD

AREA
1 SQ. INCH

PLUNGER

MATRIX AND
ELECTRODE SAMPLES

MACHINE COMPRESSION
SENSOR SURFACE

TEST FIXTURE
FIGURE 2
A-48

4. Test Runs:

   Dynamic Tests

   A. Buildup - 2 asbestos and 2 electrode test samples wet with water.
      Matrix compression - from .040 to solid height (steep slope on
                              recorder) - feed .02 or .05 inches/min.
      Instrumentation - recorder, force versus deflection indicator -
                              distance traveled.
      No. of tests - 2; change samples for each test.

   B. Buildup - 3 asbestos and 2 electrode test samples wet with water.
      Matrix compression - .060 to solid/height (steep slope on recorder) -
                              feed same as 4A.
      Instrumentation - same as 4A.
      No. of Tests - 2; change samples for each test.

   C. Buildup - 2 asbestos and 2 electrode test samples wet with water.
      Matrix compression - first run .040 inches to solid height (ste)
                              slope on recorder) - feed same as 4A.
                              second to fifth run .040 inches to solid/height
                              (steep slope on recorder) - same feed as 4A.
      Instrumentation - same as 4A.
      No. of tests - 5; same test samples.

   D. Buildup - 2 asbestos and 2 electrode test samples wet with water.
      Matrix compression - .040 inches to .022 inches (matrix thickness) -
                              feed same as 4A.
                              Hold at .022 inches for ≈ 2 hours.
                              Repeat run .022 to solid height (steep slope
                              on recorder).
      Instrumentation - same as 4A except record reading on recorder
                              every 15 minutes during the two hour hold period.
      No. of tests - 1.

5. Pre-Set Tests

   A. Buildup - 2 asbestos and 2 electrode test samples wet with Cs_2CO_3.
      Matrix compression - .040 inches to .022 inches (matrix thickness) -
                              feed same as 4A.
                              Hold (clamp) at .022 inches for one week (seven
                              days)in chamber at 45°F dew point and 52°F dry
                              bulb - mount so weight of plunger is not acting
                              on compressed matrix.
                              After seven days hold time, make run from .022
                              inches to solid height (steep slope on recorder).
      Instrumentation   same as 4A.
      No. of test - 1.

   B. Same as 5A except hold time is fourteen days.

   C. Same as 5A except hold time is forty-two days.

D. Same as 5A except hold time is one hundred five days.

E. Same as 5A except hold time is one hundred eighty days.

6. Instructions

   Note - Instron machine will be run by a materials lab. technician.

   A. Mount compression sensor head on machine bed.

   B. Place samples in test fixture and carefully lower plunger in place (see figure 2).

   C. Place test fixture on compression sensor head flat surface, centering with machine spindle (see figure 1).

   D. Lower center spindle until it just touches spindle, use shims.

   E. Mount indicator so it touches spindle bottom surface, set indicator to zero.

   F. Zero recorder, compensate for weight of fixture, samples and plunger.

   G. Set full scale load knob to 50 lbs. - adjust lower or higher after observing initial test results.

   H. Set machine feed (.02 or .05 inches/min.) and recorder paper speed.

   I. Start machine and recorder - watch movement of recorder pen. When solid height is approached, slope of recorder curve becomes very steep. Shut off machine.

   J. Take indicator reading to determine the number of thousandths travel.

   K. Reverse feed to raise machine.

   L. Repeat A through K for each test run.

7. Data will be plotted and presented as shown in figure 3.

   For dynamic test 4D and pre-set tests show family of curves to show any changes with time.

