NASA-CR-192982

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Submitted to:

National Aeronautics and Space Administration
Marshall Space Flight Center
MSFC, AL 35812

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NASA Center for Aerospace Information (2 copies)

TR-1619-1-2

TECHNICAL PROGRESS REPORT

Integrated Oxygen Recovery System

Technical Progress Report No. 2

Prepared Under

Program No. 1650

for

Contract NAS8-39843

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May 7, 1993

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ACRONYMS

CReA	Carbon Dioxide Reduction Assembly
IORS	Integrated Oxygen Recovery System
OGA	Oxygen Generation Assembly
PEEK	Polyetheretherkeytone
SBIR	Small Business Innovation Research
SMC	Solid Metal Cathode

1.0 WORK PERFORMED DURING REPORTING PERIOD

This Technical Progress Report summarizes the work performed under Contract No. NAS8-39843 from 03/09/93 through 05/07/93. This contract is a Phase I Small Business Innovation Research (SBIR) program to demonstrate the feasibility of the Integrated Oxygen Recovery System (IORS). The IORS is applicable to advanced mission air revitalization. It provides the capability for electrochemically generating metabolic oxygen (O_2) and recovering O_2 from the space habitat atmosphere via a carbon dioxide (CO_2) reduction process within a single assembly. To achieve this capability, the IORS utilizes a novel Solid Metal Cathode (SMC) water electrolysis unit that simultaneously serves as the Sabatier CO_2 reduction reactor.

The IORS would enable two major life support systems currently baselined in closed loop air revitalization systems to be combined into one smaller, less complex system. It would reduce fluidic and electrical interface requirements and eliminate a hydrogen (H_2) interface. Furthermore, since the IORS utilizes an SMC, the system has the additional capability to generate high pressure O_2 (i.e., $\approx$1,000 psia) for recharging extravehicular activity O_2 bottles. This capability is not part of currently baselined or planned technologies.

During this Phase I SBIR program we will evaluate the IORS process by demonstrating its performance and quantifying key system physical characteristics, including power, weight and volume.

Work performed during this reporting period included completion of the assembly of the Breadboard IORS, checkout and shakedown testing of it and its test setup, and the initial parametric testing.

1.1 Assembly of the Breadboard IORS

Upon receipt of the polyetheretherkeytone (PEEK) to be used to construct the cap of the Breadboard IORS (Figure 1-1), the cap was fabricated. The Breadboard IORS was then assembled as described in the previous Bimonthly Technical Progress Report (TR-1619-1-1). It was integrated in the test setup (Figure 1-2, with legend in Figure 1-3).

A reference electrode, not shown in Figure 1-1, was fabricated and mounted in the Breadboard IORS. The electrode is a palladium/silver (Pd/Ag) tube, sealed at one end, and it has a geometric surface area of 10.1 cm^2. It is inserted in the electrolyte adjacent to the anode. Prior to operation of the Breadboard IORS, the reference electrode is charged with hydrogen by applying a cathodic current of 0.87 A for 12 minutes. Thereafter, no current is applied to the reference electrode, and it provides a virtually stable reference potential that can be used to measure changes in the potential of the anode or cathode.

