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# EVALUATION OF THE ELECTROCHEMICAL $O_2$ CONCENTRATOR AS AN $O_2$ COMPRESSOR

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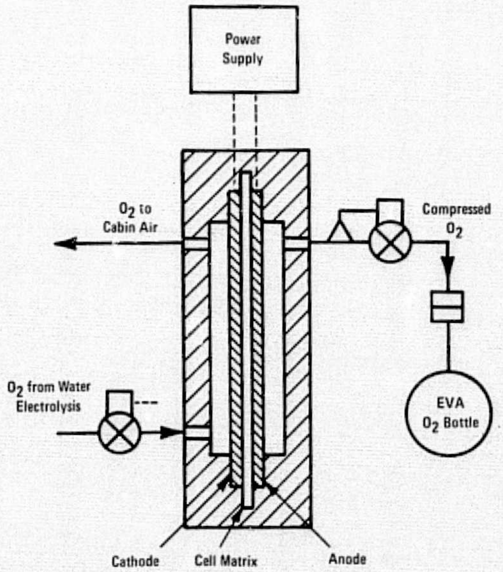
## FINAL REPORT

by

**R. D. Marshall and J. N. Carlson**

August, 1975

ELECTROCHEMICAL $O_2$ COMPRESSION



Electrochemical Cell Reactions

Cathode: $O_2 + 2H_2O + 4e^- = 4OH^-$

Anode: $4OH^- = O_2 + 2H_2O + 4e^-$

Power

Overall: $O_2$ (Low Pressure) = $O_2$ (High Pressure)

Prepared Under Contract No. NAS2 - 7704

by

*Life Systems, Inc.*
Cleveland, Ohio 44122

for

## AMES RESEARCH CENTER

National Aeronautics & Space Administration

ER-195-7

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# FOREWORD

The development work described herein was conducted by Life Systems, Inc. under NASA Contract NAS2-7704 during the period July, 1973 through May, 1975.

The Program Manager was Richard D. Marshall. Technical support was provided by Jan N. Carlson, J. David Powell, and John W. Shumar in the chemical, electrical, and product assurance areas, respectively.

The technical management of the program was under the direction of Mr. P. D. Quattrone, Chief, Environmental Control Research Branch, NASA Ames Research Center, Moffett Field, California.

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## SUMMARY

A program was successfully completed to analytically and experimentally evaluate the feasibility of using an electrochemical oxygen ($O_2$) concentrator as an $O_2$ compressor. The Electrochemical $O_2$ Compressor (EOC) would compress 345 kN/$m^2$ (50 psia) $O_2$ generated on board the space vehicle by the Water Electrolysis Subsystem (WES) in a single stage to 20,700 kN/$m^2$ (3000 psia) to refill spent extravehicular equipment $O_2$ bottles and therefore eliminate the need for high pressure $O_2$ storage. The EOC was found to be preferable to high pressure $O_2$ storage for space mission resupply periods greater than 150 days. Initial long-term manned space missions will have shorter resupply periods of 90 days or less. Considerable development time and funds will be required to develop the EOC for extravehicular activity (EVA) application. It was recommended, therefore, that additional development efforts be postponed pending the evaluation of alternate $O_2$ compression concepts for EVA application.

The single cell EOC designed, fabricated, and used for the feasibility testing is capable of being tested at $O_2$ pressures up to 41,400 kN/$m^2$ (6000 psia). The cell was designed for flexibility to accommodate testing of membranes and electrodes of various thicknesses. The flow through gas cavity geometry allows $O_2$ to circulate through the cathode compartment and electrolyte to circulate through the anode compartment.

A ground support test facility to test the EOC cell was designed, fabricated, and used for the EOC feasibility testing. The test facility is equipped with the instrumentation required to control and monitor performance for cell current densities of 0 to 400 mA/$cm^2$ (0 to 372 ASF) and $O_2$ compression pressures up to 10,300 kN/$m^2$ (1500 psia). The test facility monitors cell voltage, temperature, pressure and differential pressure and provides fail-safe shutdown, including an inert gas purge should one of these parameters exceed a preset level.

A Product Assurance Program was established, implemented, and maintained. The Product Assurance activities emphasized safety and materials compatibility associated with high pressure $O_2$ operation. A total of 17 safety features were incorporated into the test facility and cell design as a result of the effort. Strict materials controls were imposed on the selection of materials because of the severe combustion hazard associated with high pressure $O_2$. Nickel, inconel and 316 stainless steel were the only metallic parts exposed to high pressure $O_2$. The nonmetallic materials used were polysulfone, Teflon, Kel F and Viton A. Krytox lubricant was used exclusively for all lubrication requirements on regulators and valves. All components were cleaned for $O_2$ service prior to incorporation into the test facility. A Material Data Summary was prepared to insure that each component met the strict material requirements for $O_2$ compatibility.

A membrane development program was conducted to develop a membrane for EOC application. A literature survey selected fluorinated hydrocarbon base membranes for the application. Data obtained using a commercially available membrane were used to guide the development of the membranes fabricated specifically for an EOC. A total of 15 membranes were fabricated having a fluorinated hydrocarbon

base with various functional groups grafted and crosslinked into the membrane. The membranes were compatible with potassium hydroxide (KOH) and high pressure $O_2$, and had a very low $O_2$ permeability for approximately 100% current efficiency during cell operation. Membrane resistance, differential pressure tolerance, and electrolyte permeability data were gathered to screen those membranes which did not meet the specifications required prior to incorporation into an EOC cell. Eleven of the 15 membranes were capable of withstanding differential pressures greater than or equal to 20,700 kN/$m^2$ (3000 psia). Of the 11 membranes that passed the differential pressure test, six membranes met the low resistance specification of less than or equal to 1 ohm-$cm^2$ (0.16 ohm-$in^2$). None of the membranes fabricated, however, were capable of meeting the low electrolyte permeability with respect to membrane resistance specifications required to run an EOC without an anode electrolyte reservoir to maintain moisture balance.

Test activities completed included the test facilities checkout and calibration tests, a shakedown test, and EOC cell parametric tests conducted using a commercial membrane and the customized membranes developed. The testing culminated in a 213-hour cell Design Verification Test (DVT) at a current density of 100 mA/$cm^2$ (92 ASF), and a differential pressure of 10,350 kN/$m^2$ (1500 psid). The average cell voltage during the DVT was 1.4V.

Two supporting technology tasks were completed to support the EOC development. An analytical and experimental evaluation of the $O_2$ backdiffusion process was completed, verifying that membrane $O_2$ permeability is very low and capable of giving current efficiencies close to 100%. A literature search was completed which identified materials of construction compatible with high pressure $O_2$ and KOH for use in the cell and test facility.

# INTRODUCTION

High pressure gaseous oxygen ($O_2$) is required to refill extravehicular $O_2$ supply tanks for longer duration space missions having longer resupply periods and increased extravehicular activity (EVA). The weight and volume penalties associated with high pressure $O_2$ storage are excessive. An alternate to high pressure $O_2$ storage is to utilize the on-board spacecraft $O_2$ generating system and then compress the $O_2$ to the desired pressure. One method for compressing the $O_2$ for EVA and other manned spacecraft applications would be to use an electrochemical $O_2$ concentrator as an $O_2$ compressor.

## Background

High pressure $O_2$ bottle storage was selected for EVA application because of high reliability, minimum weight and volume, and the ability to rapidly provide $O_2$ during emergency situations.(1,2) A method of refilling spent $O_2$ bottles at 20,700 to 41,400 kN/$m^2$ (3000 to 6000 psia) is required at the primary space vehicle, i.e., Earth Orbiting Space Station, Lunar Base, or Mars Base. High pressure gaseous $O_2$ storage represents a simple, reliable and effective means of storing $O_2$ for EVA application. The weight and volume penalties associated with storage and logistics of expendable high pressure $O_2$ increase with increased

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(1) References in parentheses are cited on page 64.

resupply periods and EVA mission requirements. A regenerable method of refilling $O_2$ bottles is required. The concept would utilize $O_2$ generated by a Water Electrolysis System (WES) on board the spacecraft at 103 to 690 kN/$m^2$ (15 to 100 psia) and compress the $O_2$ to 20,700 to 41,400 kN/$m^2$ (3000 to 6000 psia).

Oxygen compression techniques can be divided into direct mechanical compression and electrochemical compression techniques. A third method of generating high pressure $O_2$ is cryogenic $O_2$ storage which takes advantage of the cold space environment during non-sunlit side operation. The cryogenic $O_2$ is then heated to generate $O_2$ at the desired pressure.

### Direct Mechanical Compression

Direct mechanical compression methods consist of the standard electrically operated $O_2$ compressor and an $O_2$ booster pump(3) which uses energy obtained from the $O_2$ flow rate and pressure to compress the $O_2$ to the desired pressure.

Commercially available $O_2$ compressors compress $O_2$ in two to four stages and are generally heavy (over 454 kg (1000 lb)), bulky (over 0.4 $m^3$ (15 $ft^3$)), consume 746 to 1491 W (1 to 2 hp) of power, and are very noisy. Additional studies conducted at Life Systems in parallel to the present program, however, indicate that a customized $O_2$ compressor could weigh as little as 11.4 to 22.7 kg (25 to 50 lb), have a volume of 0.003 $m^3$ (0.1 $ft^3$) and consume only 298 to 373 W (0.4 to 0.5 hp).

The booster pump approach is a method best adapted to high flow rates and low compression ratios of three to four. Additional studies are required to determine if the concept is adaptable to the low $O_2$ flow rates and high compression ratios of 30 to 60 required for the application.

### Electrochemical $O_2$ Compression

Two electrochemical $O_2$ compression methods were identified. The first is simply to use a WES to generate $O_2$ at the desired pressure. The primary disadvantages of the high pressure WES are the safety hazards associated with high pressure $O_2$ and hydrogen ($H_2$) in close proximity, and low current efficiencies caused by $O_2$ and $H_2$ backdiffusion.

The second electrochemical method uses an electrochemical $O_2$ concentrator to consume $O_2$ at the cathode and compress $O_2$ generated at the anode by controlling the anode gas pressure. The process can be carried out in an acid, solid polymer or base electrolyte. The latter is the subject of the present program and is discussed in detail in the following sections.

## Program Objectives and Organization

The objective of this program was to determine the feasibility of utilizing an EOC to pressurize $O_2$ from ≤690 kN/$m^2$ (100 psia) to a range of 20,700 to 41,400 kN/$m^2$ (3000 to 6000 psia) for EVA application. The program was divided into seven tasks whose specific objectives were to:

1. Perform an EOC end-item application analysis to establish the system design specifications and a preliminary system design to serve as a foundation for subsequent cell hardware designs.

2. Design, fabricate, and assemble a single cell EOC capable of being tested at ≤1.0V at ≥100 mA/cm$^2$ (92.9 ASF) and pressures ≥20,700 kN/m$^2$ (3000 psia).

3. Design, fabricate, and assemble the ground support test facility to test the electrochemical cell.

4. Establish, implement, and maintain a Product Assurance Program consistent with the end-item EVA application, and emphasizing safety and materials compatibility with a high pressure $O_2$ atmosphere.

5. Identify, develop, and evaluate electrolyte membranes for EOC application. The following membrane properties were used as a goal:

   a. Differential pressure tolerance of ≥20,700 kN/m$^2$ (3000 psid).
   b. Compatibility with KOH and high pressure $O_2$.
   c. Low resistance (≤1.0 ohm-cm$^2$).
   d. Low electrolyte permeability (≤1.4 x 10$^{-6}$ g/hr-cm$^2$-kN/m$^2$ (10$^{-5}$ g/hr-cm$^2$-psi)).
   e. High resistance to $O_2$ backdiffusion for ∿100% current efficiency.

6. Perform single cell EOC parametric and design verification feasibility tests to determine the effect of current density, differential pressure, and operating time on cell performance for various cell configurations.

7. Conduct materials compatibility evaluations and an $O_2$ backdiffusion analysis to support the EOC technology development and feasibility evaluation.

The objectives of the program were met. The following sections summarize the results obtained, and the conclusions and recommendations reached.

## ELECTROCHEMICAL OXYGEN COMPRESSION

Oxygen is compressed from a low pressure $O_2$ stream to a higher pressure in an electrochemical cell consisting of two porous electrodes separated by electrolyte solution of aqueous potassium hydroxide (KOH). The electrolyte is retained between the electrodes in a membrane capable of withstanding the differential pressure resulting from the compression of $O_2$. Compartments adjacent to the electrodes provide passageways for manifolding the $O_2$ into and out of the cell.

On-board generated $O_2$ from the Water Electrolysis Subsystem (WES) is electrochemically consumed at the cathode of the electrochemical cells and regenerated at the anode. The pressure of the gas generated at the anode is controlled to an elevated pressure for refilling the high pressure $O_2$ bottles. Unused $O_2$ at the cathode exhausts to the cabin. The electrochemical $O_2$ compression process requires power and generates heat.

## Design Specifications

A preliminary estimate of EVA mission requirements and the $O_2$ bottle requirements are given in Table 1.(1,2) An EOC would not be applicable to Space Shuttle EVA missions because only 14 $O_2$ bottles are required. For longer resupply periods and increased EVA missions, such as will be required for a Space Station or Lunar Base, the number of $O_2$ bottles required increases and on-board $O_2$ compression becomes attractive. Future space missions such as a Mars Base which will not be resupplied would necessitate on-board $O_2$ compression.

The EOC design specifications to refill spent $O_2$ bottles are given in Table 2. The desired recharge period is 8 to 12 hours for a bottle containing 0.59 kg (1.3 lb) of $O_2$. The EOC current required for each compression stage at 100% current efficiency is 247A for an eight-hour recharge and 165A for a 12-hour recharge.

## Process Description

A functional diagram showing the major EOC cell components and electrochemical reactions is presented in Figure 1. On-board spacecraft generated $O_2$ enters the cathode compartment of the EOC cell. When DC power is supplied to the cell electrodes, $O_2$ at the cathode reacts with electrons and water to form hydroxyl ions ($OH^-$) within the electrolyte. The equation for the cathode half-cell reaction is:

$$O_2 + 2H_2O + 4e^- = 4OH^- \tag{1}$$

Hydroxyl ions generated at the cathode migrate through the electrolyte-membrane to the anode. At the anode, $OH^-$ react to form water, electrons, and $O_2$. The equation for the anode half-cell reaction is:

$$4OH^- = O_2 + 2H_2O + 4e^- \tag{2}$$

The electrolyte concentration gradient between the electrodes causes the water generated at the anode to diffuse to the cathode for reaction with more $O_2$.

The pressure of the $O_2$ generated at the anode is controlled by a backpressure regulator to a higher pressure than that of the feed $O_2$ at the cathode, thus effecting the compression process.

### Process Requirements

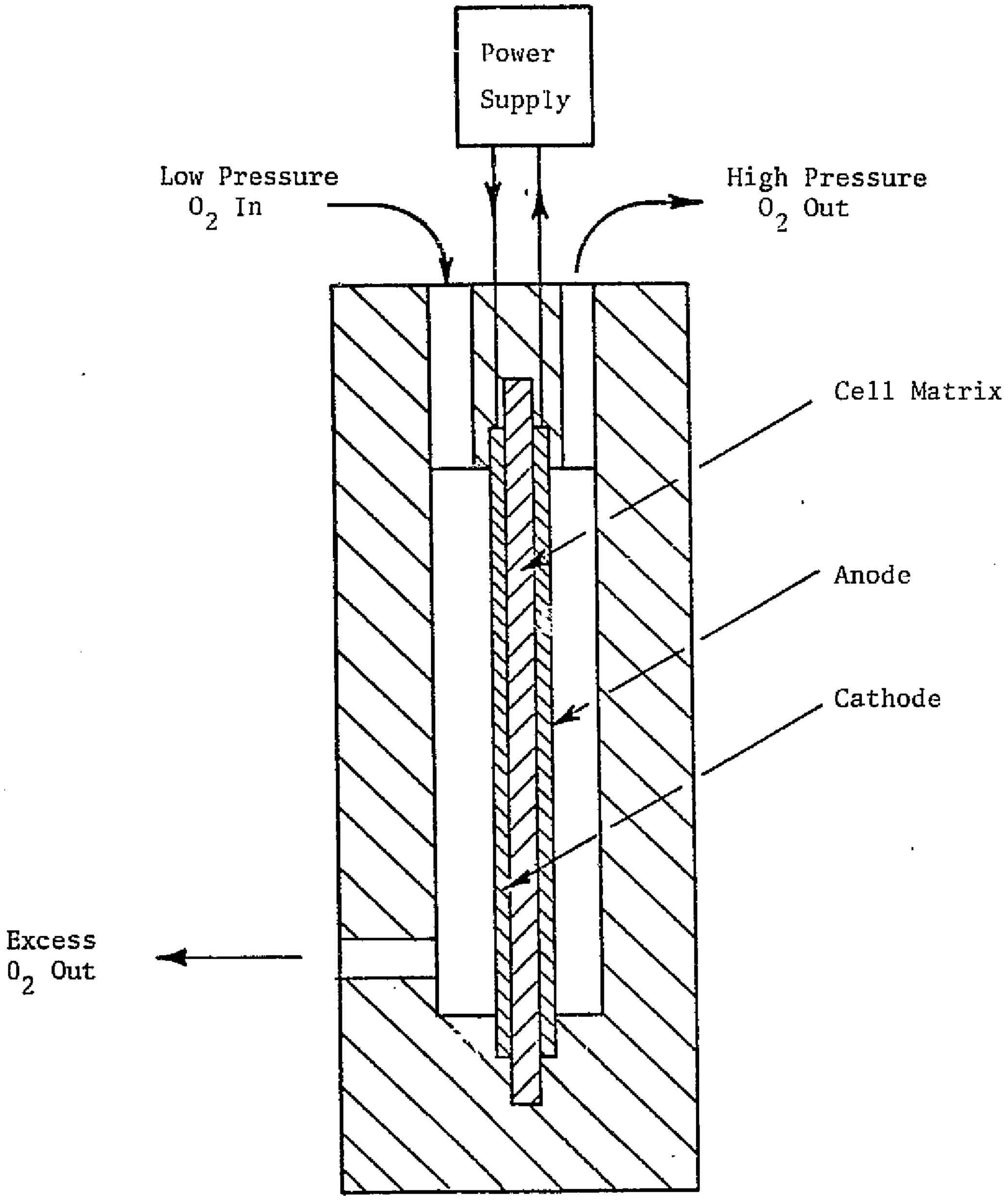
The rate at which $O_2$ is compressed is proportional to the amount of current flowing through the cell. According to Faraday's Law of Electrolysis, the $O_2$