SAMPLE DATA
FIGURE 3

HAMILTON STANDARD
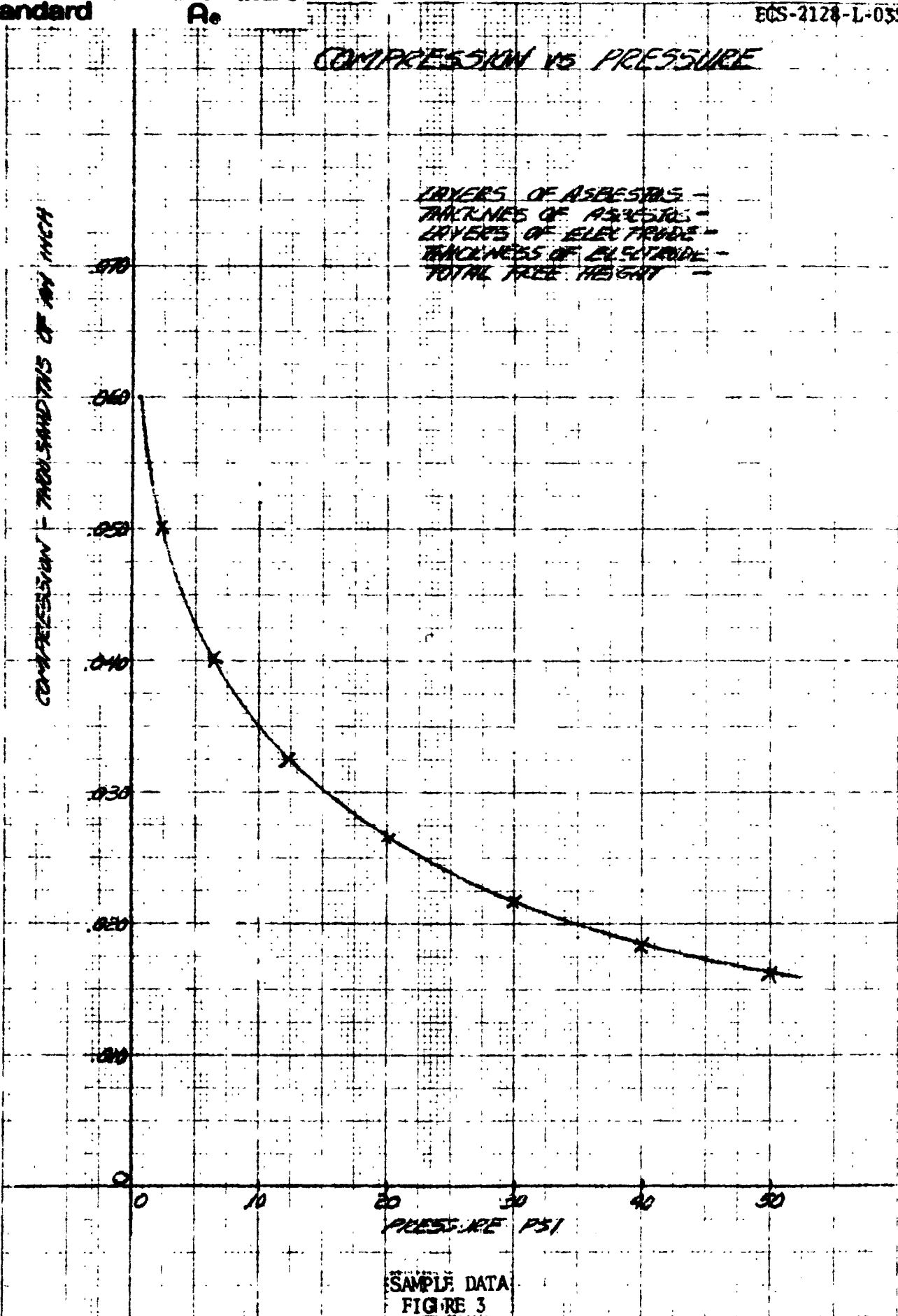
<u>PLAN OF TEST</u>

JOB: HDC Analytical Cell Tests (WBS 9A.)   PLAN PREPARED BY: J. Woodlezton

PROJECT & ORDER: NAS 9-12920   APPROVED BY: _____

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.

<u>NUMBER ENTRY AS LISTED ABOVE AND DESCRIBE BELOW</u>

1.	Item Being Tested: HDC Analytical Cell, Reference figure 1, of various configurations as noted in Table I.
2.	Purpose of Test: To evaluate various cell modifications which are proposed (electrodes, matrix thickness, CO_2 catalyst), that will reduce the power decay and improve the CO_2 removal rate. This evaluation will be done on the analytical test cell instead of a full size cell pair.
3.	Test Procedure: Testing of the analytical cell per the configurations and parameters of Table I will be performed on the Analytical Test Rig as as presented in Sections V and VII of the Master Test Plan.
4.	Test Runs: There will be four basic tests as outlined in Table I.
	#1 Test - one test for a week duration.
	#2 Test - four tests (minimum of 4 electrodes to be screened) of approximately 2 days each.
	#3 Test - two tests (2 best electrodes) of approximately one week each.
	#4 Test - two tests (minimum and maximum matrix thickness) of approximately one week each.
5.	Special Instructions: None.
6.	Presentation of Data: The performance of the cell will be compared to tha of previous Analytical Cell tests (Section VII) and to that of a full size cell pair.

PARAMETER	#1 - CO₂ Catalyst Test	#2 - Electrode Screening (4) Tests	#3 - New Electrode (2) Tests	#4 - Matrix Thickness (2) Test
Cell Configuration Deviation from Std Cell*	Additive to Electrolyte of Carbonic Anhydrase	No. Reference Electrodes New Electrode Configurations	New Electrode Configuration	New Electrode Configuration One Layer of Matrix Four Layers of Matrix No Reference Electrodes
SET CONDITIONS:				
Inlet Air Temp. (°F)	70	70	70	70
Inlet Air Dew Point (°F)	63	63	63	63
Inlet Air PCO₂ (mmHg)	2.5	2.5	2.5	2.5
Inlet Air O₂ Concen. (%)	21 - 22	21 - 22	21 - 22	21 - 22
Air Flow (ft/sec)	7	7	7	7
ΔP Across Matrix (psi)	None	None	None	None
H₂ Flow (cc/min)	650	650	650	650
Current Density (asf)	12 & TBD	TBD	12 & TBD	TBD
Monitored Parameters:				
All Set Conditions	X	X	X	X
H₂ + CO₂ Outlet Flow	X	X	X	X
% CO₂ in H₂ + CO₂ Flow	X	X	X	X
H₂ Temp.	X	X	X	X
Outlet Air Temp.	X	X	X	X
Cell Voltage	X	X	X	X
Reference Voltages (5)	X	·	X	·
Anode & Cathode pH	X	X	X	X
IR of Cell	X	·	X	X
Calculated Data:				
Power	X	X	X	X
CO₂ Removal Rate	X	X	X	X
CO₂ Removal Efficiency	X	X	X	X