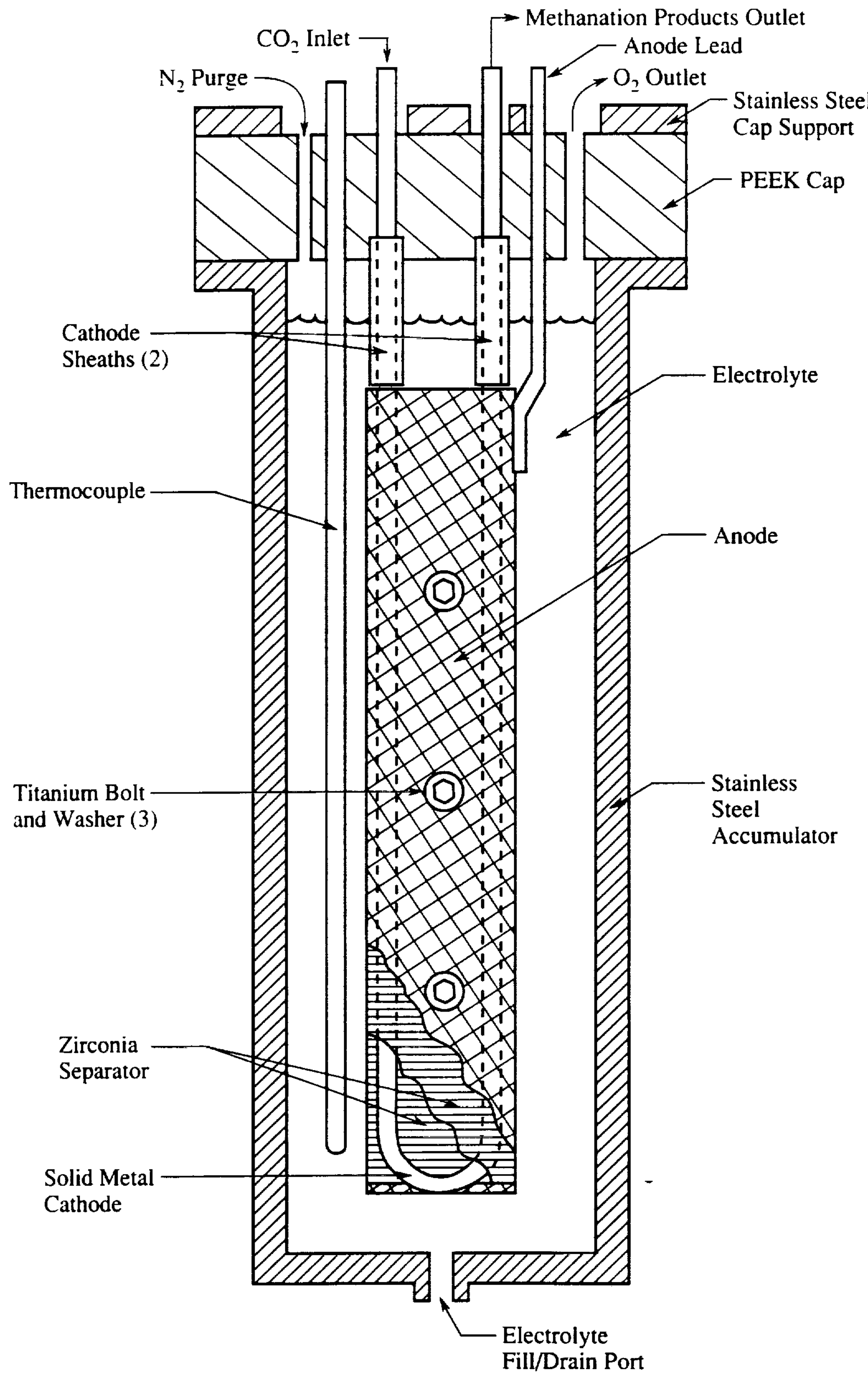

FIGURE 1-1 BREADBOARD IORS CELL

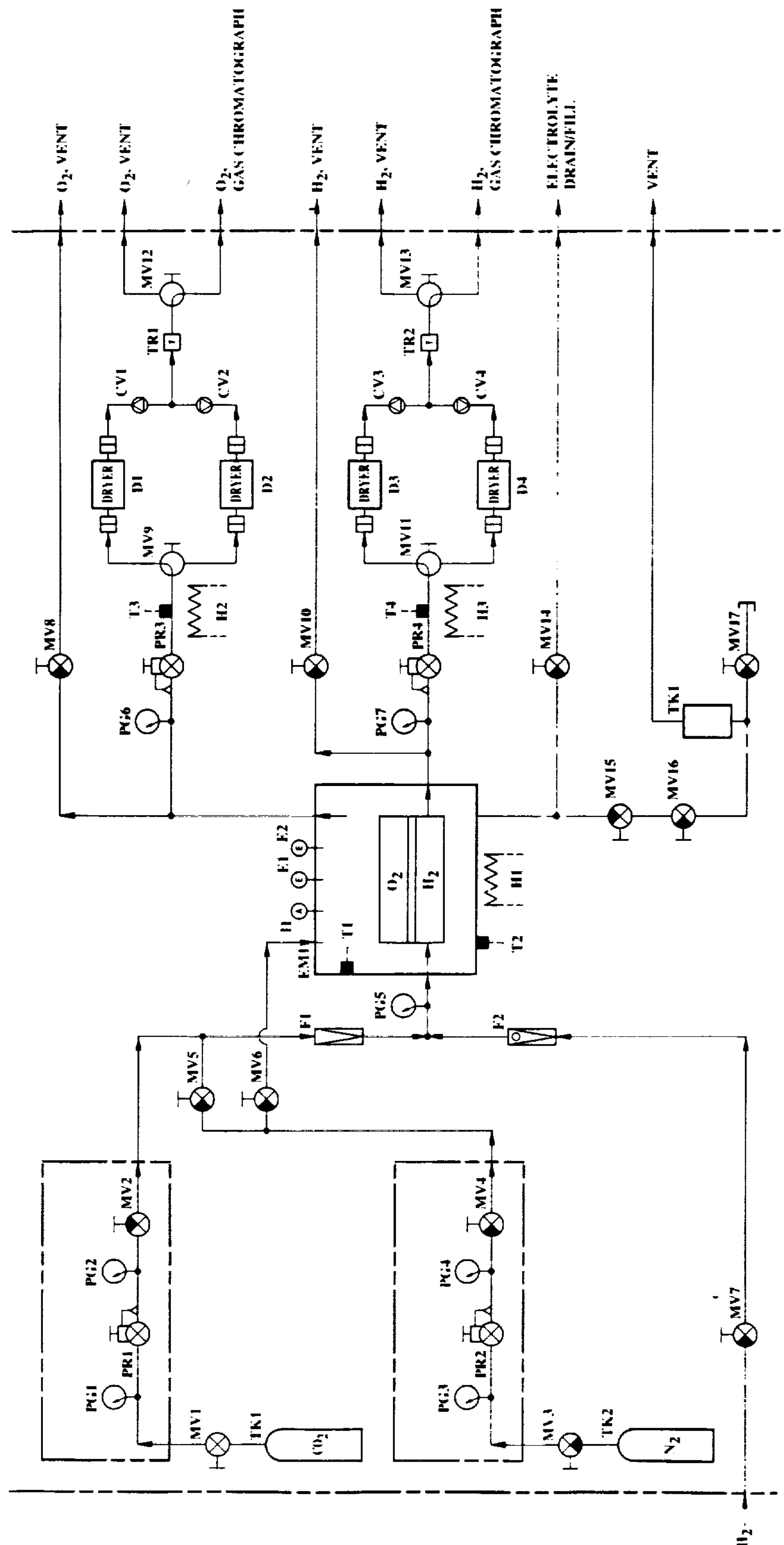

FIGURE 1-2 BREADBOARD IORS TEST SETUP MECHANICAL SCHEMATIC

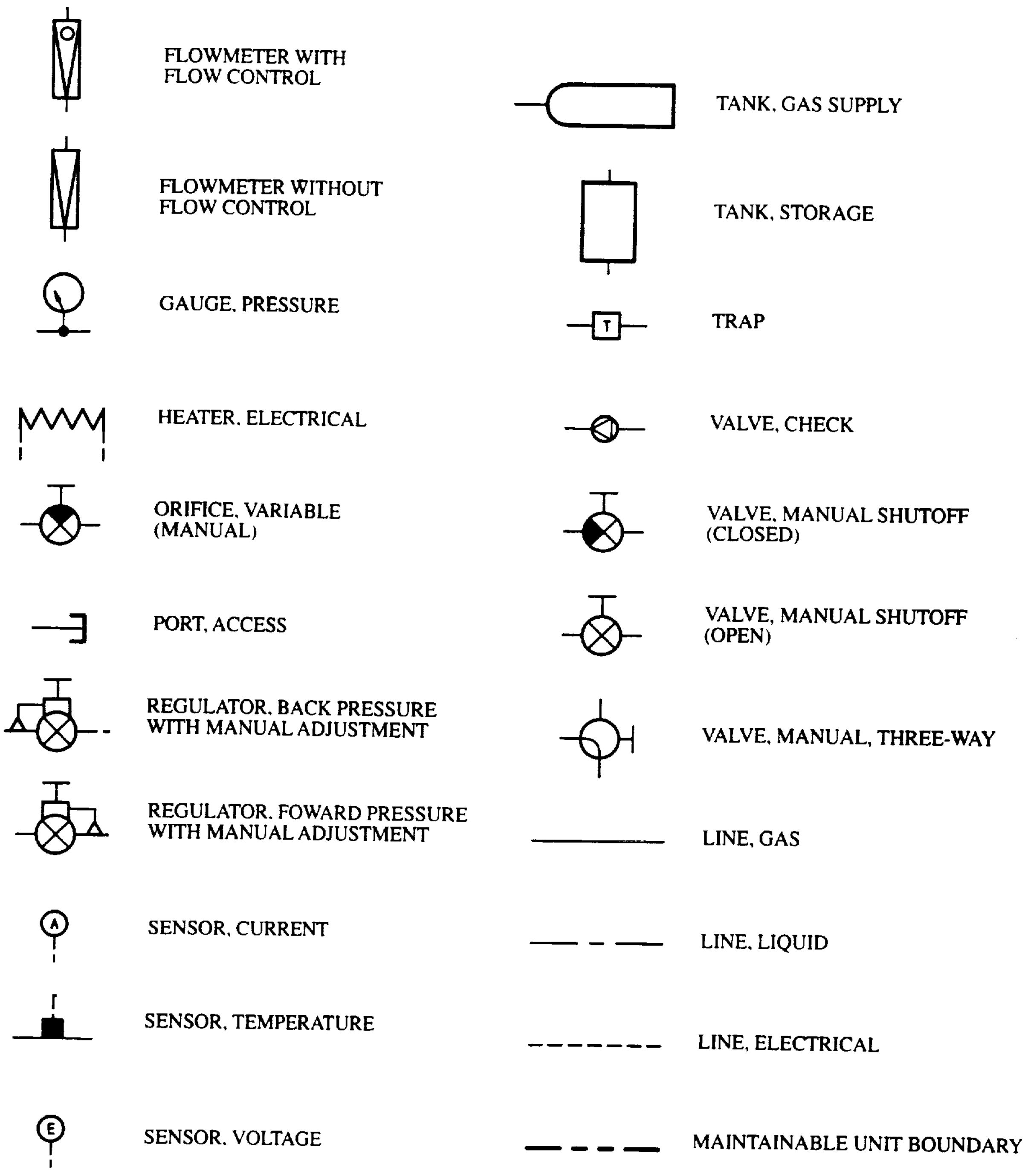

FIGURE 1-3 BREADBOARD IORS TEST SETUP MECHANICAL SCHEMATIC (LEGEND)

The gas chromatograph to be used for analysis of the product gases was also setup and calibrated during this reporting period.

1.2 Checkout and Shakedown Testing

These tests were performed to verify the integrity of the Breadboard IORS and its test setup. The tests were performed by operating the Breadboard IORS as an electrolyzer, without the methanation catalyst in the cathode.

During these tests it was found that the stainless steel accumulator was oxidized at 140 C, where it contacted the electrolyte (55% potassium hydroxide (KOH)). To avoid oxidation products from contaminating the electrodes, a Teflon cup was fabricated and inserted in the accumulator to hold the electrolyte.

During these tests it was also determined that the titanium nuts, bolts and washers (Figure 1-1) used to hold the anode against the separator and cathode also oxidized. These items were removed and replaced with nickel wire retainers that performed the same function.

After these changes, a new anode, cathode and separator were fabricated and mounted in the Breadboard IORS. No further oxidation products were detected in the electrolyte or on the electrodes. However, the zirconia cloth used