TABLE 1 PRELIMINARY ESTIMATE OF EVA MISSION REQUIREMENTS

| | Space Shuttle | Space Station | Lunar Base | Mars Base |
|---|---|---|---|---|
| Estimated Number of Missions | 30 | 500 | 500 | 500 |
| EVA Mission Duration, Hr | 4 | 4 | 8 | 8 |
| Number of Men/EVA | 2 | 2 | 2 | 2 |
| Resupply Period, Day | 7 | 90-180 | 180-360 | None |
| $O_2$ Required at Average Metabolic Rate, kg (Lb) | 8.2 (18) | 139 (307) | 278 (613) | 278 (613) |
| $O_2$ Bottles Required at 0.59 kg $O_2$/Bottle (1.3 Lb $O_2$/Bottle) | 14 | 236 | 472 | 472 |
| Bottle Weight, kg (Lb) | | | | |
| at 0.89 kg/kg $O_2$ and 3000 Psia | 7.3 (16) | 124 (273) | 248 (546) | 248 (546) |
| at 1.0 kg/kg $O_2$ and 6000 Psia | 8.2 (18) | 139 (307) | 278 (613) | 278 (613) |
| Volume of Bottles, $m^3$ ($Ft^3$) | | | | |
| at 3000 Psia and 0.00362 $m^3$/kg $O_2$ (0.058 $Ft^3$/Lb $O_2$) | 0.029 (1.04) | 0.498 (17.8) | 0.997 (35.6) | 0.997 (35.6) |
| at 6000 Psia and 0.0068 $m^3$/kg $O_2$ (0.027 $Ft^3$/Lb $O_2$) | 0.014 (0.49) | 0.232 (8.3) | 0.045 (16.6) | 0.045 (16.6) |

## TABLE 2 EOC DESIGN SPECIFICATIONS

| | |
|---|---|
| $O_2$ Bottle Recharge: | |
| $O_2$ Required/Recharge, kg (Lb) | 0.59 (1.3) |
| Recharge Time, Hr | 8-12 |
| $O_2$ Storage Pressure, $kN/m^2$ (Psia) | 2.07-4.14 x $10^4$ (3000-6000) |
| $O_2$ Feed: | |
| Flow, kg/d (Lb/Day) | ≥1.18-1.77 (2.6-3.9) |
| Temperature, K (F) | 294 (70) |
| Pressure, $kN/m^2$ (Psia) | 103-6900 (15-100) |
| Dew Point, K (F) | 287 (57) |
| $O_2$ Exhaust: | |
| Flow, kg/d (Lb/Day) | 1.18-1.77 (2.6-3.9) |
| Temperature, K (F) | 298 (77) |
| Pressure, $kN/m^2$ (Psia) | 2.07-4.14 x $10^4$ (3000-6000) |
| Dew Point, K (F) | 287 (57) |



Electrochemical Cell Reactions

| | |
|---|---|
| Cathode: | $O_2 + 2H_2O + 4e^- = 4OH^-$ |
| Anode: | $4OH^- = O_2 + 2H_2O + 4e^-$ |
| Total: | $O_2$ (Cathode) = $O_2$ (Anode) |

FIGURE 1 FUNCTIONAL EOC CELL SCHEMATIC

compression rate is given by the equation:

$$W = 8.3 \times 10^{-8} (N)(I)(\eta) \tag{3}$$

where

$W = O_2$ compression rate, kg/s

$N$ = Number of series connected cells

$I$ = Cell current, A

$\eta$ = Current efficiency

As the amount of current flowing through the cell increases, each electrode polarizes, thereby increasing cell voltage. The cell voltage required to maintain a given current is positive (i.e., the process requires power), and is a function of temperature, pressure, cell internal resistance and electrode overvoltages.

Theoretical Cell Voltage. The theoretical cell voltage is given by the Nernst equation as:

$$E_{th} = E_o + \frac{RT}{nF} \ln P_2/P_1 \tag{4}$$

where

$E_{th}$ = Theoretical cell voltage, V

$E_o$ = Standard cell potential, V

$R$ = Universal gas constant, 8.31 J $K^{-1}mol^{-1}$

$T$ = Absolute temperature, K

$n$ = Number of equivalents per mole, 4 eq $mol^{-1}$

$F$ = Faraday's constant, 96,487 coulomb/eq

$P_2$ = Final $O_2$ pressure, $kN/m^2$

$P_1$ = Initial $O_2$ pressure, $kN/m^2$

To compress $O_2$ from 103 to 20,700 $kN/m^2$ (15 to 3000 psia) the theoretical cell voltage is only 0.034V and for calculation purposes can be assumed to be zero over the entire $O_2$ compression range.

Power Required. The power required by the EOC is given by the equation:

$$P = (N)(I)(E) \tag{5}$$

where

P = Power, W

E = Cell voltage, V

Heat Generated. Cell inefficiencies cause cell voltage to be above the theoretical voltage. The excess power is dissipated as waste heat within the cell. Heat removal must therefore be provided to maintain a constant cell temperature. The heat generated is given by the equation:

$$Q = (N)(I)(E-E_{th}) \tag{6}$$

where

Q = Heat generated, J/s

The amount of heat rejected is equal to the power required since the theoretical cell voltage is approximately zero.

Performance Parameters

The performance of an EOC is described by four parameters: differential pressure, cell voltage, current (Faradic) efficiency and moisture balance.

Differential Pressure. The differential pressure at which an EOC cell can operate is a function of the structure of the electrolyte retaining membrane (pore size, thickness and material) and how the membrane is supported between the electrodes. Higher cell differential pressure capability reduces the number of stages required for the $O_2$ compression process. Each additional stage means that an additional and equal increment of power is required and heat is generated. It is therefore desirable to operate at the highest differential pressure attainable with a single $O_2$ compression stage as a goal.

Cell Voltage. Cell voltage directly determines the power required and heat generated by the electrochemical process and is therefore a direct indication of process efficiency. Cell voltage increases with current density, the anode to cathode pressure ratio, cell internal resistance and electrode overvoltage, and decreases with temperature.

Current Efficiency. Cell current efficiency is defined as the ratio of the actual cell $O_2$ output to the theoretical cell output, $\eta = 1$ (Equation 3). Current efficiency is a function of the amount of $O_2$ that diffuses back through the matrix from the anode to the cathode under the $O_2$ partial pressure gradient:

$$\eta = 1 - \frac{D\ (P_2-P_1)}{(k)(t)(i)} \tag{7}$$

where

D = Diffusion constant, $cm^2/sec$

$\beta$ = $O_2$ solubility constant, $cm^3\ O_2/(cm^3)(kN/m^2)$

$P_1$, $P_2$ = Initial and final $O_2$ pressures, $kN/m^2$

k = $O_2$ generation constant from Faraday's Law

t = Membrane thickness, cm

i = Cell current density, $A/cm^2$

The $O_2$ backdiffusion rate depends on the solubility of $O_2$ in the electrolyte and membrane, cell differential pressure and cell operating temperature. As current efficiency decreases, the amount of current and, hence, power required and heat generated increases.

Moisture Balance. At steady-state the amount of water leaving the EOC in the compressed $O_2$ and unreacted $O_2$ streams must equal the amount of water entering the $O_2$ feed stream. Although there is no overall water generation or consumption within the cell, water is generated at the anode (Equation 2) and must transfer through the electrolyte retaining membrane to the cathode for consumption (Equation 1).

The two major driving forces within the cell which cause water to transfer from anode to cathode are the cell differential pressure and the electrolyte concentration gradient. Anode to cathode differential pressure simply forces water and bulk electrolyte through the electrolyte retaining membrane. The effect of the electrolyte concentration gradient is more complex since the concentration gradient is a function of the cell electrical potential gradient which is a function of current density. The electrolyte concentration gradient, and hence the anode to cathode water transfer rate increases with current density.

The rate at which water transfers through the electrolyte retaining membrane due to cell differential pressure and electrolyte concentration gradients must be greater than or equal to the water generation-consumption rate at steady-state. Insufficient water transfer caused by low membrane water permeability would result in an accumulation of water at the anode. When the water transfer rate equals the water generation-consumption rate, moisture balance can be maintained by controlling the inlet $O_2$ moisture content at a level in equilibrium with the desired catholyte concentration. When the transfer rate exceeds the consumption-generation rate, an electrolyte reservoir at the anode is required to supply the electrolyte that transfers through the cell into the cathode compartment. The amount of electrolyte that transfers for a given set of operating conditions is a function of the physical properties of the electrolyte retaining membrane.

## Preliminary EOC System Designs

Two EOC system designs were evaluated. The first design was based on developing an electrolyte-retaining membrane capable of maintaining moisture balance without an electrolyte reservoir. This simplified EOC system design represents the optimum or lowest total spared system equivalent weight design envisioned. Development of an electrolyte membrane having the desired resistance and water permeability properties, however, would be difficult, if even possible. A second system design having an electrolyte reservoir at the anode to maintain cell moisture balance was therefore considered as an alternative to the simple system design.

## Optimum EOC System Design

Figure 2 shows the optimum EOC system flow schematic. The component characteristics including number required, power, weight and volume are given in Table 3.

Oxygen from the WES enters the electrochemical modules and is manifolded to the cathode compartments of the cells. The $O_2$ is then electrochemically compressed to the desired storage pressure, which is mechanically controlled using a backpressure regulator. The $O_2$ not consumed in the electrochemical cells is manifolded to the spacecraft cabin. An $O_2$ bottle is simply connected to the high pressure line and allowed to slowly fill across the backpressure regulator. Nitrogen ($N_2$) purge is provided during startup and shutdown sequences. Heat rejection is provided using a circulating liquid coolant loop through the electrochemical modules with the heat generated in the process rejected directly to spacecraft liquid coolant via a liquid-liquid heat exchanger.

## EOC System Design with Electrolyte Reservoir

Figure 3 is a schematic of an EOC system with an electrolyte reservoir connected to the anode compartments of the electrochemical cells. The component characteristics, including number required, power, weight, and volume are given in Table 4.

Oxygen from the WES enters the electrochemical modules and is manifolded to the cell cathode compartments. Oxygen is electrochemically compressed to the desired storage pressure at the anode. The $O_2$ not consumed at the cathode is manifolded to the spacecraft cabin. A gas/liquid separator is used to remove electrolyte that transfers into the cathode compartment. The electrolyte separated from the cathode exhaust is collected in an accumulator for reuse. Makeup electrolyte is added at the anode through an electrolyte feed accumulator attached to a circulating electrolyte loop. High pressure $O_2$ is then separated from the electrolyte loop for use in refilling $O_2$ bottles in a gas/liquid separator. The electrolyte collected from the cathode compartments is used to refill the feed accumulator after each compression cycle.

Nitrogen purge is provided during startup and shutdown sequences. Heat rejection is provided by a liquid/liquid heat exchanger located in the circulating electrolyte loop. Heat is rejected directly to spacecraft liquid coolant.

## Comparison to High Pressure $O_2$ Storage

The two EOC system designs were compared to high pressure $O_2$ storage as a function

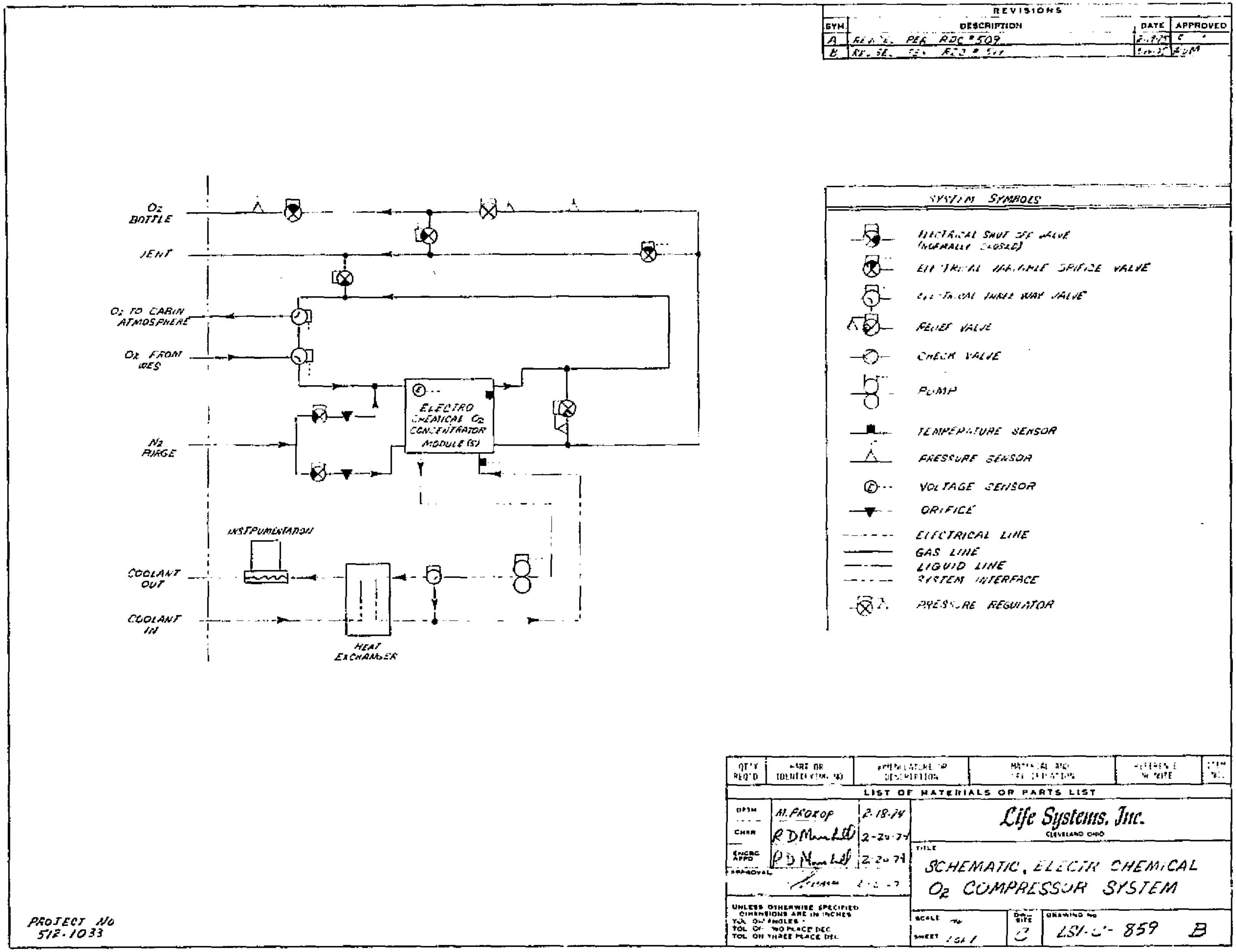


FIGURE 2 OPTIMUM EOC SYSTEM SCHEMATIC

## TABLE 3 EOC SYSTEM COMPONENT CHARACTERISTICS

| Component | No. Req | Weight,[a] kg (Lb) | Volume,[b] $cm^3$ ($In^3$) | Power, W[c] |
|---|---|---|---|---|
| Module, EOC | 1 | 11.35 (25.0) | 3769.7 (230.0) | Variable |
| Valve, Electrical, Normally Closed | 3 | 0.36 (0.8) | 753.9 (46.0) | None |
| Valve, Electrical, Three-Way | 3 | 0.36 (0.8) | 753.9 (46.0) | None |
| Valve, Electrical, Variable Orifice | 3 | 0.36 (0.8) | 753.9 (46.0) | None |
| Valve, Relief | 1 | 1.18 (2.6) | 3245.2 (198.0) | None |
| Regulator, Backpressure | 1 | 1.18 (2.6) | 3245.2 (198.0) | None |
| Restrictor, Flow | 2 | 0.09 (0.2) | 1.6 (0.1) | None |
| Pump, Liquid | 1 | 0.45 (1.0) | 1103.0 (67.3) | 12 |
| Heat Exchanger, Liquid-Liquid | 1 | 1.73 (3.8) | 819.5 (50.0) | None |
| Sensor, Temperature | 2 | 0.32 (0.7) | 622.8 (38.0) | 5 |
| Sensor, Pressure | 2 | 0.18 (0.4) | 278.6 (17.0) | 5 |
| Instrumentation | 1 | 4.54 (10.0) | 7457.5 (455.0) | 10 |

---

(a) Total weight, kg (Lb): 24.9 (54.8)
(b) Total component volume, $dm^3$ ($Ft^3$): 28.2 (1.0)
(c) Total component power, W: 42