* Standard Cell Configuration - 1/24 fullsize cell pair, Cs₂CO₃ Electrolyte, P & WA Asbestos Matrix - 2 sheets of .020 compressed to .022, 4 reference electrodes, reservoir and modified housing to allow checking of pH at anode and cathode.

TEST CONDITIONS

TABLE I

A-53

HAMILTON STANDARD

PLAN OF TEST

JOB: HDC cell Pair Testing (WBS 9B.)   PLAN PREPARED BY: J. Woolever

PROJECT & ORDER: NAS 9-12920   APPROVED BY: _____

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.	Item Being Tested: HDC cell pair, with reservoir incorporated on the inlet air side of the cell pair.
2.	Purpose of Tests: (a) Testing of cell pairs #017 and #019 will be done to investigate the cause of power decay and to improve CO_2 removal rates (b) Testing of two new cell pair assemblies, one using TMAC electrolyte and one using cesium carbonate, will be done to verify that the cell modifications have decreased the power decay and improved the CO_2 remova
3.	Test Procedure: Testing will be conducted in the HDC test facility usin the separate cell pair test fixture. Test conditions and monitored parameters are outlined in Table I with the following exceptions: (a) Cell #017 will be operated at 15 asf (if possible). - Power decay may cause the current density to fall below this value. (b) Cell #019 will be operated at 18 asf only.
4.	Test Runs: (a) Cell #017 will be operated until late January 1973. (b) Cell #019 will be operated until late January 1973 at which time the cathode will be changed and testing continued. Duration TBD. (c) The verification testing will be targeted for 2000 hours.
5.	Special Instructions: Lira Calibration - the lira which monitors the CO_2 in the test chamber w have its end points checked once/week.

- the lira which monitors the CO_2 in the outlet H_2+CO_2 gas flow will
have its end points checked twice/week.

Changes in Operation - any change in the operating parameters of the cell
pairs will be cleared through NASA/MSC prior to change. In emergencies
where changes must be made to protect the test item, corrective action
will be made without prior NASA/MSC review. However, a full review of
the incident will be made within twenty-four hours (except on weekends).

6. Presentation of Data: The performance of the cell pair will be graphically
presented versus time, i.e., power, CO_2 removal rate, current efficiency
and voltage.

TABLE I

TEST CONDITIONS

SET TEST CONDITIONS		Cs₂CO₃ CELL PAIR					TMAC CELL PAIR						
		CONDITIONING	PARAMETRIC TEST INITIAL & END			ENDURANCE	CONDITIONING	PARAMETRIC TEST INITIAL & END			ENDURANCE	ARAMETRIC TEST INITIAL & END	
INLET AIR TEMPERATURE	(°F)	52				➤	52				➤	Room (74°F)	
INLET AIR DEW POINT	(°F)	49	45			➤	49	45			➤	Room (60°F)	
INLET AIR P_{CO_2}	(mm Hg)	3.0				➤	3.0				➤	1/4	3.0
INLET AIR O_2 CONCENTRATION	(%)	20-21				➤	20-21				➤		
AIR FLOW THROUGH CELL PAIR	(SCFM)	10				➤	1r						
ΔP ACROSS CELL MATRIX (min)	(PSI)	1				➤	1						
H_2 INFLOW TO CELL PAIR	(cc/min)	900				➤	900				➤	②	②
CURRENT DENSITY	(asf)	18	18	14	22	18	18	18	14	22	18	18	10
MIN. DURATION ON CONDITION	(hours)	120	24	24	24	①	120	24	24	24	①	48	48

① TOTAL TIME OF ALL TESTING OF 2000 HRS AS A GOAL

② ADJUST H_2 FLOW TO OBTAIN GOOD H_2/CO_2 LIRA READING

● MONITORED PARAMETERS

    All Set Conditions
    H_2 + CO_2 Outlet Flow
    % CO_2 in H_2 + CO_2 Outlet Flow
    Cell Voltage
    H_2 Temp.

● CALCULATED DATA

    Power
    CO_2 Removal Rate
    Current Efficiency

● TEST DATA WILL BE TAKEN ONCE/DAY WITH THE EXCEPTION OF WEEKENDS