as the separator tended to be torn where the anode and cathode were compressed against it. This occurred after six to ten hours of testing, and is attributed to the relatively low mechanical strength of the zirconia cloth when it is wet, compounded by the fact that the cathode expands and contracts as it absorbs and desorbs hydrogen.

The zirconia cloth was replaced with zirconia felt (0.127 cm thick). Further, the felt separator is replaced each time the Breadboard IORS is disassembled for electrolyte replenishment.

The electrochemical performance of the Breadboard IORS at 140 C is shown in Figure 1-4. The terminal voltage and the cathode-to-reference electrode potential is shown as a function of current density over the range of 45 to 200 mA/cm^2. These data show that the cell potential is relatively low, and that there is little increase in the potential of the cathode, even at the relatively high current density of 200 mA/cm^2.

Figure 1-5 shows the performance of the SMC in transferring H_2 from its exterior to the interior of the SMC. The Breadboard IORS can produce H_2 at a flow rate of more than 51 scc/min, with a transfer efficiency of 96% or greater, and a power consumption of only 13.5 W at 140 C.

Methanation of CO_2 in the SMC does not occur in the absence of catalyst. Prior to installation of catalyst in the Breadboard IORS, it was operated at a current density of 200 mA/cm^2 at a temperature of 143 C, with CO_2 flowing through the SMC at