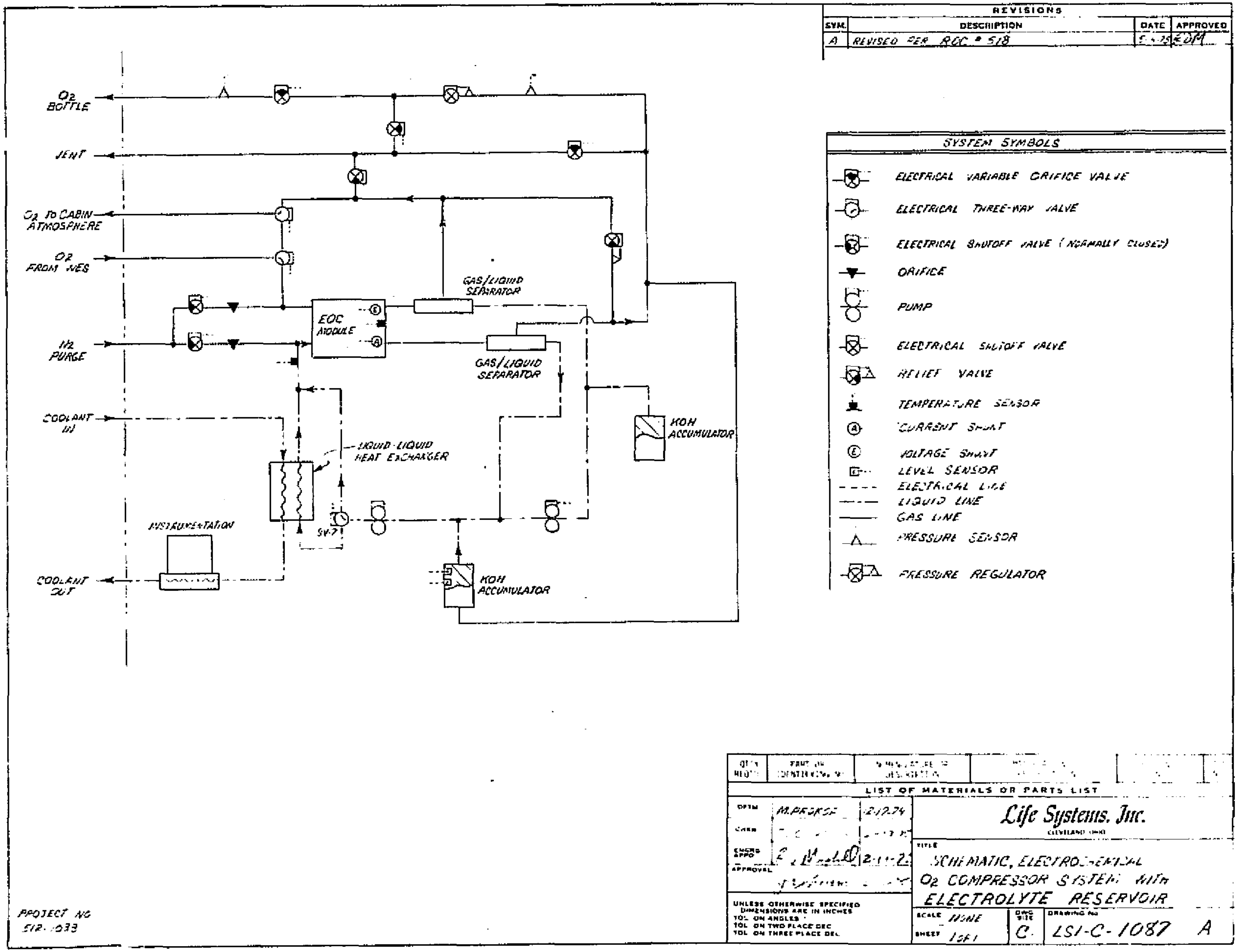


FIGURE 3 EOC SYSTEM SCHEMATIC WITH ELECTROLYTE RESERVOIR

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TABLE 4 EOC SYSTEM COMPONENT CHARACTERISTICS (WITH ELECTROLYTE RESERVOIR)

| Component | No. Req | Weight,[a] kg (Lb) | Volume,[b] $cm^3$ ($In^3$) | Power, W[c] |
|---|---|---|---|---|
| Module, EOC | 1 | 11.35 (25.0) | 3769.7 (230.0) | Variable |
| Valve, Electrical, Normally Closed | 3 | 0.36 (0.8) | 753.9 (46.0) | None |
| Valve, Electrical, Normally Open | 1 | 0.36 (0.8) | 753.9 (46.0) | None |
| Valve, Electrical, Three-Way | 3 | 0.36 (0.8) | 753.9 (46.0) | None |
| Valve, Electrical, Variable Orifice | 3 | 0.36 (0.8) | 753.9 (46.0) | None |
| Valve, Relief | 1 | 1.18 (2.6) | 3245.2 (198.0) | None |
| Regulator, Backpressure | 1 | 1.18 (2.6) | 3245.2 (198.0) | None |
| Restrictor, Flow | 2 | 0.09 (0.2) | 1.6 (0.1) | None |
| Pump, Liquid | 2 | 0.45 (1.0) | 1103.0 (67.3) | 12 |
| Heat Exchanger, Liquid-Liquid | 1 | 1.73 (3.8) | 819.5 (50.0) | None |
| Sensor, Temperature | 2 | 0.32 (0.7) | 662.8 (38.0) | 5 |
| Sensor, Pressure | 2 | 0.18 (0.4) | 278.6 (17.0) | 5 |
| Sensor, Level | 2 | 0.32 (0.7) | 41.0 (2.5) | 5 |
| Instrumentation | 1 | 4.54 (10.0) | 7457.5 (455.0) | 10 |
| Separator, Gas-Liquid, Zero-Gravity | 2 | 0.90 (2.0) | 163.9 (10.0) | None |
| Accumulator | 2 | 4.54 (10.0) | 999.8 (61.0) | None |

---

(a) Total weight, kg (Lb): 37.2 (82.0)
(b) Total component volume, $dm^3$ ($Ft^3$): 32.6 (1.2)
(c) Total component power, W: 64

of the number of EVA missions and resupply period. Table 5 shows the EOC module characteristics for an eight-hour and 12-hour $O_2$ bottle refill. For the comparison, the bottle refill period is averaged over the entire resupply period so that if there are only an average of two EVA missions per day, the refill period is 12 hours. Module power requirements and heat generation are therefore minimized and are a function of the number of EVA missions per resupply period.

The estimated spared system equivalent weights for the two systems as a function of EVA missions for a 90-day and a 180-day resupply period is shown in Figures 4 and 5, respectively. Figure 6 shows the total equivalent weight as a function of resupply period for two EVA missions per day. The assumptions used in the equivalent weight estimates are presented in Appendix 1. The projected and optimistic lines on the graphs refer to the EOC system designs with (Figure 3) and without (Figure 4) an electrolyte reservoir using the projected and optimistic module performance characteristics (Table 5), respectively. The projected and optimistic lines therefore form the envisioned EOC system equivalent weight range.

For the projected number of EVA missions per resupply period (Table 1) the EOC system equivalent weight, in general, would be greater than that for high pressure $O_2$ storage for resupply periods ≤90 and less than high pressure storage for resupply periods ≥180 days. The break-even point occurs at 105 days for the optimistic system and at 152 days for the projected system for two EVA missions per day.

## EOC CELL DESIGN

The disassembled and assembled EOC test cell is shown in Figures 7 and 8, respectively. The EOC was designed for operation at pressures up to 41,400 $kN/m^2$ (6000 psia). The circular design concept was chosen because of its high strength, low deflection, low cost and low weight characteristics compared to a rectangular cell design. The structure of the cell consists of anode and cathode frames positioned together by three insulating locating pins. The cell housing, which is female threaded, is fitted over the cathode and anode frames. The housing is screwed onto the male threaded anode frame, to provide the compression necessary for sealing, and the strength and rigidity required for high pressure operation.

### Cell Configuration

The EOC test cell schematic is shown in Figures 9 and 10. Component characteristics are given in Table 6 which is keyed to Figures 9 and 10.

#### Gas Manifolding

Low pressure $O_2$ is manifolded into the cell through the cathode frame to the cathode manifold disc (see Figure 10). The gas flow is directed to the center of the disc by a slot cut in the back of the manifold disc. An O-ring prevents gas from passing between the cathode frame and manifold disc and out the exhaust

## TABLE 5 EOC MODULE CHARACTERISTICS

| | | |
|---|---|---|
| Number of Modules | 1 | |
| Weight, kg (Lb) | 11.35 (25.0) | |
| Volume, $dm^3$ ($In^3$) | 3.8 (230) | |
| Basic Dimensions, cm (In) | 19.8 dia x 12.2<br>(7.8 dia x 4.8) | |
| Number of Cells | 10 | |
| Active Cell Area, $cm^2$ ($Ft^2$) | 232 (0.25) | |
| Recharge time, h | 8 | 12 |
| Cell Current, A | 250 | 168 |
| Current Density, $mA/cm^2$ (ASF) | 108 (100) | 72 (67) |
| Current Efficiency, % | 98.7 | 97.8 |
| Cell Voltage, V | | |
| Optimum | 0.4 | 0.4 |
| Projected | 1.0 | 1.0 |
| Power Required, W | | |
| Optimum | 100 | 67 |
| Projected | 250 | 168 |
| Heat Rejected, J/s (Btu/Hr) | | |
| Optimum | 100 (342) | 67 (229) |
| Projected | 250 (855) | 168 (575) |
| $O_2$ Compression Rate, kg/d (Lb/Day) | 1.77 (3.9) | 1.18 (2.6) |

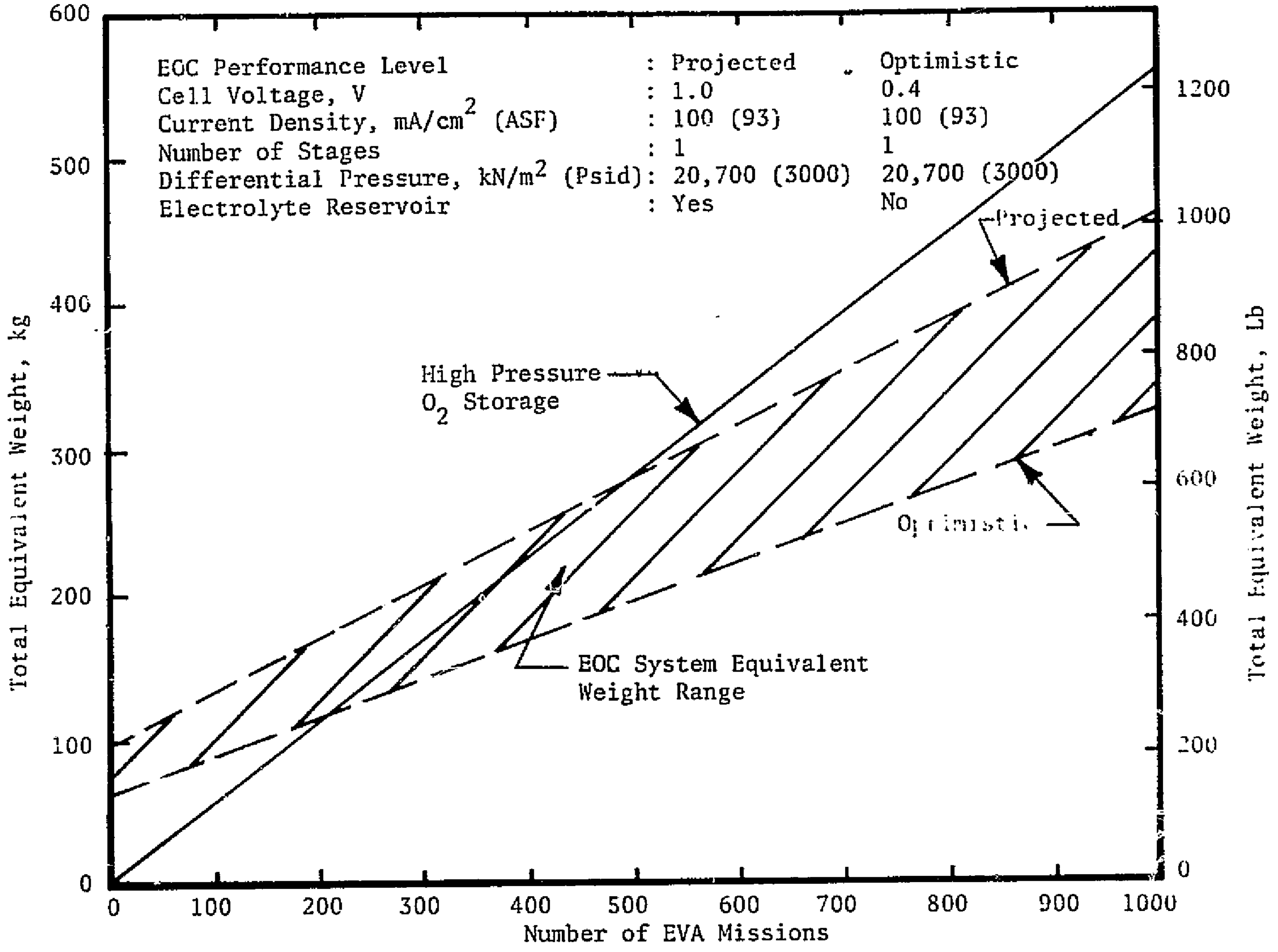


FIGURE 4 TOTAL EQUIVALENT WEIGHT VERSUS EVA MISSIONS (90-DAY RESUPPLY PERIOD)

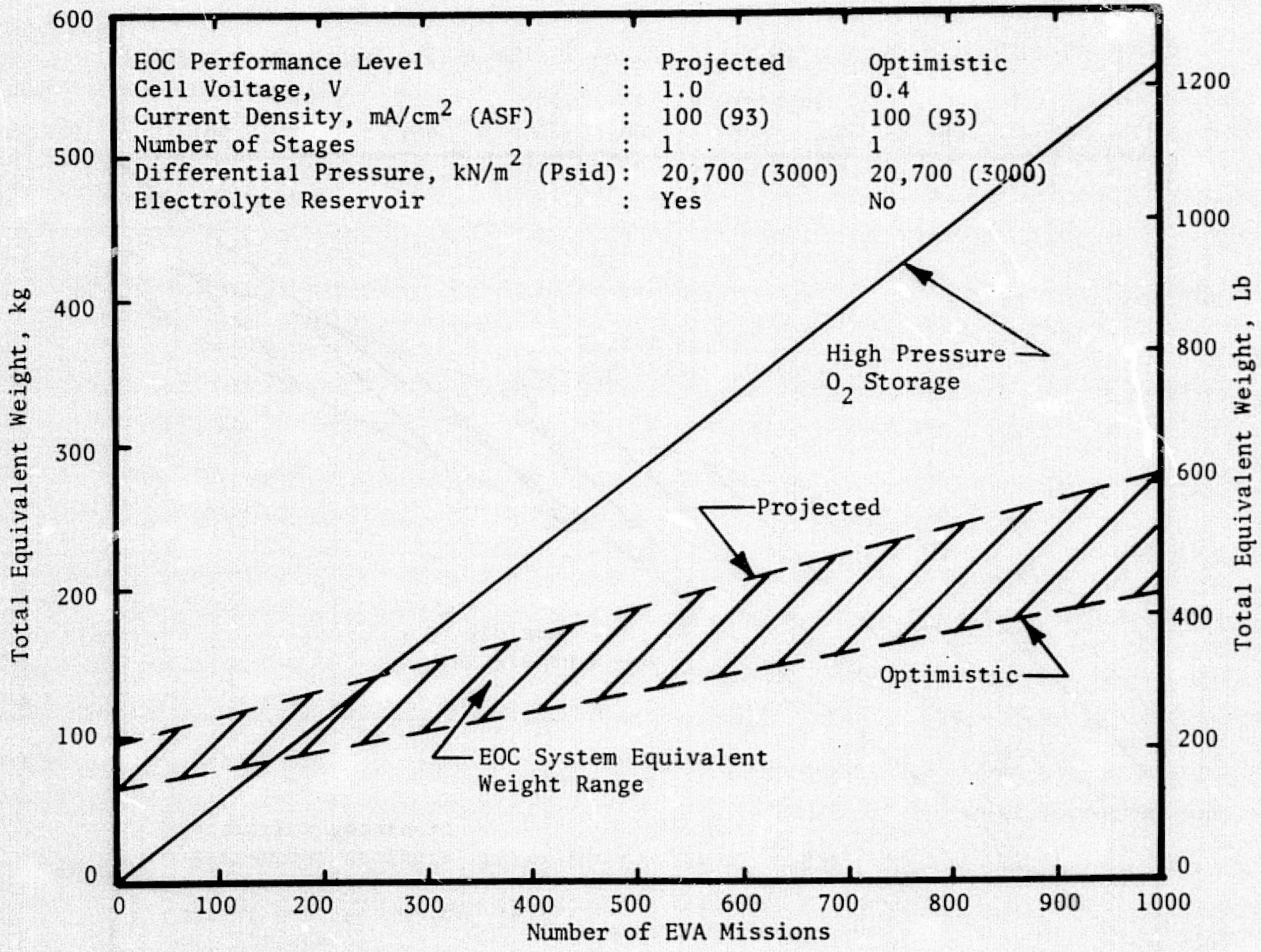


FIGURE 5 TOTAL EQUIVALENT WEIGHT VERSUS EVA MISSIONS (180-DAY RESUPPLY PERIOD)

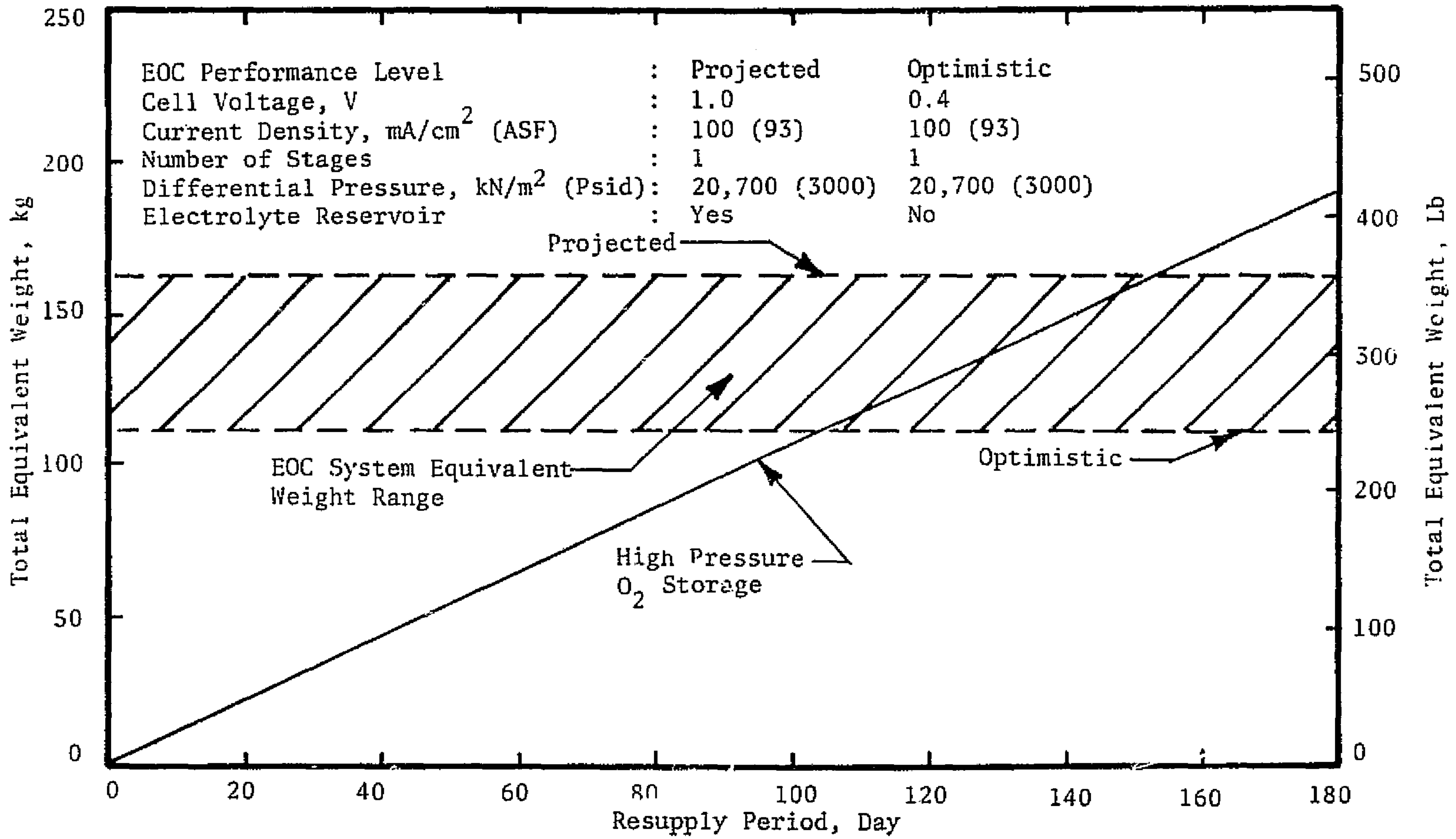


FIGURE 6 TOTAL EQUIVALENT WEIGHT VERSUS RESUPPLY PERIOD (TWO EVA MISSIONS PER DAY)