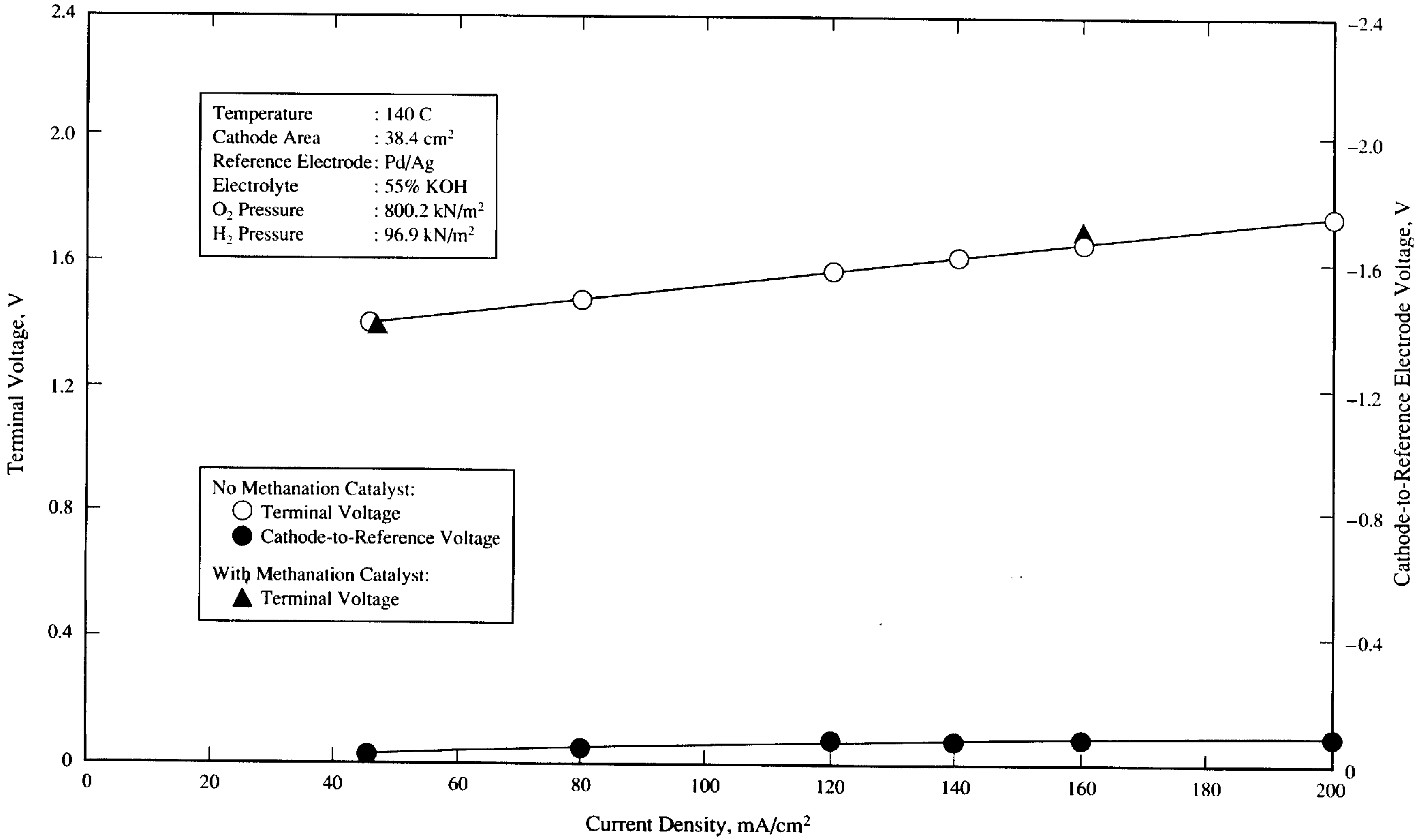

FIGURE 1-4 VOLTAGE AS A FUNCTION OF CURRENT DENSITY

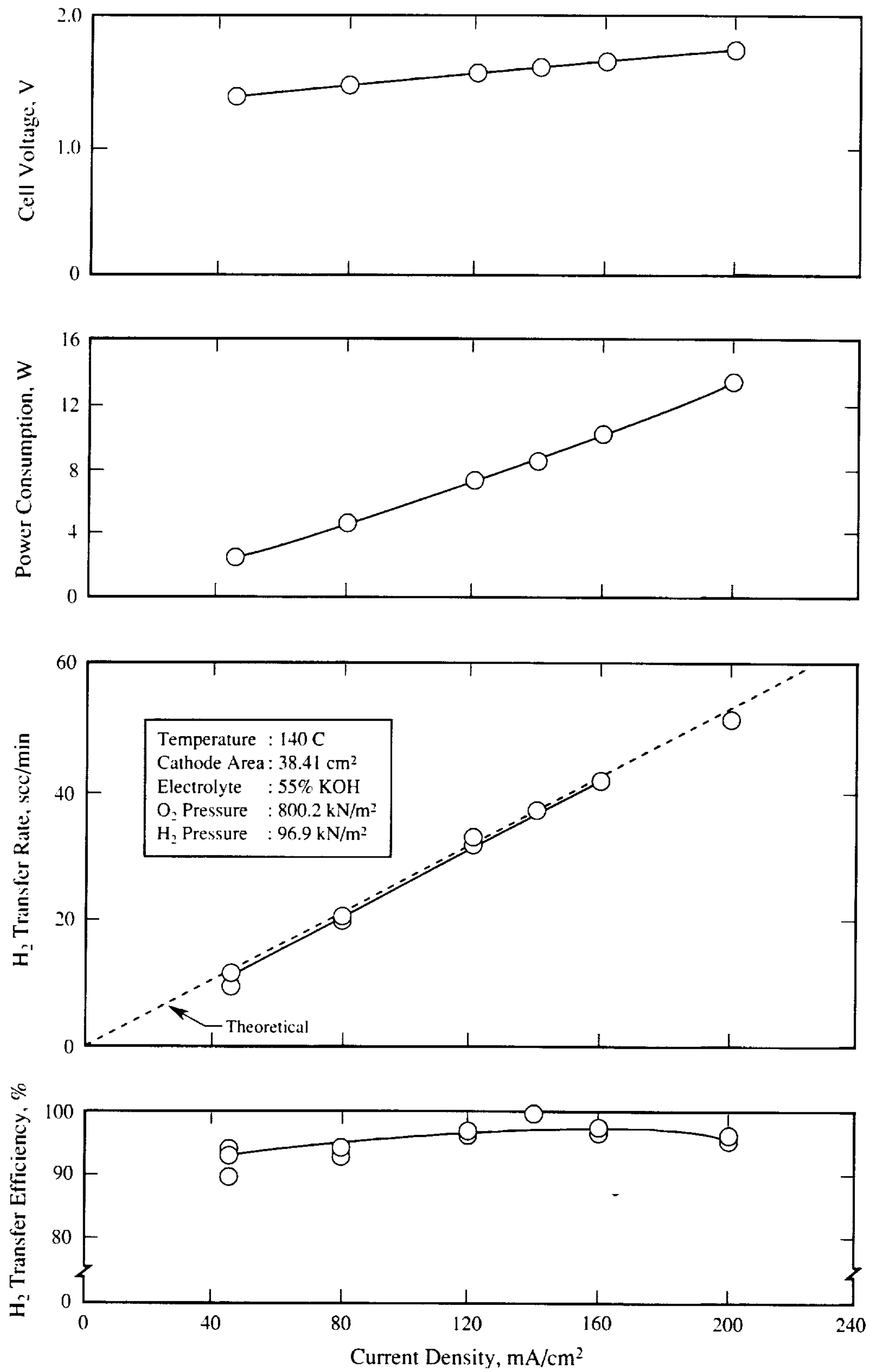

FIGURE 1-5 HYDROGEN TRANSFER CHARACTERIZATION

18.8 scc/min. This results in a molar ratio of H_2/CO_2 of 2.5:1. No methane was detected by chromatographic analyses of the product gases.

Prior to the start of the parametric testing, the cathode was filled with the methanation catalyst. This catalyst is 20% ruthenium (Ru) on alumina extrudates (0.16 cm in diameter). The inner diameter of the SMC is 0.27 cm, so the catalyst extrudates had to be inserted coaxially in the SMC. The total weight of catalyst used in the Breadboard IORS is 1.47 g, and the length of the catalyst is 38.5 cm. The catalyst is retained in the cathode by plugs of Pyrex glass wool.

The presence of catalyst in the SMC does not affect its performance during the electrolyses of water, as shown by the two data points in Figure 1-4.