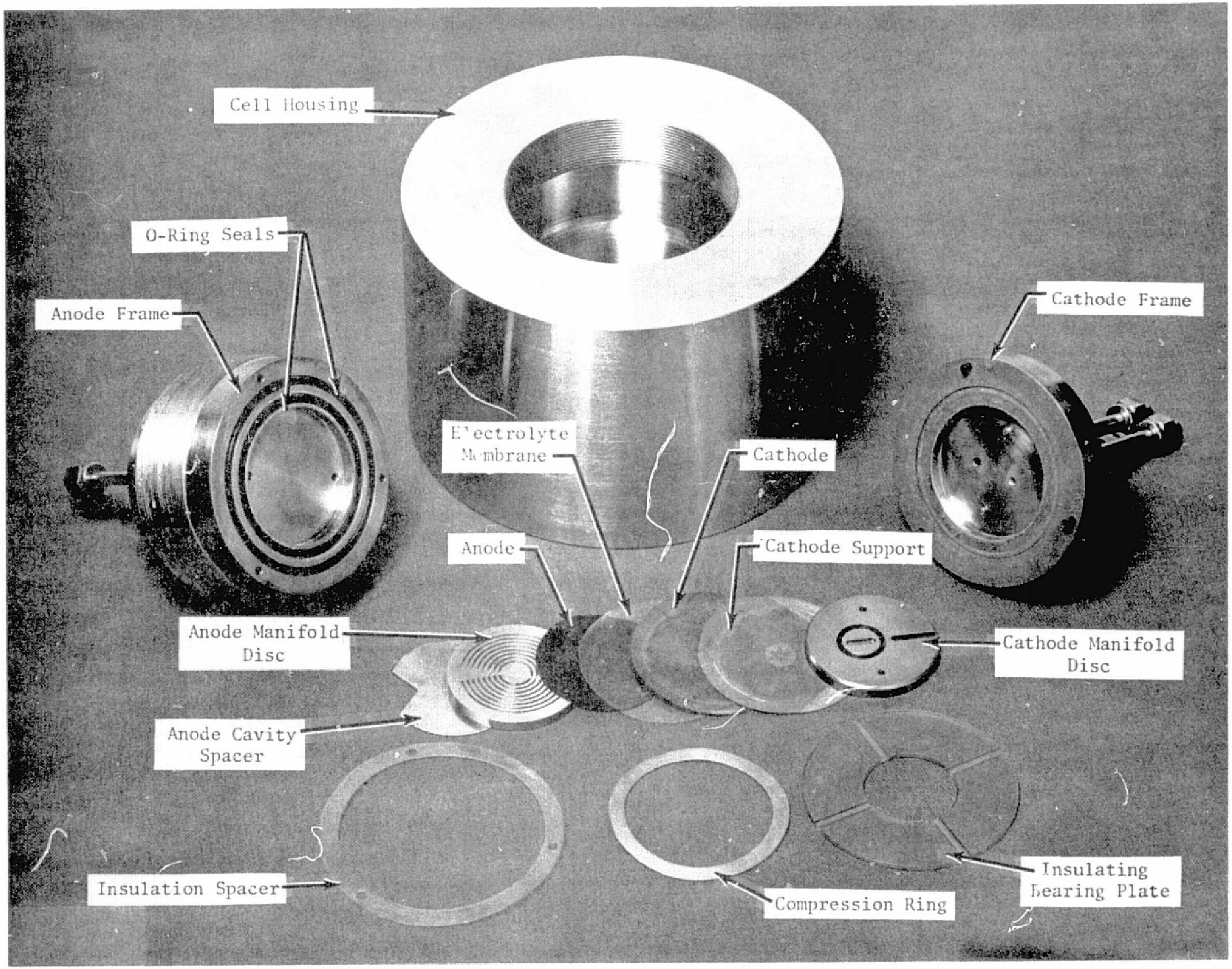


FIGURE 7 EOC CELL DISASSEMBLED

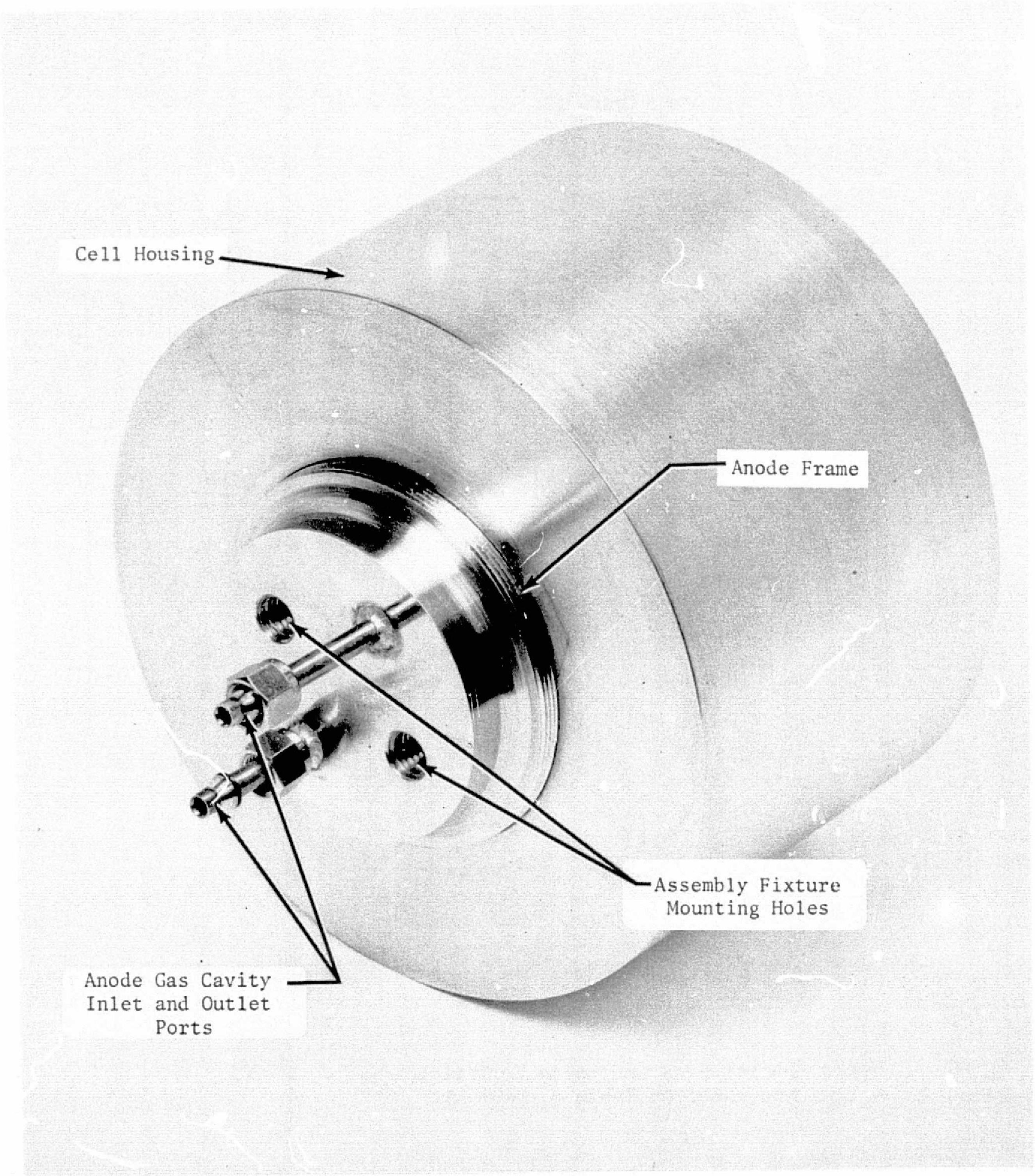
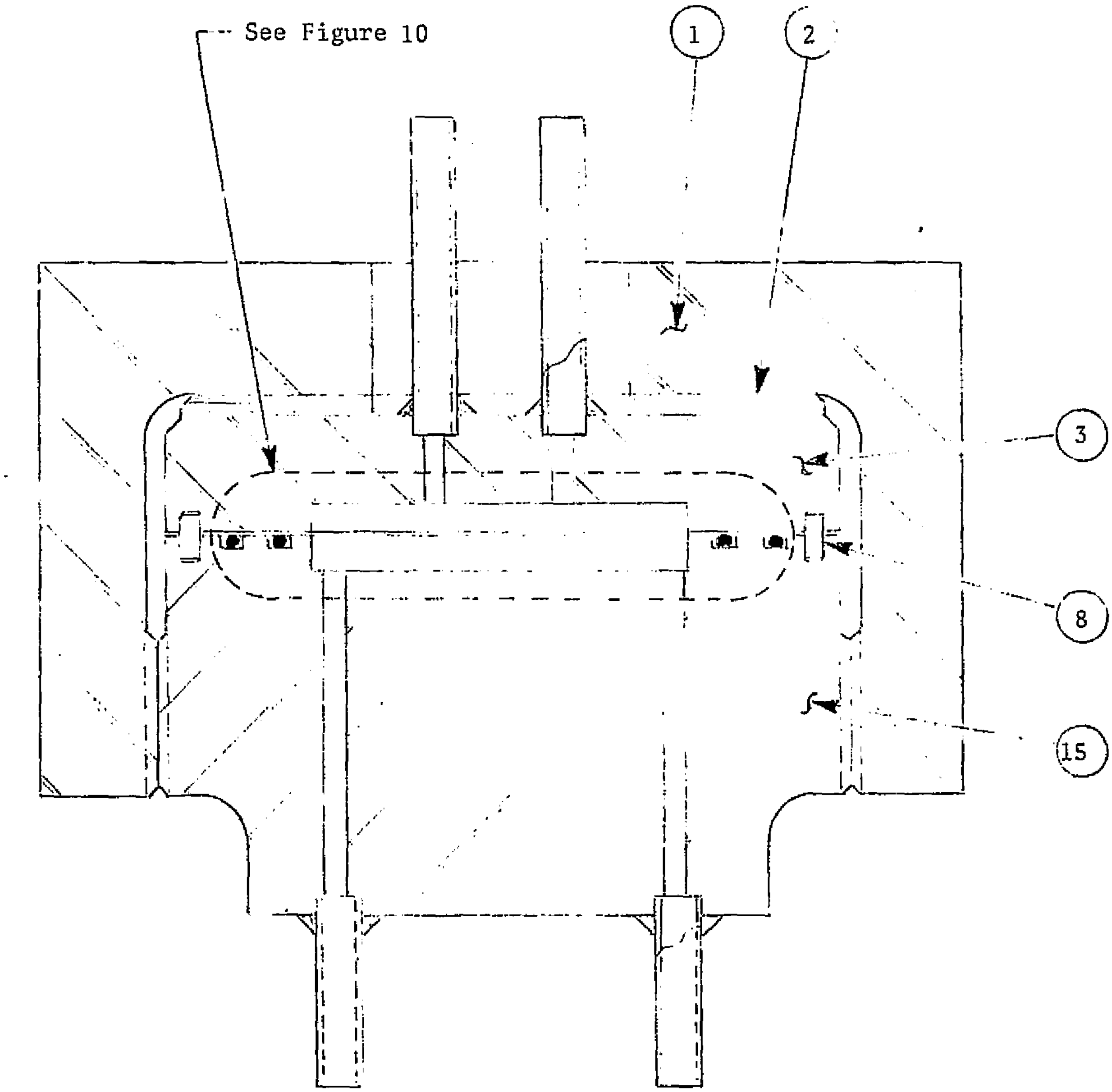
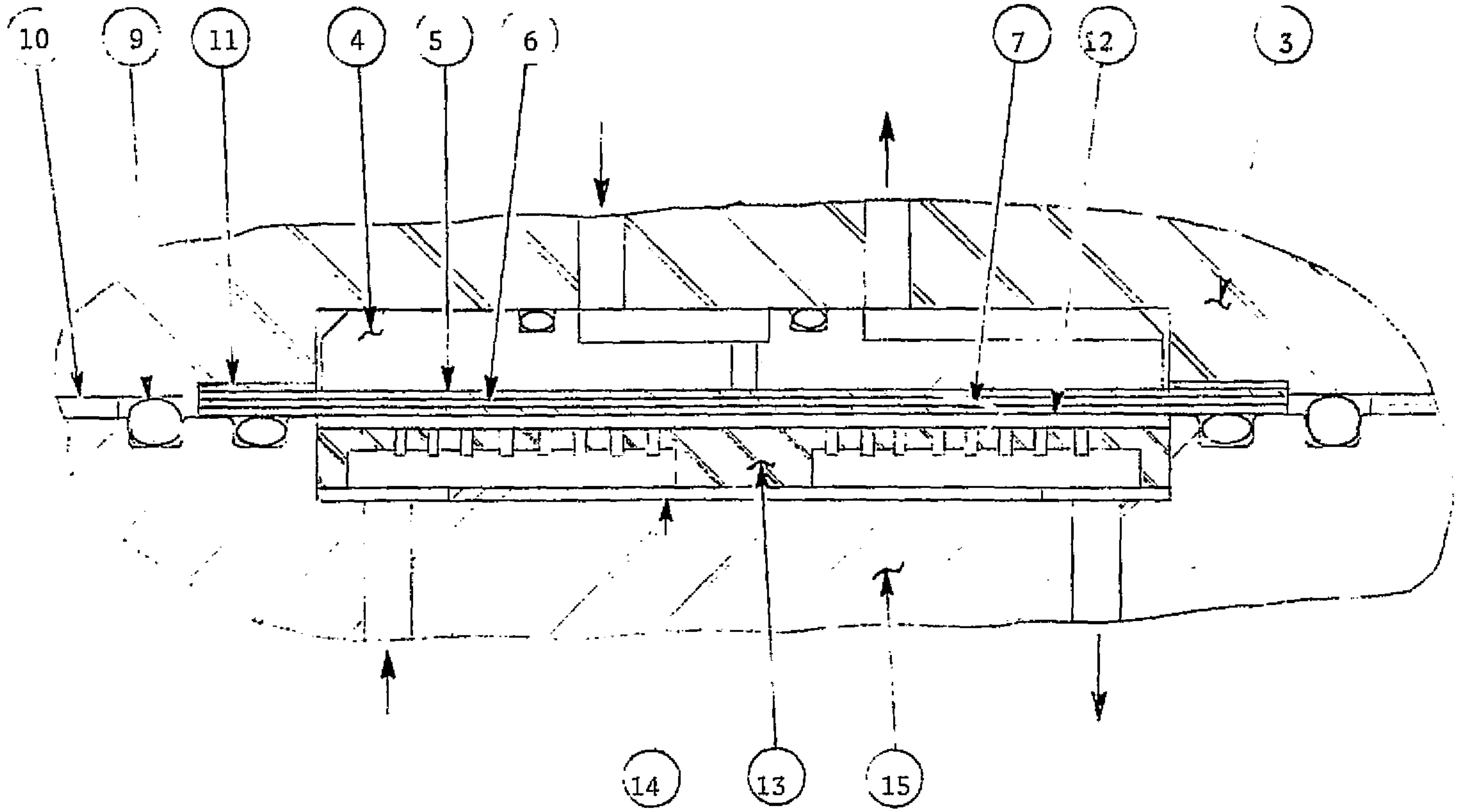


FIGURE 8 ASSEMBLED EOC CELL



Note: Definitions and Characteristics are found on Table 6, page 26

FIGURE 9 EOC CELL SCHEMATIC



Note: Definitions and Characteristics are found on Table 6, page 26

FIGURE 10 EOC CELL STACKUP SCHEMATIC

## TABLE 6 EOC CELL COMPONENTS

| Item No. | Description | |
|---|---|---|
| 1 | Cell Housing | Stainless Steel |
| 2 | Insulating Bearing Plate | Polysulfone |
| 3 | Cathode Frame | Inconel |
| 4 | Cathode Manifold Disc | Inconel |
| 5 | Cathode Support | Porous Nickel |
| 6 | Cathode | LSI Proprietary |
| 7 | Electrolyte Membrane | Fluorinated Hydrocarbon Base |
| 8 | Locating Pins | Polysulfone |
| 9 | O-Ring Seals | Viton |
| 10 | Insulation Spacer | Polysulfone |
| 11 | Compression Ring | Teflon-Coated Nickel |
| 12 | Anode | LSI Proprietary |
| 13 | Anode Manifold Disc | Inconel |
| 14 | Anode Cavity Spacer | Nickel |
| 15 | Anode Frame | Inconel |

port. Oxygen passes through the central hole in the manifold disc and through the porous nickel (Ni) gas cavity support spacer to the electrode surface. The gas passes radially over the electrode to the periphery of the gas cavity. Oxygen not consumed passes around the edge of the manifold disc and exhausts the cell through the cathode frame. The evolved $O_2$ at the anode is collected in the eight circular channels that are machined into the anode manifold disc. The eight circular channels are connected together by two radial channels on the back of the anode manifold disc. Gas collected in the radial channels is carried out of the cell at high pressure through either of the tubes in the anode frame.

## Sealing

The cathode and anode frames each contain a circular recess into which the gas manifold discs and electrodes are inserted. The membrane separates the high pressure $O_2$ in the anode cavity from the low pressure $O_2$ in the cathode cavity and is pressed against the cathode. Membrane sealing is accomplished by an O-ring in the anode frame. When the cell is operating at pressure, this O-ring is pressed outwards and against the membrane by the high pressure $O_2$ in the anode cavity. A second outer O-ring seals the anode frame to the cathode frame and isolates the low pressure $O_2$ of the cathode cavity from the ambient pressure air within the cell housing. The O-ring sealing concept was chosen because, unlike gaskets, the sealing characteristics of O-rings improve with increasing pressure and the O-ring can accommodate some deflection as cell pressure increases between the two sealing members.

## Electrical Insulation

The cathode, cathode manifold disc and the cathode frame are in electrical contact. The two tubes welded to the cathode frame serve as the electrical connections for the cathode. The anode, anode manifold plate, anode frame and cell housing are in electrical contact. The anode frame serves as the electrical connection to the anode. The electrodes are not insulated from the structural members of the cell, so that the sealing and current leakage problems associated with current taps are eliminated. The cell housing, anode frame and high pressure tubing are all electrically grounded. The cathode frame is electrically insulated from the cell housing by a polysulfone bearing plate. The bearing plate is lubricated and rotates with the cell housing as it is secured onto the anode frame. The cathode frame is electrically insulated from the anode frame by a polysulfone spacer and polysulfone locating pins which are located outside the O-rings. In addition, the face of the cathode frame outside the cathode cavity is teflon-coated to eliminate current leakage. The tubing welded to the cathode frame was insulated from the tubing of the test stand which is grounded. Since the cathode is the low pressure side of the cell, teflon fittings were used for this purpose.

## Design Flexibility

The cell design can accommodate testing of membranes and electrodes of various thicknesses. Spacers between the cathode frame and cathode manifold plate and between the membrane and cathode frame in the O-ring region are used to achieve this flexibility.

Safety Aspects

In the unlikely event of extreme overpressure in the cell, loss of sealing at the O-rings would not cause a buildup of pressure within the cell housing. Radial channels in the polysulfone bearing plate would release the pressure. The cell includes a designed weak area, the equivalent of a rupture disc. This is the central portion of the cathode frame not supported by the polysulfone bearing plate and cell housing.

### Cell Design Features

The following design features were incorporated into the EOC test cell:

1. The cell is capable of operating at pressures up to 41,400 kN/$m^2$ (6000 psia).
2. The cell has an internal anode-to-cathode cavity differential pressure capability of up to 41,400 kN/$m^2$ (6000 psid) and depends only on the differential pressure tolerance of the membrane used.
3. The materials of construction for the EOC test cell are completely compatible with high pressure $O_2$ and KOH electrolyte.
4. The cell manifolding design allows for flow through or dead-ended operation.
5. Electrode thicknesses up to 0.27 cm (0.050 in) can be used in the cell.
6. Membrane thicknesses up to 0.076 cm (0.030 in) can be used in the cell.
7. The cell utilizes a simple and effective O-ring sealing technique.
8. Cell electrical leads connect externally to the cell structural parts and therefore do not require gas or electrolyte seals.
9. The cell automatically relieves any pressure buildup should an O-ring seal or membrane fail.

## GROUND SUPPORT TEST FACILITIES

A ground support test facility was developed for single cell EOC feasibility testing. The test facility continuously feeds $O_2$ at the required pressure and dew point to the EOC single cell. The $O_2$ evolved on the high pressure side is regulated to the desired pressure using a backpressure regulator to simulate the compression process prior to venting the system. The mechanical and electronic components are contained in separate enclosures. The mechanical portion of the test facility provides the necessary pressure regulation, valving and tubing for process $O_2$, purge argon (Ar) and the $N_2$ used to operate the pneumatic valves. The electronic enclosure contains the control and monitoring instrumentation and can be located at distances of up to 20 feet from the mechanical components. The two-package test facility design was incorporated for remote operation during testing at high $O_2$ pressures as an added safety feature. A photograph showing the test facility is presented in Figure 11.

## Mechanical Design

The ground support test facility was initially designed for EOC cell operation without an electrolyte reservoir. The test facility was later modified to allow testing with a cell electrolyte reservoir. Both designs are discussed below.

### Initial Design

The EOC test facility instrumentation and flow schematic is shown in Figure 12. The assembled mechanical components are shown in Figure 13. Bottled $O_2$ is fed through a solenoid valve (SV1) to the $O_2$ feed pressure regulator (PR1) which controls the $O_2$ feed pressure and flow rate through the cathode compartment of the EOC cell. Prior to entering the cell, the $O_2$ is humidified by bubbling through water. The dew point of the $O_2$ stream is controlled by adjusting the humidifier water temperature. Water from a controlled temperature bath is circulated through copper tubing attached to the humidifier housing to give the desired humidifier water temperature. The humidified $O_2$ then enters the cathode compartment of the EOC cell where $O_2$ is electrochemically consumed at the cathode and regenerated at the anode. The pressure of the $O_2$ evolved into the anode compartment is controlled by a backpressure regulator (PR3). The $O_2$ flow rate venting the system is measured downstream of PR3 by a soap bubble flow meter and is used to calculate cell current efficiency.

Argon is used as the purge gas to remove $O_2$ from the system following a shutdown. Argon was chosen because high purity Ar was commercially available at a lower cost than the same purity $N_2$. Following a shutdown, the system is slowly depressurized by opening pneumatic valves PV3 and PV4 which allow $O_2$ to vent through orifices FR1 and FR2. Pneumatic valves PV1 and PV2 then open allowing Ar to purge the system. Following the purge all pneumatic valves close,thereby sealing the system with a positive Ar pressure.

Pneumatically operated, nonrotating ball valves were used since normal, electrically actuated solenoid valves constitute an ignition source when used in high pressure $O_2$ lines. The actuation of the pneumatic valves is controlled by solenoid valves SV2, SV3, SV4, and SV5 which allows $N_2$ to pressurize and open the valves, or vent and close the valves.

Cell temperature is maintained by circulating water from a controlled temperature bath through tubing attached to the cell housing. This cell temperature control

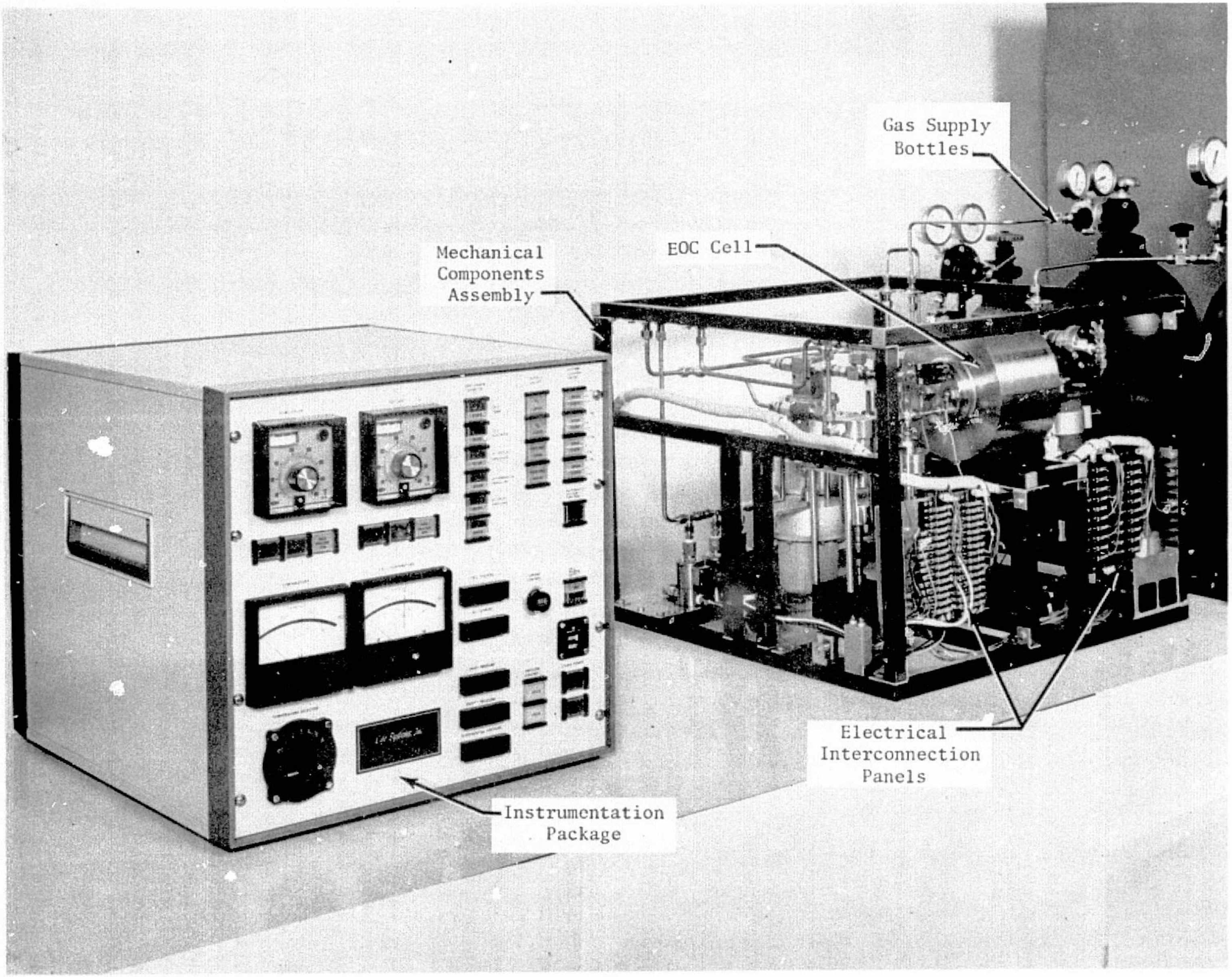


FIGURE 11 EOC GROUND SUPPORT TEST FACILITY

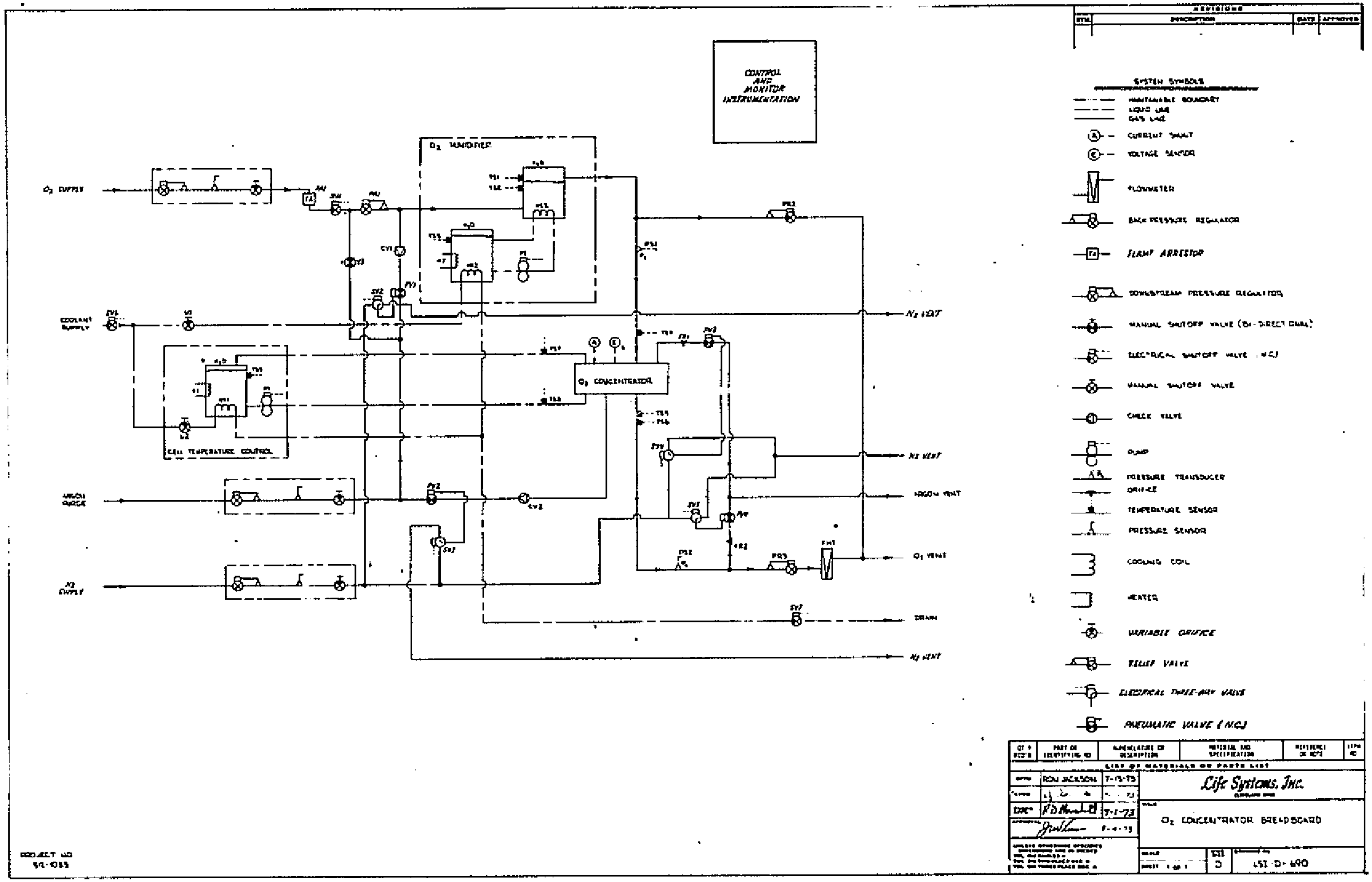


FIGURE 12 EOC TEST FACILITY SCHEMATIC

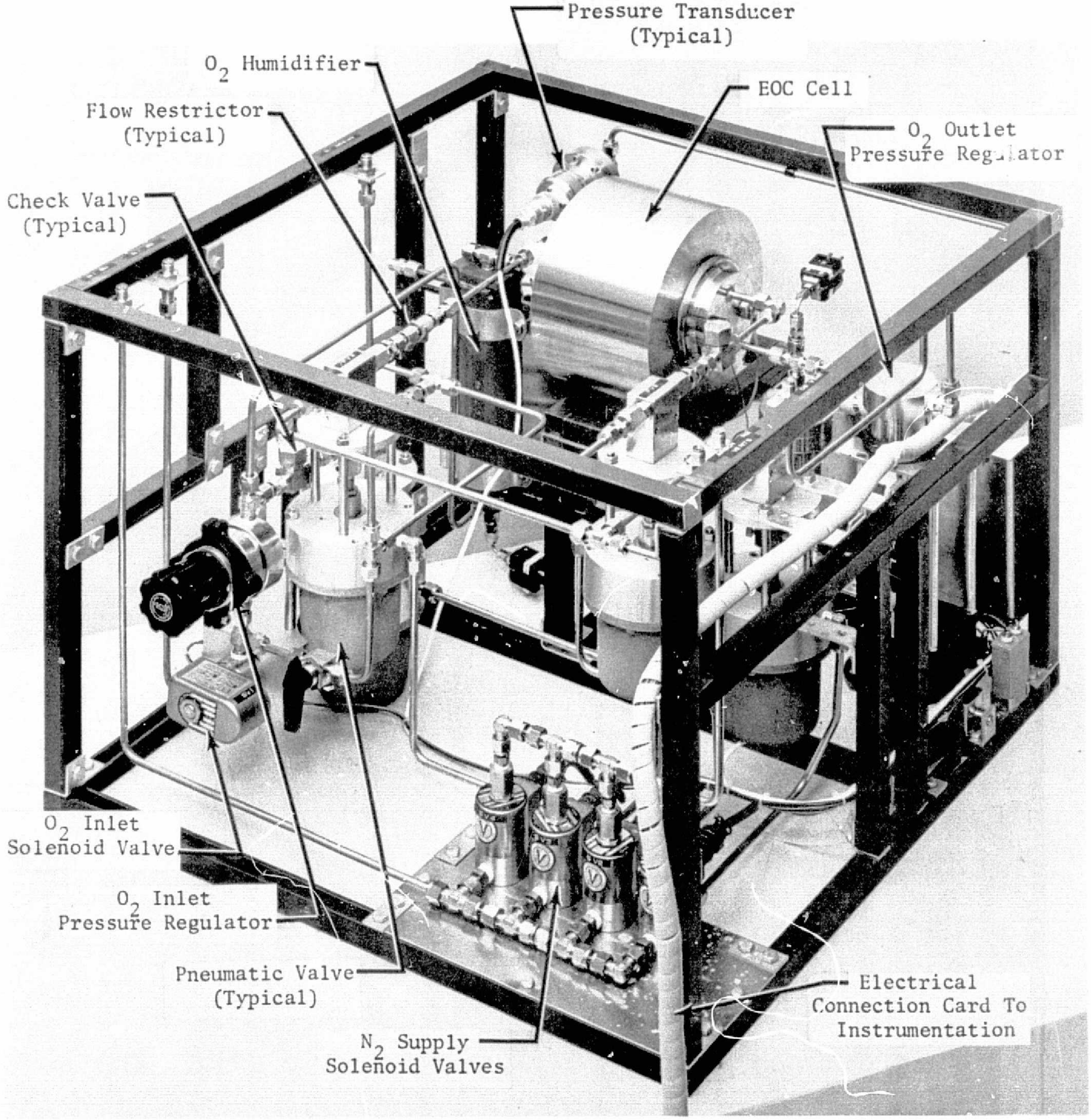


FIGURE 13 EOC MECHANICAL COMPONENTS ASSEMBLY

loop is used to operate the cell at temperatures above or below ambient room temperature. Temperature control is not required when operating at room temperature since the cell housing acts as a heat sink to dissipate the heat generated by cell inefficiencies.