1.3 Parametric Testing

The parametric testing involves studies of the effects of temperature, reactant gas composition and flow rate on the performance of the Breadboard IORS.

1.3.1 Temperature

Figure 1-6 shows the effects of temperature changes on the terminal voltage of the Breadboard IORS during the electrolysis of water. These data were obtained without CO_2 flowing through the cathode. Data are provided for two current densities, 45.6 and 160 mA/cm^2. The decrease in terminal voltage with increasing temperature is -2.3 mV/C and -3.7 mV/C, respectively, for the two current densities.

The effect of temperature changes on the methanation reaction was studied through testing of the Breadboard IORS and use of the mathematical model for the IORS. The Breadboard IORS was operated at the conditions listed in Table 1-1, and the conversion efficiencies of H_2 and CO_2 to CH_4 were measured. The measured conversion efficiencies are compared in Figure 1-7 to the efficiencies calculated using the model. This figure shows excellent correlation of the performance predicted by the model and the actual performance of the Breadboard IORS over the range of operating parameters tested.

The effect of temperature on the performance of the Breadboard IORS, as predicted by the model, is presented in Figure 1-8. Curves are provided for inlet CO_2 flow rates of from 2 to 20 scc/min.

1.3.2 Reactant Gas Composition

The reduction of CO_2 with H_2 consumes four moles of H_2 for every mole of CO_2 that reacts. However, future space craft are expected to have less H_2 available, so the methanation reaction will occur with a H_2/CO_2 molar ratio of less than 4.0.

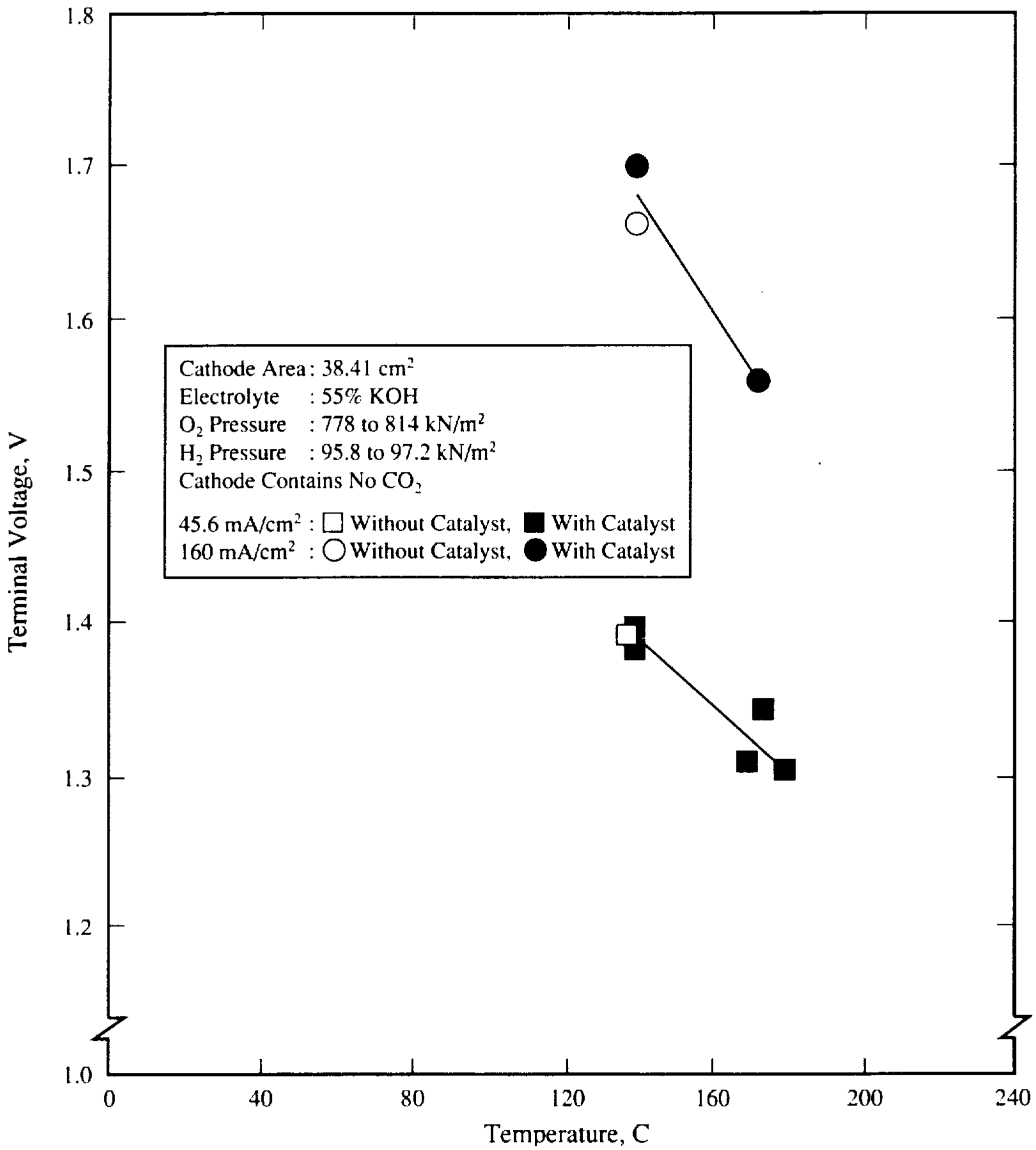

FIGURE 1-6 EFFECT OF TEMPERATURE ON ELECTROLYSIS VOLTAGE

TABLE 1-1 DATA FOR COMPARISON OF MEASURED AND CALCULATED CONVERSION EFFICIENCIES

Initial CO_2 Flow Rate, scc/min	H_2/CO_2 Molar Ratio	Temp., C	Conversion Efficiency, %(a) Hydrogen Actual	Hydrogen Calculated	Carbon Dioxide Actual	Carbon Dioxide Calculated
19.1	2.05	141.1	10.4	11.2	5.33	5.81
18.6	2.11	142.8	10.4	11.7	5.48	6.16
10.1	3.88	142.8	10.7	13.4	10.4	13.0
7.96	4.37	144.4	10.8	15.7	11.8	17.2
2.03	20.0	144.4	9.4	13.3	46.8	66.5
24.0	1.59	145.0	10.6	11.6	4.23	4.60
10.5	3.40	172.2	32.0	26.7	27.4	22.7
1.14	8.41	176.1	47.5	47.6	100	100
5.95	1.89	177.2	72.3	60.4	34.2	28.5
6.26	1.57	177.2	74.7	62.7	29.4	24.6
6.47	2.98	177.2	48.4	46.8	36.1	34.9
6.76	3.08	177.2	45.3	44.6	34.9	34.3
6.61	3.60	177.8	40.6	41.7	36.5	37.5
6.80	3.49	177.8	41.1	41.6	35.9	36.3
2.90	8.60	177.8	37.4	39.7	80.4	85.4
3.47	2.88	178.3	71.7	67.5	51.7	48.6
2.69	4.34	178.9	67.8	64.0	73.6	69.4
2.32	5.26	178.9	63.1	62.0	83.1	81.6
3.80	3.11	178.9	63.9	63.3	49.8	49.2
3.70	3.20	178.3	64.5	63.0	51.8	50.4
1.71	6.19	178.9	62.2	63.3	96.4	97.9
3.89	3.43	185.0	68.8	62.2	59.0	53.3