### Modified Design

The modified test facility schematic is presented in Figure 14. The only differences between the modified and initial designs are the addition of an electrolyte reservoir tank to the anode side of the EOC cell and the electrolyte trap on the cathode $O_2$ outlet stream.

During operation the electrolyte reservoir keeps the cell anode cavity flooded. Oxygen generated at the anode exhausts at the top of the reservoir tank, with the tank acting as a 1 g gas/liquid separator. Nitrogen is fed into the electrolyte loop upstream of the reservoir tank to keep electrolyte circulating through the anode cavity. At low pressures, 3450 kN/$m^2$ (500 psia), the volumetric $O_2$ flow keeps the electrolyte circulating and the $N_2$ flow is not required. At higher pressures, the actual $O_2$ volumetric flow rate is very low and the $O_2$ forms a foam with the electrolyte. The addition of $N_2$ prevents the foam from blocking the tubes and keeps electrolyte circulating.

At the cathode, electrolyte that permeates into the cathode cavity is removed from the exiting $O_2$ stream and is collected in the trap for analysis.

## Instrumentation

A photograph of the instrumentation enclosure is given in Figure 15. Instrumentation was provided to:

1. Control cell temperature, humidifier temperature, and cell current.
2. Monitor cell voltage and current, system pressures and temperature.
3. Provide automatic, fail-safe shutdown should critical parameters exceed preset levels.

The electrical power supplies required to operate the instrumentation unit are a 115V, 16A, 60 Hz alternating current supply for the control and monitoring instrumentation and an uninterruptable 28V, 6A direct current supply to run the shutdown sequencing circuits. Each power supply to the instrumentation unit is protected by a circuit breaker located on the back panel and its condition is indicated by a green light on the front panel.

### Control Features

Cell current is controlled using a constant current feedback control circuit. Alternating current line power is transformed, rectified, filtered and fed into this control circuit. Cell current is set by an adjustable, 0 to 10A, digital potentiometer located on the front panel and maintained by the current controller.

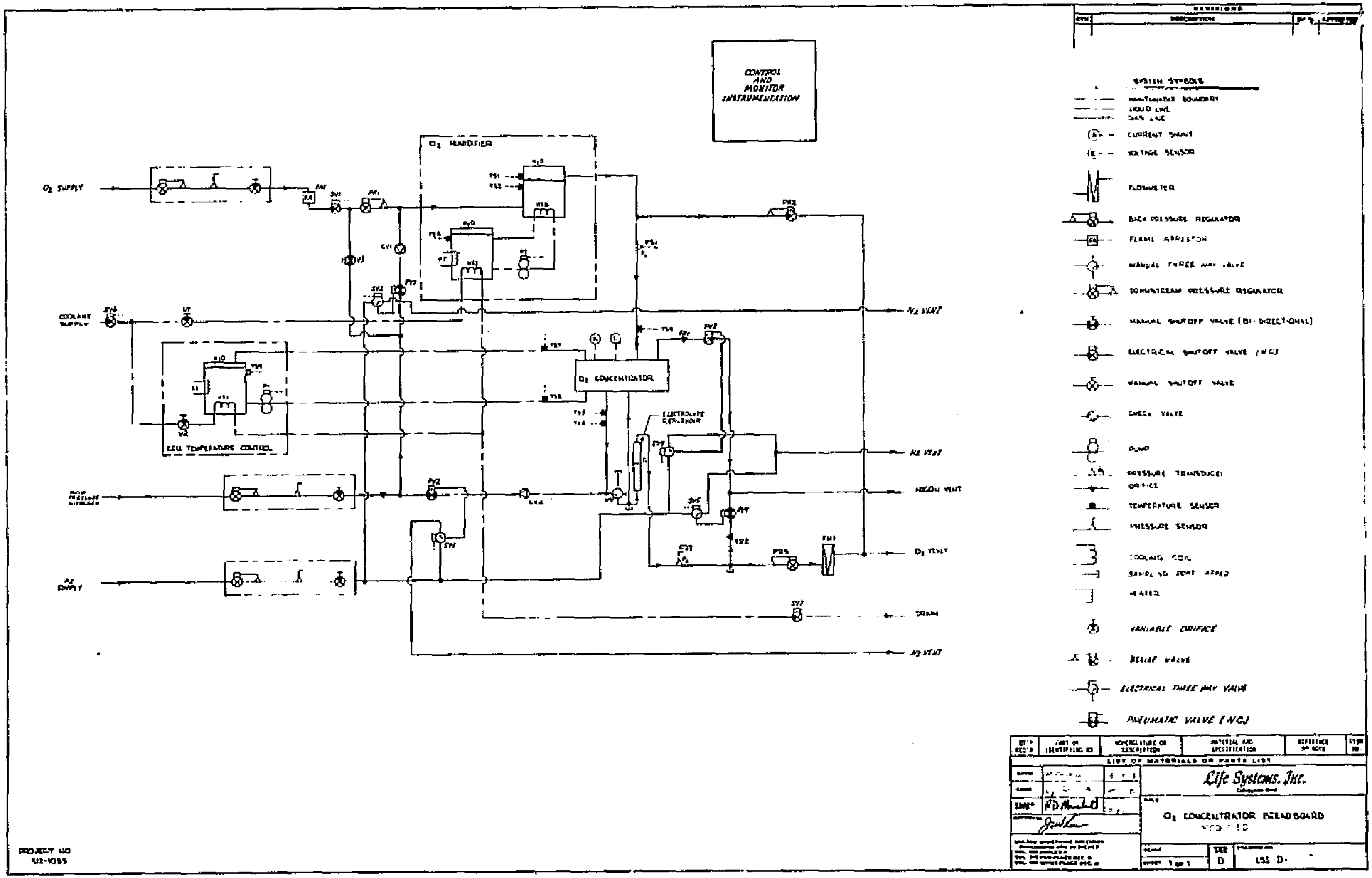


FIGURE 14 MODIFIED EOC TEST FACILITY SCHEMATIC

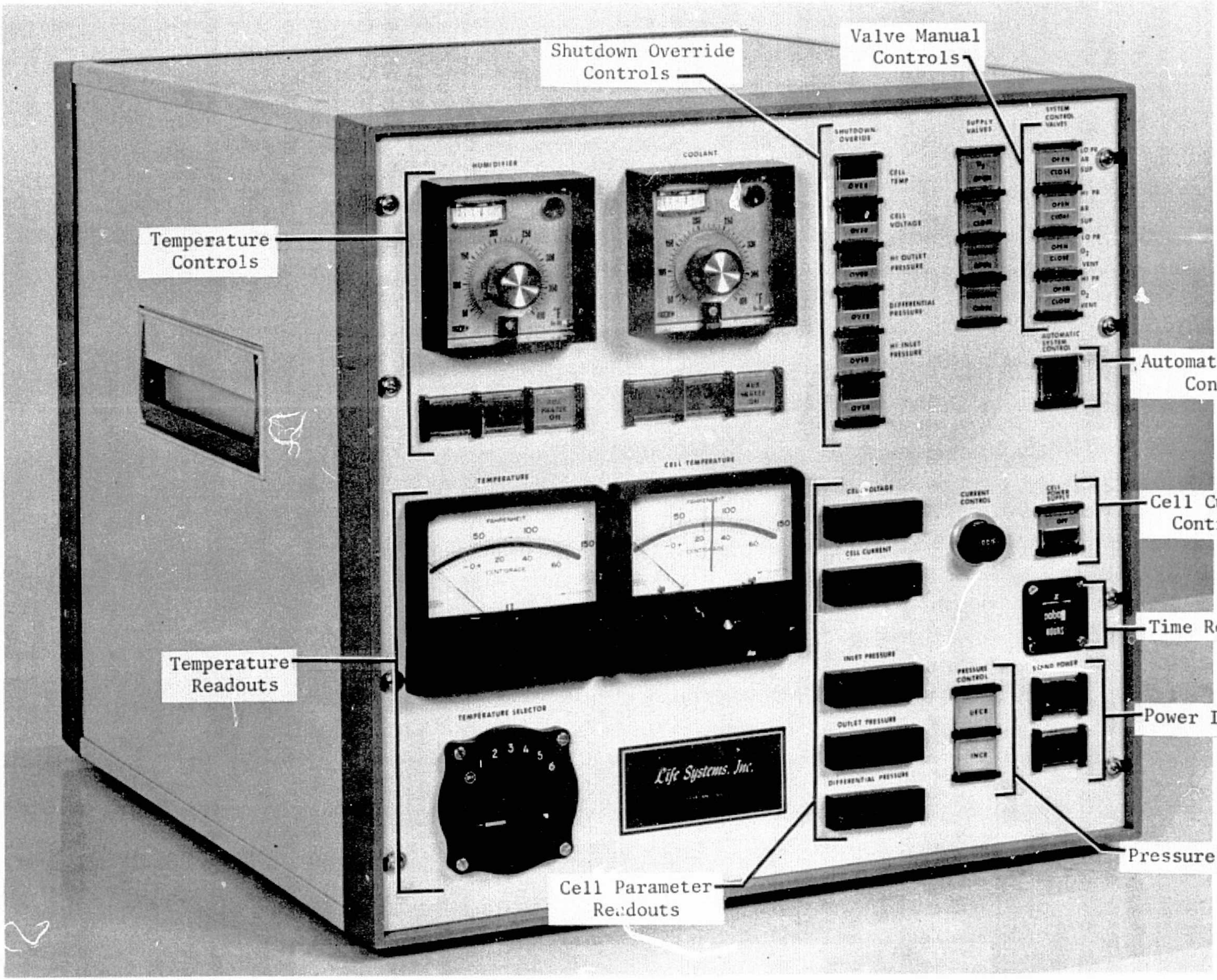


FIGURE 15 TEST FACILITY INSTRUMENTATION PACKAGE

Cell temperature is controlled by measuring and comparing the $O_2$ outlet temperature (TS5) to the setting on the coolant temperature controller. The temperature controller then regulates ON-OFF power to a band heater located on the liquid reservoir tank to maintain cell temperature at the controller set point. The temperature of the humidifier water is controlled by measuring and comparing the actual humidifier water temperature (TS2) to the setting on the humidifier temperature controller. The temperature of the humidifier coolant reservoir is again regulated by supplying power to the band heater located on the reservoir tank.

## Monitoring Features

Cell voltage, cell current, $O_2$ inlet pressure, $O_2$ outlet pressure and cell differential pressure are monitored and displayed in engineering units on digital panel meters. Cell voltage is measured by attaching voltage leads to the cell housing and cathode tubing. Cell current is measured by a shunt connected in series with the cell current leads. Absolute pressure is measured using pressure transducers (PS1 and PS2) located in the appropriate gas lines. Differential pressure is then obtained by electrically subtracting the signals from the absolute pressure transducers.

Humidifier water, humidifier coolant reservoir, $O_2$ feed, $O_2$ exhaust, cell coolant in, cell coolant out, and cell coolant reservoir temperatures are monitored using thermocouples TS1, TS3, TS4, TS6, TS8, TS7, and TS9, respectively. A six-position temperature selector switch located on the front panel selects the desired temperature which is then displayed on the pyrometer. Cell temperature is displayed on a separate meter which is also used to set the cell temperature shutdown level.

## Shutdown Features

Test facility operation is automatically terminated when cell voltage, $O_2$ feed pressure, $O_2$ outlet pressure, cell differential pressure or cell temperature exceed a preset level. Immediately after a shutdown signal, cell current drops to zero and the $O_2$ feed gas supply is turned off by closing SV1. The system is allowed to depressurize by energizing SV4 and SV5 which cause PV3 and PV4 to open. The depressurization takes approximately 120 seconds after which SV2 and SV3 are energized to open PV1 and PV2 and admit purge Ar. The purge continues for another 120 seconds. All solenoid valves are then de-energized to close the pneumatic valves and seal the system.

The power required for the shutdown sequencing is supplied by the uninterruptable power supply. Fail-safe automatic shutdown is therefore provided even during a total lab power failure.

## PRODUCT ASSURANCE

The Product Assurance activities carried out during the design, fabrication and testing of the EOC included Quality Control, Safety and Materials Control. The

Product Assurance Program was established based on standard Life Systems' procedures and information obtained from literature.(4-11)

## Quality Control

The Quality Control activities conducted during the fabrication and assembly of the EOC included performance or receiving inspections on all components purchased to insure that drawing requirements and specifications regarding configuration, materials of construction and cleanliness for $O_2$ service were complied with. In addition, Quality Control established the $O_2$ cleaning specification (MSFC Spec-164A) and participated in selecting a vendor for performing the cleaning operation.

## Safety

Due to the hazardous nature of high pressure $O_2$ gas a major effort was made during the early stages of the EOC program to define safety procedures and guidelines which would insure personnel safety. These activities included performing a literature survey on high pressure $O_2$ safety and materials compatibility. From this information a document detailing test facility and operational safety guidelines for the system was prepared and is included as Appendix 2 to this report. A procedure for cleaning components for $O_2$ service was also prepared and a component cleaning and assembly area was set up and utilized. A pre-experiment safety inspection of the EOC and test facility was performed. Additional safety inspections were conducted after each maintenance activity performed on the EOC system.

A list of the major safety features that have been incorporated into the EOC system follows.

1. System overpressure protection was designed into the EOC. Pressure transducer PS2 will trigger a system shutdown when pressure exceeds a preset level. (For the testing conducted under the present program, the level was set at 11,040 $kN/m^2$ (1600 psia)).

2. The cell was designed with provisions for venting the high pressure $O_2$ through a 1-inch diameter passage in the event the cell is exposed to excessive pressure.

3. A pressure relief regulator, PR2, was incorporated into the system to vent $O_2$ in the event of unexpected pressure increases such as a cell membrane rupture.

4. All components that are exposed to high pressure $O_2$ were rated for $O_2$ service in excess of the system operating pressure.

5. The electrochemical cell housing has been designed to withstand 103,500 $kN/m^2$ (15,000 psia) internal pressure. This represents a 2.5 safety factor above the maximum end-item application pressure of 41,400 $kN/m^2$ (6000 psia).

6. A cell high temperature shutdown is included in the system and occurs when cell temperature exceeds a preset level. (For the testing conducted under the present program, the level was set at 311K (100F)).

7. The system has a high pressure shutdown on the inlet $O_2$ side (PS1) that will trigger a shutdown at pressures in excess of 2760 $kN/m^2$ (400 psia). This also protects against membrane rupture as this sensor would see a pressure spike and initiate a shutdown.

8. The materials of construction that are incorporated into the system have been proven safe for utilization in high pressure $O_2$ systems, i.e., 316 stainless steel, Inconel, Monel, Teflon, Kel F, Viton.

9. An automatic Ar purge which occurs after any system shutdown was incorporated into the test facility.

10. The system has a ΔP shutdown which occurs when PS2-PS1 exceeds a preset level. (For the testing conducted under the present program, the level was set at 11,040 $kN/m^2$ (1600 psid)).

11. The system has a high cell voltage shutdown to protect the electrochemical cell when cell voltage exceeds 2.0V.

12. The system was designed so that pressure checks and flow setup can be accomplished with Ar.

13. The system was designed such that the control instrumentation is remote from the mechanical components assembly.

14. All components in the EOC system, including tubing and fittings, were cleaned for $O_2$ service to the requirements of MSFC Spec-164A.

15. The shutoff valves in the high pressure $O_2$ lines have nonrotating stems. These have a minimum closure force and produce minimum friction thereby reducing the possibility of ignition when operating in the high pressure $O_2$.

16. The system was designed and constructed such that the volume of high pressure $O_2$ is minimized. The system contains only 25 $cm^3$ of high pressure $O_2$.

17. The Test Facility and Operational Safety Guidelines, Appendix 2, were adhered to in the performance of the program. These include:

    a. Cleanliness provisions for assembling the system
    b. $O_2$ cylinder handling techniques
    c. Precautions taken in the plumbing of the system and valve and regulator operation

### Materials Control

A Material Control Program was carried out in conjunction with the design and fabrication of the EOC. Because of the severe combustion hazard of materials in high pressure $O_2$, it was imperative that strict controls be imposed on the selection of materials. This activity included control of both metallic and nonmetallic materials. The materials of construction of all components were identified and screened for acceptability in high pressure $O_2$ systems.

A Material Data Summary (Table 7) was prepared for each component in the system. This table was prepared by the Design Engineer and was verified and disposition assigned by the Safety Engineer. Unacceptable material was identified and replaced. Table 7 is the final iteration of the Material Data Summary and describes the materials present in each component in the EOC.