(a) Catalyst = 0.76 cc of 20% Ru on Alumina. O_2 Pressure = 723 to 800 kN/m^2. H_2 Pressure = 96 to 104 kN/m^2. Electrolyte = 55% KOH.

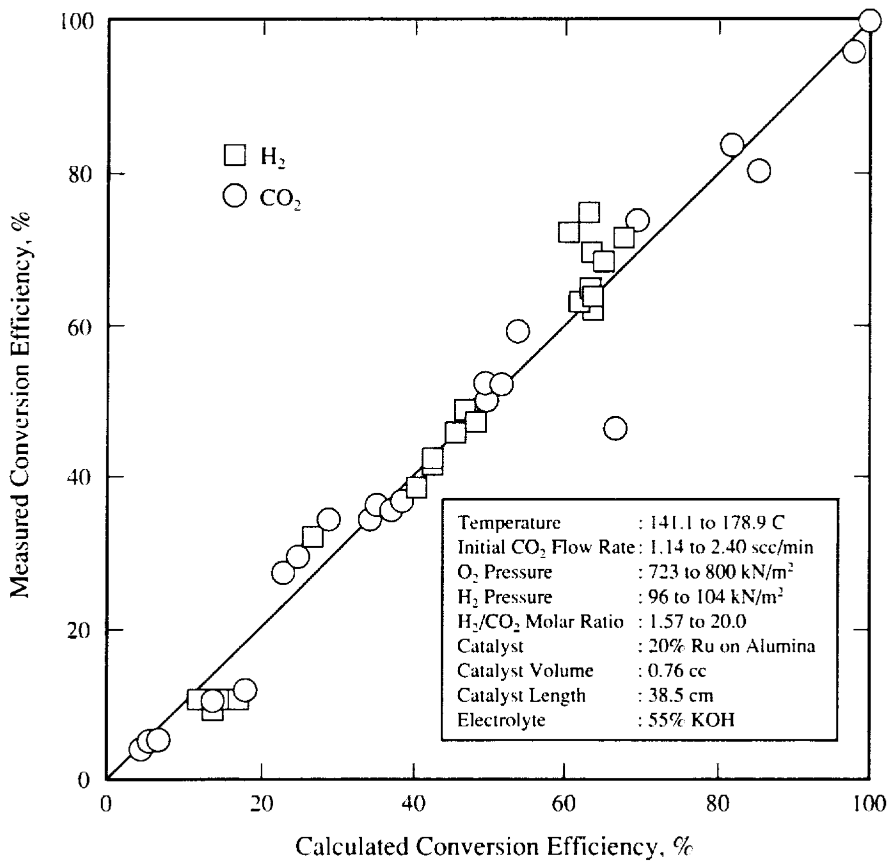

FIGURE 1-7 COMPARISON OF MEASURED AND CALCULATED CONVERSION EFFICIENCIES

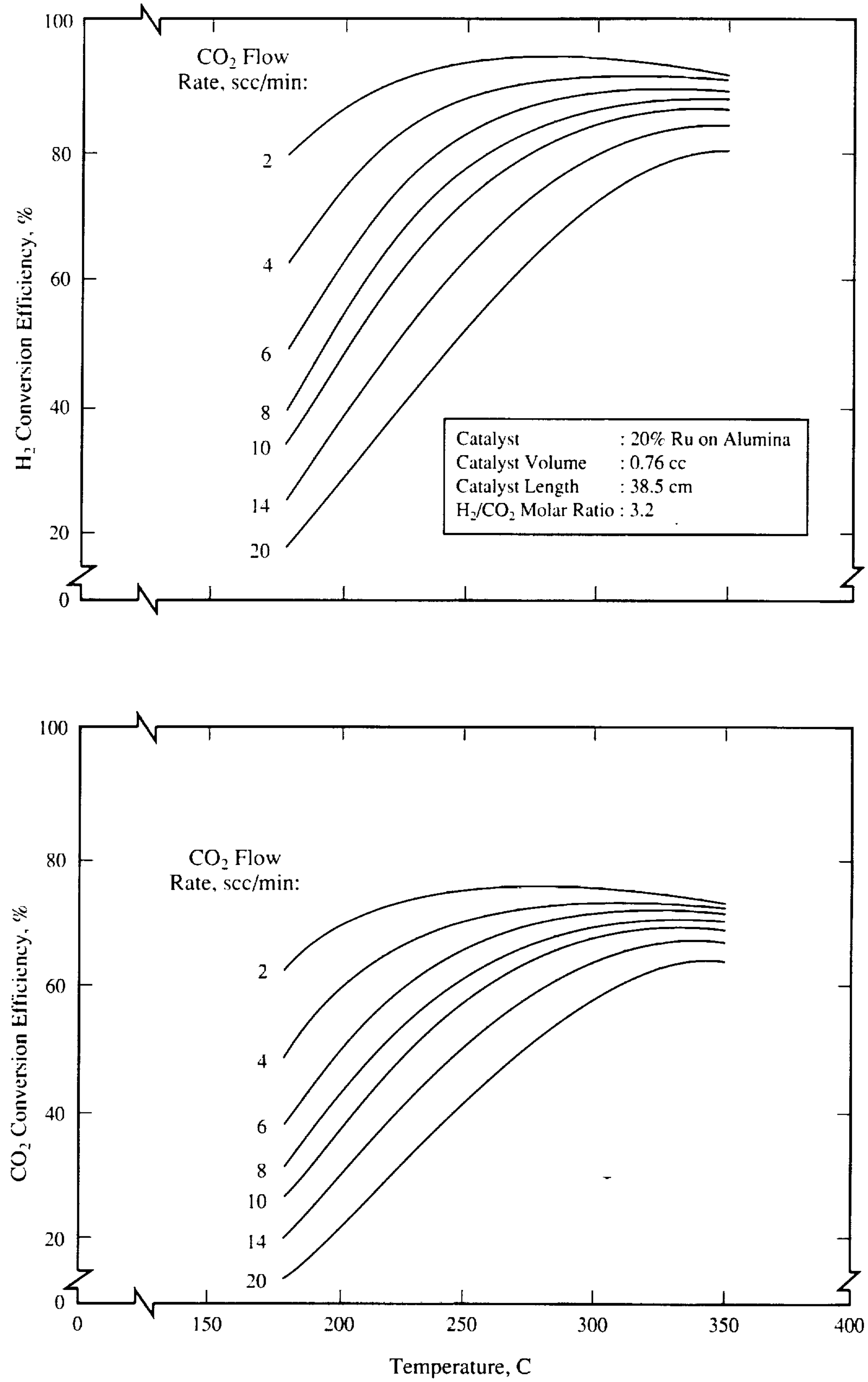

FIGURE 1-8 EFFECT OF TEMPERATURE ON METHANATION EFFICIENCY

To identify the molar ratio that may be expected in an end-item application, the specifications of Oxygen Generation Assembly (OGA) and the Carbon Dioxide Reduction Assembly (CReA) being developed for Space Station Freedom were reviewed. The OGA is to provide 4.65 kg O_2 per day during normal operation with a 4-person crew. The CReA is to process 4.0 kg CO_2 per day under normal (4-person) operating conditions. The CReA is to achieve 99% conversion of the lean feed constituent (i.e., H_2). Both assemblies operate continuously.

With this information, the molar ratio anticipated in an end-item IORS, operating with a 4-person crew, was calculated to be 3.2:1, assuming 100% transfer efficiency of the H_2 through the cathode.

Figure 1-9 shows the effect of the molar ratio on the conversion efficiency of the Breadboard IORS operating at 178 C. These data show the conversion efficiencies for both H_2 and CO_2. Data are shown for values of the initial flow rate of CO_2 into the Breadboard IORS from 2 to 20 scc/min.

1.3.3 Reactant Gas Flow Rate

As shown in Figure 1-8 there is a relationship between the flow rate of the gases and the efficiency of the conversion of the reactant gases to CH_4 and H_2O. This is also shown in Figure 1-10 where the conversion rates of CO_2 and H_2 are shown as functions of the inlet flow rate of CO_2.