## MEMBRANE DEVELOPMENT PROGRAM

The identification, development, and selection of electrolyte membranes for use in an EOC were carried out in three steps:

1. Definition of membrane specifications
2. Review of vendor literature and selection of a commercially available membrane for the application
3. Development of a customized membrane using standard membrane fabrication techniques to obtain the desired membrane properties

### Membrane Specifications

The selection of an appropriate membrane material for EOC application is governed by six criteria:

1. Compatibility with high pressure $O_2$ and KOH electrolyte
2. The ability to provide an $O_2$ and electrolyte-tight seal at the periphery of the active electrode area
3. High differential pressure stability ($\geq$20,700 kN/$m^2$ (3000 psid)) when well supported
4. Low $O_2$ permeability for high cell current efficiency
5. Low electrical resistance ($\leq$1.0 ohm-$cm^2$ (6.5 ohm-$in^2$))
6. Low water permeability ($\leq 1.4 \times 10^{-6}$ g/hr-$cm^2$-kN/$m^2$ ($1 \times 10^{-5}$ g/hr-$cm^2$-psid)) for steady-state operation without water accumulation at either electrode

## TABLE 7 MATERIAL DATA SUMMARY

| | | | | |
|---|---|---|---|---|
| **Life Systems, Inc.**<br>CLEVELAND, OHIO 44122 | MATERIAL DATA SUMMARY | ☒ NONMETALLIC<br>☒ METALLIC | NO. 512-1033 | REVISION LTR. A |
| | TITLE ELECTROCHEMICAL OXYGEN COMPRESSOR TEST FACILITY | DRAWING NO. LSI-D-690, LSI-D-1138 | PAGE 1 OF 3 | DATE 5/75 |

| Part Description | Manufacturer And Mfgr's Designation | Material Description | Use | Max. Pressure kN/m$^2$ (Psig) | Max. Oper. Pressure kN/m$^2$ (Psig) | Environment | Dwg. Ref. |
|---|---|---|---|---|---|---|---|
| Valve, Pneumatic | Sno-Trik Company | | | 413,640 | 41,364 | | PV1, |
| | SS-44-6-NC-NR | 455 Stainless Steel | Stem and Actuator | (60,000) | (6000) | $O_2$ | PV2 |
| | | TFE | Seat | | | $O_2$ | |
| | | 17-4 PH Stainless Steel | Back Ferrule | | | $O_2$ | |
| | | Phosphor Bronze | Packing Nut Insert | | | Air | |
| | | 316 Stainless Steel | Body and Front Ferrule | | | $O_2$ | |
| Valve, Electric | Skinner Precision Ind. | | | 689 | 689 | | SV1 |
| | V52DB2100 | ZMD & Chromate Coating | Coil Housing | (100) | (100) | Air | |
| | | 303 Stainless Steel | Sleeve | | | $O_2$ | |
| | | 18-8 Stainless Steel | Body | | | $O_2$ | |
| | | TFE | Seat | | | $O_2$ | |
| | | Epoxy | Coil Coating | | | Air | |
| Valve, Electric | Skinner Precision Ind. | | | 1034 | 896 | | SV2 - |
| | V53DB2150 | ZMD & Chromate Coating | Coil Housing | (150) | (130) | Air | SV5 |
| | | 303 Stainless Steel | Sleeve | | | $N_2$ | |
| | | 18-8 Stainless Steel | Body | | | $N_2$ | |
| | | TFE | Seat | | | Air | |
| | | Epoxy | Coil Coating | | | Air | |
| Valve, Electric | Skinner Precision Ind. | | | 689 | 417 | | SV6, |
| | V52DB2100 | ZMD & Chromate Coating | Coil Housing | (100) | (60) | Air | SV7 |
| | | 303 Stainless Steel | Sleeve | | | Water | |
| | | 18-8 Stainless Steel | Body | | | Water | |
| | | TFE | Seat | | | Air | |
| | | Epoxy | Coil Coating | | | | |
| Valve, Regulating | Whitey Company | | | 20,682 | 417 | | V1, |
| | B-IRS4 | Brass | Body | (3000) | (60) | Water | V2 |
| | | 303 Stainless Steel | Stem | | | Water | |
| | | TFE | Seat | | | Water | |
| | | Phenol | Knob | | | Air | |
| Valve, Ball | Whitey Company | | | 20,682 | 689 | | V3 |
| | SS-43S4 | 316 Stainless Steel | Body, Stem | (3000) | (100) | $O_2$ | |
| | | Nylon | Handle | | | Air | |
| | | TFE | Packing | | | $O_2$ | |
| Valve, Three-Way[(a)] | Whitey Company | | | 17,235 | 10,341 | | V4 |
| | SS-43XS4 | 316 Stainless Steel | Body, Stem | (2500) | (1500) | KOH | |
| | | Nylon | Handle | | | Air | |
| | | TFE | Packing | | | KOH | |
| Valve, Check | Nupro Company | | | 41,364 | 41,364 | | CV1, |
| | SS-53S4 | 316 Stainless Steel | Body | (6000) | (6000) | $O_2$ | CV2 |
| | | 17-4 PH Stainless Steel | Lift Plug | | | $O_2$ | |
| Valve, Relief | Nupro Company | | | 20,682 | 689 | | PR2 |
| | SS-4CA-3 | 316 Stainless Steel | Body, Poppet, Nuts, Ferrules | 3000 | (100) | $O_2$ | |
| | | 302 Stainless Steel | Spring | | | $O_2$ | |
| | | TFE | Gasket | | | $O_2$ | |
| Regulator, Pressure | Tescom Corp. | | | 41,364 | 689 | | PR1 |
| | 26-1520-24-007 | 316 Stainless Steel | Body, Bonnet, Trim | (6000) | (100) | $O_2$ | |
| | | TFE | Seat, Backup Rings | | | $O_2$ | |
| | | Viton-A | O-Rings | | | $O_2$ | |

(a) Installed on Modified Test Stand, (Dwg. No. LSI-D-1138) Only

continued-

Table 7 - continued

| | | | | |
|---|---|---|---|---|
| **Life Systems, Inc.**<br>CLEVELAND, OHIO 44122 | MATERIAL DATA SUMMARY | ☒ NONMETALLIC<br>☒ METALLIC | | NO. 512-1033 | REVISION LTR. A |
| | TITLE ELECTROCHEMICAL OXYGEN COMPRESSOR TEST FACILITY | | DRAWING NO. LSI-D-690, LSI-D-1138 | PAGE 2 OF 3 | DATE 5/75 |

| Part Description | Manufacturer And Mfgr's Designation | Material Description | Use | Max. Pressure kN/m$^2$ (Psig) | Max. Oper. Pressure kN/m$^2$ (Psig) | Environment | Dwg. Ref. |
|---|---|---|---|---|---|---|---|
| Regulator, Back | Tescom Corp. | | | 68,940 | 41,364 | | PR3 |
| Pressure | 26-1725-24-014 | 316 Stainless Steel | Body, Bonnet, Trim | (10,000) | (6000) | $O_2$ | |
| | | TFE | Seat, Backup Rings | | | $O_2$ | |
| | | Viton-A | O-Rings | | | $O_2$ | |
| Regulator, Cylinder | Tescom Corp. | | | 689 | 689 | | $O_2$ |
| | 44-2461-24 | 316 Stainless Steel | Body, Bonnet, Diaphragm, Trim | (100) | (100) | $O_2$ | Suppl. |
| | | TFE | Seat | | | $O_2$ | |
| Regulator, Cylinder | Tescom Corp. | | | 1034 | 896 | | $N_2$, Ar |
| | H-1530-580 | Brass | Body, Trim | (150) | (130) | $N_2$, Ar | Suppl. |
| | | Buna-N | Seats, Diaphragm | | | $N_2$, Ar | |
| | | Bronze | Filter | | | $N_2$, Ar | |
| | | Steel | Bonnet | | | $N_2$, Ar | |
| Restrictor, Flow | Lee Company | | | 41,364 | 41,364 | | FR1, |
| | 13-EVL1-CM | 316 Stainless Steel | Housing | (6000) | (6000) | $O_2$ | FR2 |
| | | 304 Stainless Steel | Screen Assembly | | | $O_2$ | |
| | | 303 Stainless Steel | Body, Pin | | | $O_2$ | |
| Restrictor, Flow(a) | Lee Company | | | 41,364 | 10,340 | | FR3 |
| | JETA 1875 300H | 316 Stainless Steel | Housing | (6000) | (1500) | $N_2$ | |
| | | 304 Stainless Steel | Screen Assembly | | | $N_2$ | |
| | | 303 Stainless Steel | Body, Pin | | | $N_2$ | |
| Thermocouple | Marlin Mfg. | | | 41,364 | 41,364 | | TS1- |
| | G-57-J-F12A-6-CE | 316 Stainless Steel | Sheath | (6000) | (6000) | $O_2$, Water | TS8 |
| | | Iron-Constantan | Thermocouple Junction | | | Air | |
| | | Plastic | Connector | | | Air | |
| Transducer, Pressure | Viatran Corp. | | | 51,705 | 34,470 | | PS1, |
| | Model 103 | 316 Stainless Steel | All | (7500) | (5000) | $O_2$ | PS2 |
| Cylinder, Sample | Hoke Inc. | | | 2758 | 689 | | $O_2$ |
| | 4HD150 | 304 Stainless Steel | All | (400) | (100) | $O_2$, Water | Humid. |
| Cylinder, Storage(a) | Hoke Inc. | | | 12,409 | 10,341 | | T1 |
| | 4HD5000 | 304 Stainless Steel | All | (1800) | (1500) | KOH | |
| Beaker | Matheson Scientific | | | NA(b) | Ambient | | Cell |
| | 3950-70 | 316 Stainless Steel | All | | | Water | Temp |
| | | | | | | | Cont. |
| Heater, Cylindrical | Watlow Electric | | | NA | Ambient | | H1, |
| | 7533 BX | Steel | Heater Band | | | Air | H2 |
| | | Ceramic | Terminal Covers | | | Air | |
| Pump, Coolant | S.Gelber & Sons Inc. | | | NA | NA | | P1, |
| | 2E-38N | Plastic | All | | | Water | P2 |
| Cell, Electrochem. | Life Systems Inc. | | | 103,410 | 41,364 | | $O_2$ |
| | NA | Nickel 200 | Electrodes, Shims | (15,000) | (6000) | $O_2$ | Conc. |
| | | Inconel 600 | Base, Cover | | | $O_2$ | |
| | | 316 Stainless Steel | Housing | | | Air | |
| | | Polysulfone | Locating Pins, Spacers | | | Air | |
| | | Viton-A | O-Rings | | | $O_2$ | |
| | | TFE | Membrane | | | $O_2$ | |
| | | Asbestos | Matrix | | | $O_2$ | |

(b) NA = Not Applicable

Table 7 - continued

| | | | | | |
|---|---|---|---|---|---|
| **Life Systems, Inc.**<br>CLEVELAND, OHIO 44122 | MATERIAL DATA SUMMARY | ☒ NONMETALLIC<br>☒ METALLIC | | NO. 512-1033 | REVISION LTR. A |
| | TITLE ELECTROCHEMICAL OXYGEN COMPRESSOR TEST FACILITY | | DRAWING NO. LSI-D-690, LSI-D-1138 | PAGE 3 OF 3 | DATE 5/75 |

| Part Description | Manufacturer And Mfgr's Designation | Material Description | Use | Max. Pressure kN/m$^2$ (Psig) | Max. Opgr. Pressure kN/m$^2$ (Psig) | Environment | Dwg. Ref. |
|---|---|---|---|---|---|---|---|
| Flowmeter, Soap | Chemical Research | | | Ambient | Ambient | | FM1 |
| Bubble | Services Inc. | Glass | Tube | | | $O_2$,Air, | |
| | 10 ml | | | | | Water | |
| | | Steel | Mounting | | | Air | |
| Fittings | Crawford Fitting Co. | | | 82,728 | 41,364 | | NA |
| | NA | 316 Stainless Steel | All | (12,000) | (6000) | $O_2$ | |
| Fittings | Crawford Fitting Co. | | | 25,852 | 417 | | NA |
| | NA | Brass | All | (3750) | (60) | Water | |
| Tubing | Williams & Company | | | 58,599 | 41,364 | | NA |
| | NA | Inconel 600 | All | (8500) | (6000) | $O_2$ | |
| Tubing | Williams & Company | | | 40,674 | 417 | | NA |
| | NA | 316 Stainless Steel | All | (5900) | (100) | $O_2$ | |
| Tubing | Williams & Company | | | 13,030 | 417 | | NA |
| | NA | Copper | All | (1890) | (60) | Water | |
| Frame | Unistrut | | | NA | Ambient | | Sys. |
| | NA | Mild Steel | All | | | Air | Frame |

Criteria 1 and 2 represent absolute criteria that the membrane must meet for EOC application. Criteria 3-6 affect EOC system total equivalent weight for EVA application and for comparison to alternate $O_2$ compression techniques.

## Commercial Membranes

The choice of a commercial membrane material was most severely limited by the high pressure $O_2$ and KOH compatibility requirements. Possible useful inorganic materials considered were asbestos and porous glass. Asbestos has little potential for surpassing 6900 to 13,800 $kN/m^2$ (100 to 200 psid) differential pressure and was eliminated. Porous silicate glass (4 x $10^{-9}$m (1.6 x $10^{-7}$in) diameter pores, 24% porosity) has the potential for very high differential pressure tolerance, but is not compatible with KOH. No other inorganic materials for the application were identified.

The combustion hazard of organic materials was minimized by considering the fluorinated hydrocarbon materials. Fluorinated hydrocarbon based ion exchange membranes are available from DuPont and RAI Research Corporation. The properties of these membranes are listed in Table 8.

The DuPont membrane was selected for EOC testing for the following reasons:

1. The membrane is fully fluorinated, unlike the RAI membranes available.
2. The ion exchange group is sulfonic acid, a highly stable, highly oxidized functional group.
3. It was felt that the 0.008 to 0.025 cm (0.003 to 0.010 in) thickness would reduce $O_2$ backdiffusion to give better current efficiency and would have a higher differential pressure stability than the thinner RAI membranes available.
4. Water permeability of the membrane is very low.

## Customized Membranes Developed

Tables 9a and 9b (SI and common units, respectively) summarize the properties of the membranes developed specifically for EOC application. A total of 15 membranes were fabricated, having various resistance and water permeability characteristics. The difference in properties depends on (1) the side chain functional group that was attached to the basic fluorinated hydrocarbon backbone and (2) the degree of crosslinking both chain-to-chain and chain-to-backbone.

All but four membranes were capable of withstanding differential pressures greater than 10,700 $kN/m^2$ (3000 psid). Those that did not withstand the differential pressure test were brittle and had rough surfaces. Membranes No. 12 and 13 met the water permeability specifications and Membranes No. 1, 2, 3, 4, 8, and 14 met the resistance specifications. No membranes met the specification goals for all membrane properties.

## TABLE 8 COMMERCIAL FLUORINATED MEMBRANE PROPERTIES

| Manufacturer | Designation | Backbone | Graft Groups | Thickness, cm (In) | Resistance, ohm-cm$^2$ (ohm-In$^2$) | Water Permeability, g/hr-cm$^2$-kN/m$^2$ (g/hr-cm$^2$-Psid) | Diff. Pressure, kN/m$^2$ (Psid) |
|---|---|---|---|---|---|---|---|
| DuPont | Nafion XR-170 | PSEPVE/TFE (a) | -- | 0.025<br>(0.010) | 3.8<br>(0.589) | $1.6 \times 10^{-5}$<br>($1.08 \times 10^{-4}$) | 20,700<br>(>3,000) |
| RAI | Permion 1010 | FEP (b) | SSA (c) | 0.003<br>(0.001) | 0.161<br>(0.025) | NA (d) | (>3,000) |
| RAI | Permion 1020 | FEP | Pyridine | 0.003<br>(0.001) | 0.071<br>(0.011) | NA | (>3,000) |
| RAI | Permion 1025 | FEP | N-Methyl Pyridine | 0.003<br>(0.001) | 0.071<br>(0.011) | NA | (>3,000) |
| RAI | Permion 1030 | FEP | Methacrylic Acid | 0.003<br>(0.001) | 1.29<br>(0.2) | NA | (>3,000) |

---

(a) PSEPVE/TFE = Poly sulfonyl fluoride vinyl ether/tetrafluoroethylene copolymer
(b) FEP = Fluorinated ethylene-propylene
(c) SSA = Styrene sulfonic acid
(d) NA = Not Available

## TABLE 9a SUMMARY OF FABRICATED EOC MEMBRANE PROPERTIES (SI)

| Membrane Number | Backbone | Graft Group | Thickness, cm | Resistance, ohm-cm$^2$ | Water Permeability, g/hr-cm$^2$-kN/m$^2$ | Differential Pressure, kN/m$^2$ |
|---|---|---|---|---|---|---|
| 1 | FEP(a) | SSA(b) | 0.005 | 0.23 | $2.0\text{x}10^{-5}$ | 20,700 |
| 2 | FEP | SSA | 0.005 | 0.11 | $3.4\text{x}10^{-5}$ | 20,700 |
| 3 | FEP | SSA | 0.013 | 0.33 | $1.0\text{x}10^{-5}$ | 20,700 |
| 4 | FEP | SSA | 0.013 | 0.25 | $2.2\text{x}10^{-5}$ | 20,700 |
| 5 | FEP | SSA | 0.013 | 1.29 | $7.5\text{x}10^{-6}$ | 20,700 |
| 6 | FEP | AMS(c)/DVB(d) | 0.005 | 3.61 | NA(e) | Would Not Seal |
| 7 | FEP | AMS/DVB | 0.005 | 45.20 | NA | Would Not Seal |
| 8 | FEP | SSA/DVB | 0.005 | 0.45 | $13.9\text{x}10^{-6}$ | 20,700 |
| 9 | FEP | SSA/DVB | 0.005 | 10.30 | $1.4\text{x}10^{-5}$ | Would Not Seal |
| 10 | FEP | SSA/DVB | 0.005 | 45.20 | NA | Cracked On Ass'y |
| 11 | FEP | SSA/AMA(f) | 0.005 | 3.22 | $5.2\text{x}10^{-6}$ | 20,700 |
| 12 | FEP | SSA/AMA | 0.005 | 23.90 | $3.0\text{x}10^{-7}$ | 20,700 |
| 13 | FEP | SSA/AMA | 0.005 | 23.90 | $3.0\text{x}10^{-7}$ | 20,700 |
| 14 | FEP | SSA/DVB | 0.005 | 0.77 | $2.9\text{x}10^{-5}$(g) | 20,700 |
| 15 | FEP | SSA/DVB | 0.005 | 2.54 | $7.2\text{x}10^{-6}$(g) | 20,700 |

---

(a) FEP = Fluorinated ethylene-propylene
(b) SSA = Styrene sulfonic acid
(c) AMS = α-methyl styrene sulfonic acid
(d) DVB = Divinyl benzene (crosslinking agent)
(e) NA = Not Available
(f) AMA = Alyl methacrylate (crosslinking agent)
(g) Estimated based on similar membranes

TABLE 9b SUMMARY OF FABRICATED EOC MEMBRANE PROPERTIES (COMMON)

| Membrane Number | Backbone | Graft Group | Thickness, In | Resistance, ohm-In$^2$ | Water Permeability, g/hr-cm$^2$-Psid | Differential Pressure, Psid |
|---|---|---|---|---|---|---|
| 1 | FEP(a) | SSA(b) | 0.002 | 0.04 | $1.38x10^{-4}$ | >3,000 |
| 2 | FEP | SSA | 0.002 | 0.02 | $2.34x10^{-4}$ | >3,000 |
| 3 | FEP | SSA | 0.005 | 0.05 | $0.72x10^{-4}$ | >3,000 |
| 4 | FEP | SSA | 0.005 | 0.04 | $1.50x10^{-4}$ | >3,000 |
| 5 | FEP | SSA | 0.005 | 0.2 | $0.52x10^{-4}$ | >3,000 |
| 6 | FEP | AMS(c)/DVB(d) | 0.002 | 0.6 | NA(e) | Would Not Seal |
| 7 | FEP | AMS/DVB | 0.002 | 7.0 | NA | Would Not Seal |
| 8 | FEP | SSA/DVB | 0.002 | 0.07 | $9.6x10^{-5}$ | >3,000 |
| 9 | FEP | SSA/DVB | 0.002 | 1.6 | NA | Would Not Seal |
| 10 | FEP | SSA/DVB | 0.002 | 7.0 | NA | Cracked On Ass'y |
| 11 | FEP | SSA/AMA(f) | 0.002 | 0.5 | $3.6x10^{-5}$ | >3,000 |
| 12 | FEP | SSA/AMA | 0.002 | 3.7 | $2.1x10^{-6}$ | >3,000 |
| 13 | FEP | SSA/AMA | 0.002 | 3.7 | $2.1x10^{-6}$ | >3,000 |
| 14 | FEP | SSA/DVB | 0.002 | 0.12 | $2x10^{-4}$(g) | >3,000 |
| 15 | FEP | SSA/DVB | 0.002 | 0.4 | $5x10^{-5}$(g) | >3,000 |

---

(a) FEP = Fluorinated ethylene-propylene
(b) SSA = Styrene sulfonic acid
(c) AMS = α-methyl styrene sulfonic acid
(d) DVB = Divinyl benzene (crosslinking agent)
(e) NA = Not Available
(f) AMA = Alyl methacrylate (crosslinking agent)
(g) Estimated based on similar membranes

The ultimate criteria for the selection of a membrane is operation in an EOC cell. Membranes 2, 3, and 14 were selected for EOC testing for the following reasons:

1. Membranes 2 and 3 represented the best combination of low resistance and low water permeability.
2. Membrane 14 represented the best combination of low resistance and low water permeability from the crosslinked membranes fabricated.