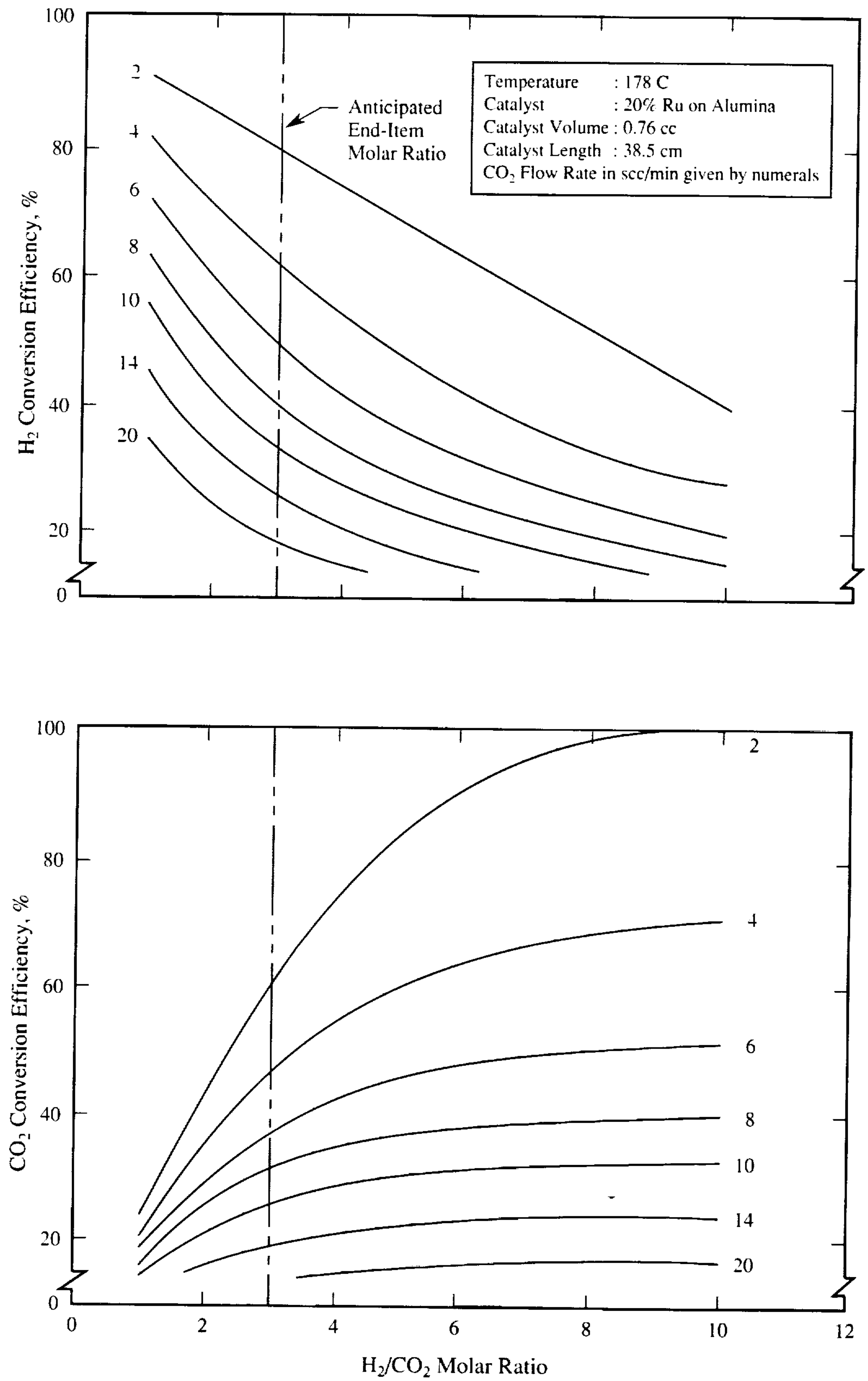

FIGURE 1-9 EFFECT OF REACTANT GAS COMPOSITION ON METHANATION EFFICIENCY

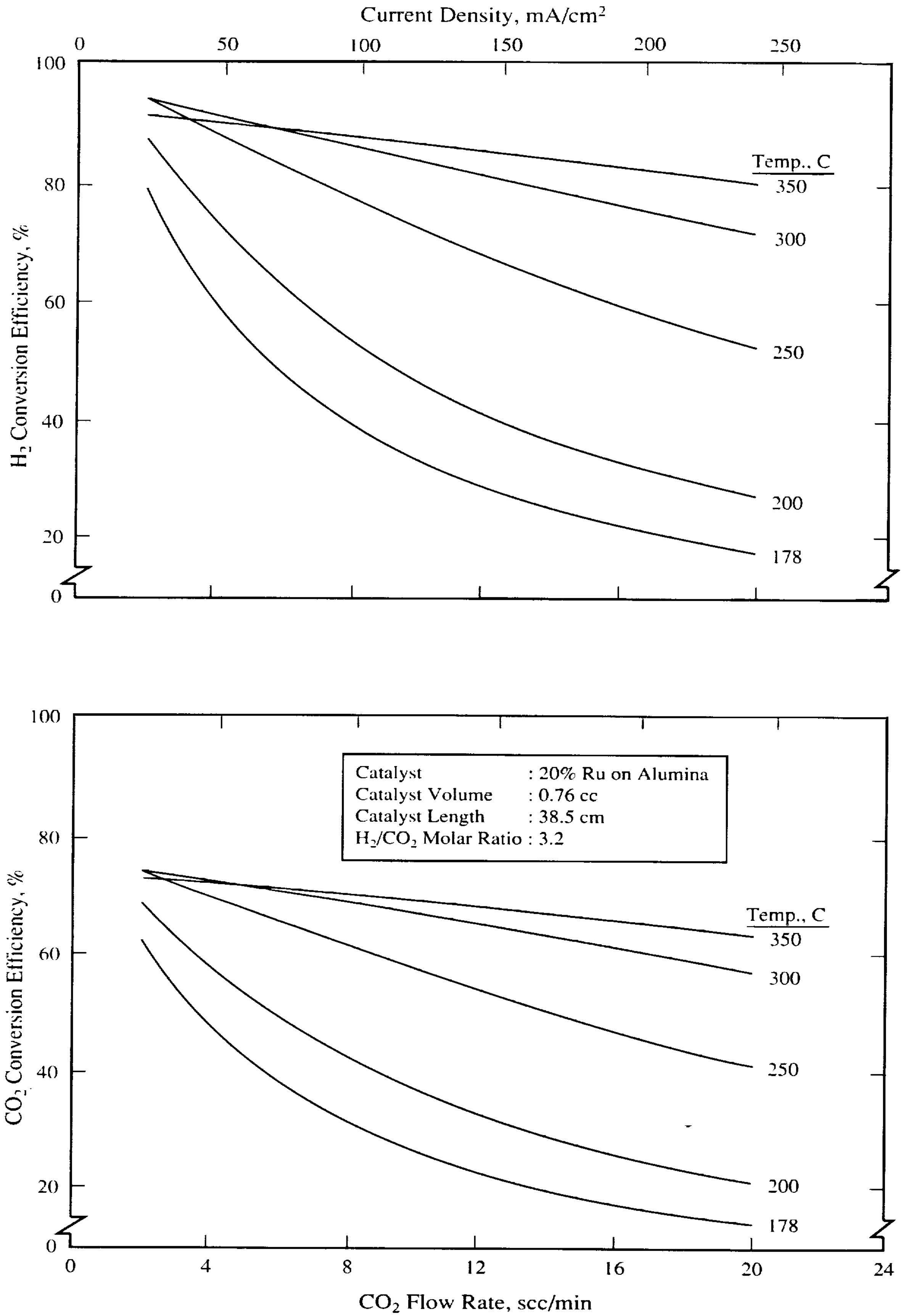

FIGURE 1-10 EFFECT OF INLET CO_2 FLOW RATE ON METHANATION EFFICIENCY

2.0 PROBLEMS

No problems have been encountered which may impede performance or impact program schedule or cost.

3.0 WORK TO BE PERFORMED NEXT REPORTING PERIOD

The parametric tests will be completed during the next reporting period. Based on the analysis of those results and the results summarized here, the preliminary process design of a 4-person IORS will be developed. The preliminary mathematical models presented in the prior Bimonthly Technical Progress Report (TR-1619-1-1) will be updated if necessary. Also, the Final Report will be prepared and submitted on or before 07/08/93.

4.0 COST STATUS

The status of the program's cost is summarized below, as required by Attachment J-2 of Contract No. NAS8-39843.

1.	Total cumulative costs as of 04/30/93:	$35,200
2.	Estimated cost to complete contract:	$14,800
3.	Estimated percentage of work completed:	70.4%

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
07 May 93

3. REPORT TYPE AND DATES COVERED
Bimonthly Technical Progress Rpt. 09 Mar 93-07 May 93

4. TITLE AND SUBTITLE
Integrated Oxygen Recovery System
Technical Progress Report No. 2

5. FUNDING NUMBERS
NAS8-39843

6. AUTHOR(S)
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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Life Systems, Inc.
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Cleveland, OH 44122

8. PERFORMING ORGANIZATION REPORT NUMBER
TR-1619-1-2

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Life Systems has conceptualized an innovative Integrated Oxygen Recovery System (IORS) applicable to advanced mission air revitalization. The IORS provides the capability to electrochemically generate metabolic oxygen (O_2) and recover O_2 from the space habitat atmosphere via a carbon dioxide (CO_2) reduction process within a single assembly. To achieve this capability, the IORS utilizes a novel Solid Metal Cathode (SMC) water electrolysis unit that simultaneously serves as the Sabatier CO_2 reduction reactor. The IORS enables two major life support systems currently baselined in closed loop air revitalization systems to be combined into one smaller, less complex system. This concept reduces fluidic and electrical interface requirements and eliminates a hydrogen (H_2) interface.

Life Systems is performing an evaluation of the IORS process directed at demonstrating performance and quantifying key physical characteristics including power, weight and volume. In this report, the results of the checkout, shakedown and initial parametric tests are summarized.

14. SUBJECT TERMS
Oxygen, carbon dioxide, reduction, hydrogen, electrochemical
Solid Metal Cathode, air revitalization, methanation, Sabatier

15. NUMBER OF PAGES
22

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	UL