## TEST PROGRAM

The EOC test program consisted of:

1. Component checkout and calibration tests
2. Shakedown test
3. Parametric tests
4. A Design Verification Test (DVT)

### Component Checkout and Calibration Test

Calibration tests were completed for all test facility parameter monitoring equipment: ammeters, voltmeters, thermocouples, and pressure transducers. In addition, the shutdown set-point controls were calibrated by electrically simulating levels at the sensor inputs. Component checkout tests were completed on all test facility components to insure designed operation prior to shakedown testing.

### Shakedown Testing

The following activities were completed to insure the integrated operability of the test facility and EOC cell:

1. Shutdown set-points were set using actual sensor inputs.
2. The automatic shutdown and purge sequence was tested by initiating an actual excessive EOC cell temperature shutdown.
3. All system shutdowns and set-points were verified by simulating actual shutdown conditions.
4. The test facility and EOC cell were operated continuously for 24 hours.

During the shakedown testing a problem with an electrical ground loop which caused erratic transducer readings was discovered and fixed.

## Parametric Test Program

The Parametric Test Program was divided into two phases. The first phase consisted of a series of four parametric tests to characterize the performance of an EOC cell using the selected commercially available membrane. Data generated during this phase was used to support the membrane development work completed. The second phase consisted of testing the membranes selected from those fabricated specifically for EOC application to determine EOC feasiblity.

### Initial Parametric Tests

A series of four parametric tests were run to characterize EOC performance as a function of:

1. Current density
2. Differential pressure
3. Time after startup
4. Oxygen backdiffusion

Effect of Current Density. Cell voltage as a function of current density is shown in Figure 16. At 35 $mA/cm^2$ (33 ASF) activation polarization has approached a maximum. The limiting current density is approximately 80 $mA/cm^2$ (74 ASF).

Effect of Differential Pressure. The effect of differential pressure on electrochemical cell performance was determined by imposing a constant potential between the electrodes and monitoring cell current density as a function of differential pressure. The results of the test are shown in Figure 17. At 1.5V cell voltage, the current density was 33 $mA/cm^2$ (31 ASF). Current density decreases rapidly with increasing differential pressure. At 1946 $kN/m^2$ (282 psid) the current density was only 2.7 $mA/cm^2$ (1.5 ASF). The drop in current density occurs as follows: as differential pressure increases, the anode to cathode water permeation rate increases while the water generation rate at the anode is proportional to the current. The resulting imbalance leaves the larger anode pores dry and the cathode flooded. This explanation was supported upon cell disassembly. The cathode contained excess electrolyte while the anode was dry.

Cell Startup Polarization. Figure 18 shows the development of cell voltage as a function of time after startup for a current density of 50 $mA/cm^2$ (46 ASF). Activation polarization is fully developed almost immediately. Cell voltage rises slowly at first, then more rapidly, then levels off. The data can be explained as follows. The initial slow rise is the effect of a gradual drying of the anode and a gradual moistening of the cathode. The rapid rise occurs when the cathode pores become saturated, preventing sufficient $O_2$ from reaching active reaction sites with the electrode. At this point the voltage rises until

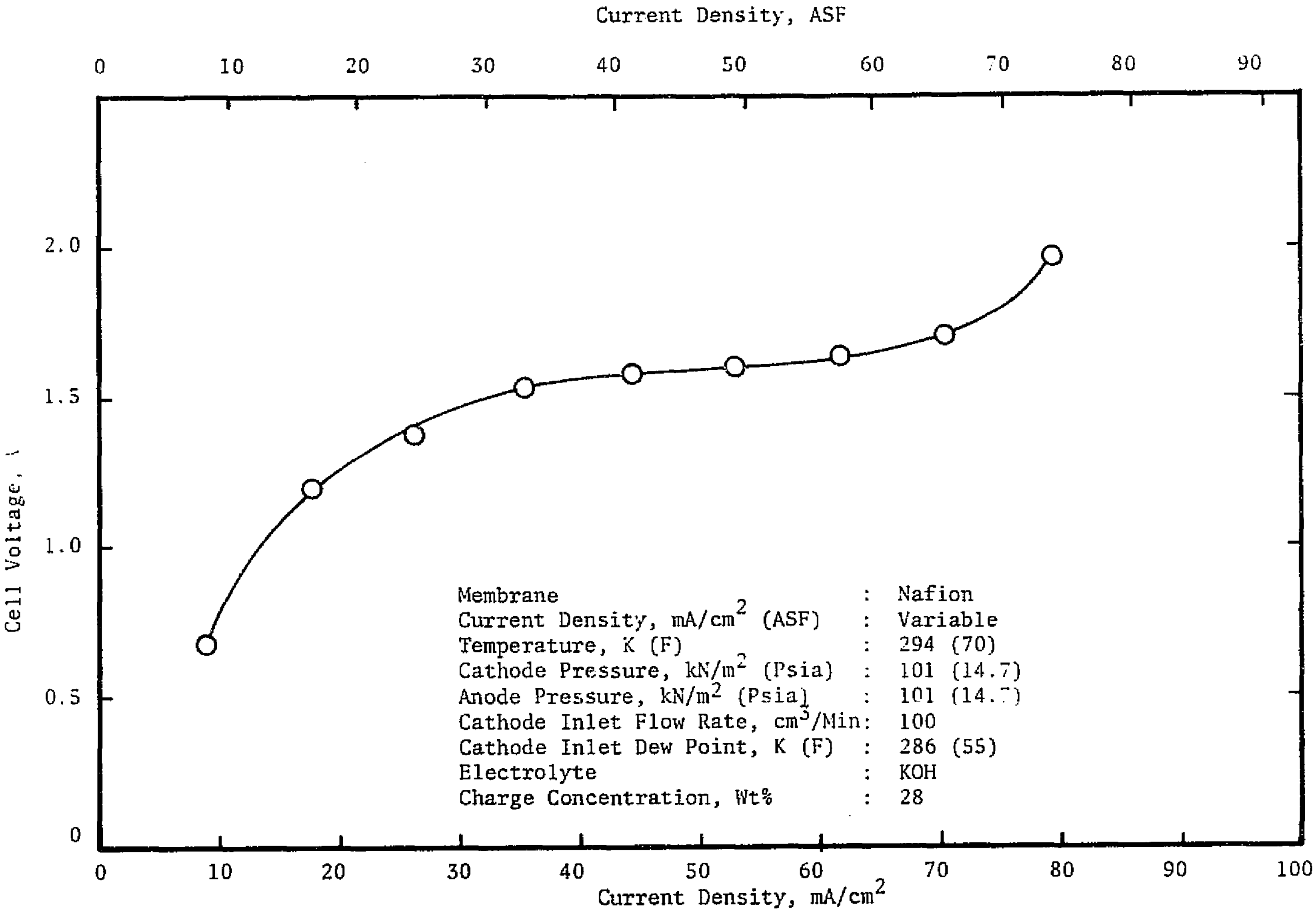


FIGURE 16 CELL VOLTAGE VERSUS CURRENT DENSITY (NAFION)

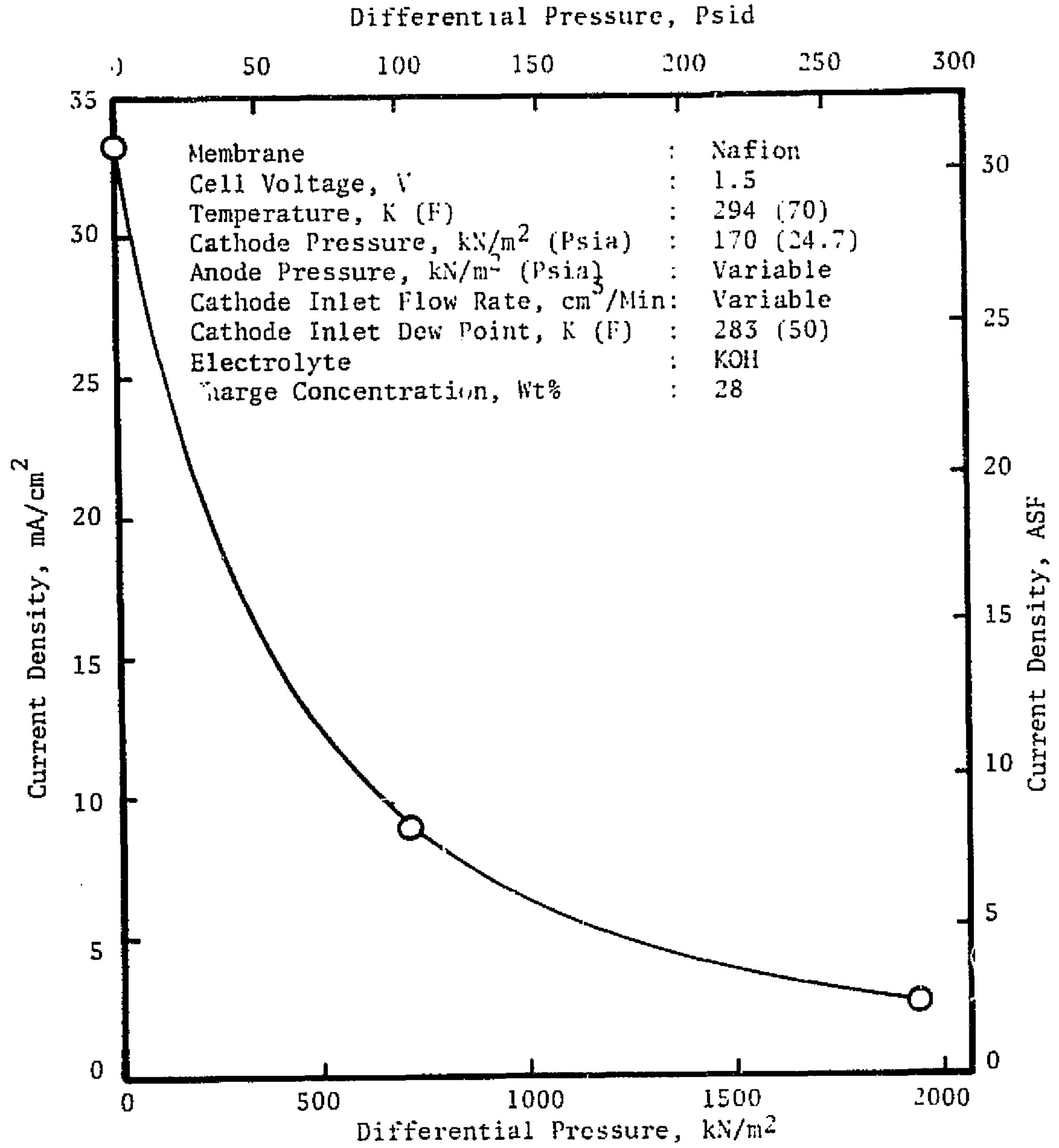
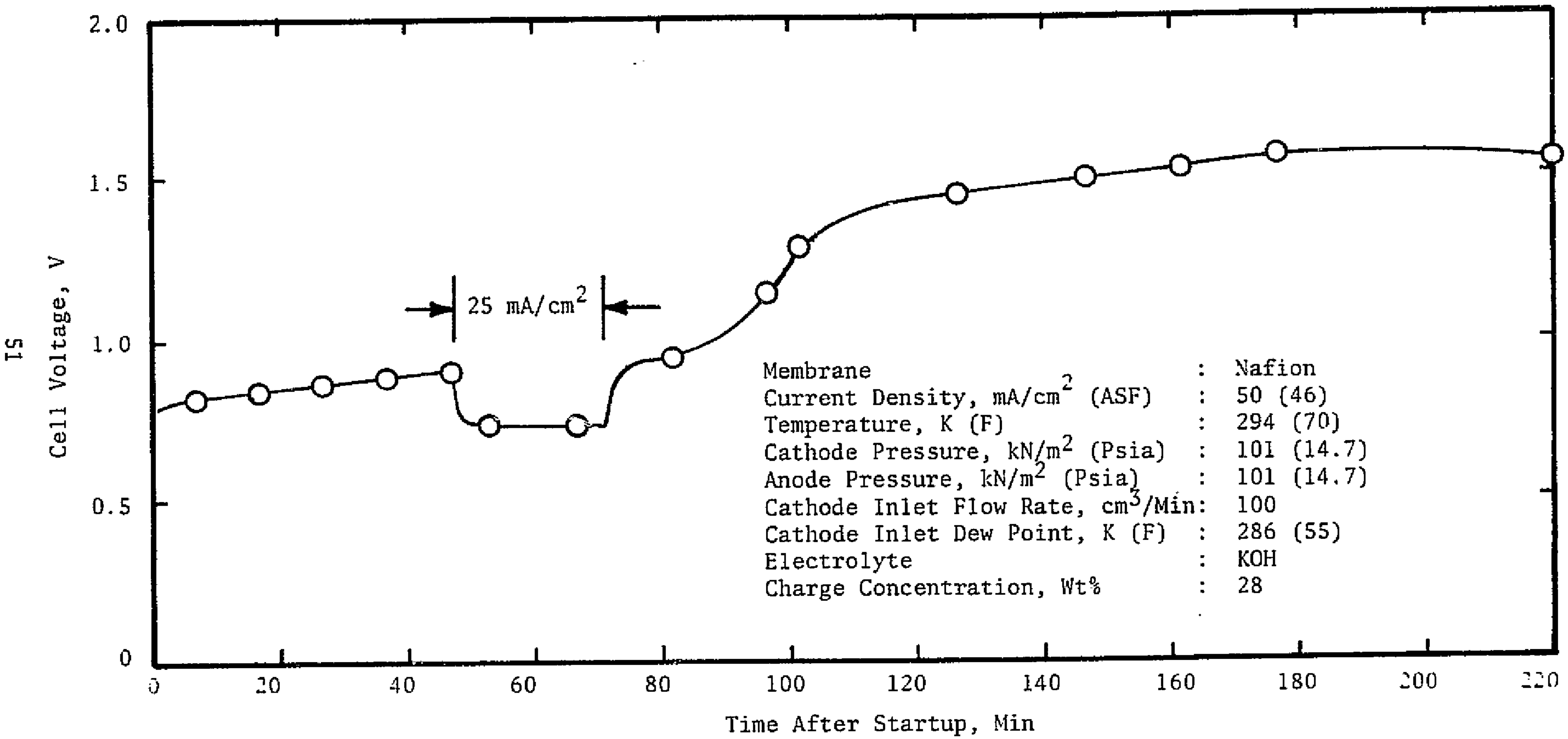


FIGURE 17 CURRENT DENSITY VERSUS DIFFERENTIAL PRESSURE



Membrane : Nafion
Current Density, mA/cm$^2$ (ASF) : 50 (46)
Temperature, K (F) : 294 (70)
Cathode Pressure, kN/m$^2$ (Psia) : 101 (14.7)
Anode Pressure, kN/m$^2$ (Psia) : 101 (14.7)
Cathode Inlet Flow Rate, cm$^3$/Min: 100
Cathode Inlet Dew Point, K (F) : 286 (55)
Electrolyte : KOH
Charge Concentration, Wt% : 28

FIGURE 18 CELL VOLTAGE VERSUS TIME AFTER STARTUP

$H_2$ evolution occurs. Any $H_2$ formed, however, is oxidized immediately and does not leave the cell unless the current density exceeds about 70 to 80 $mA/cm^2$ (65 to 74 ASF). The stoichiometric amount of $O_2$, therefore, is still consumed at the cathode.

The mechanism of the moisture imbalance can be represented by three sequential steps, although they actually overlap.

1. At startup, $OH^-$ are generated at the cathode faster than they can transfer through the membrane to the anode. Potassium ions ($K^+$) migrate towards the cathode to maintain electrical neutrality. The cell current is therefore carried by the migration of both $K^+$ and $OH^-$. During this transient period, cell voltage remains low as both ions carry current.
2. The transfer of $K^+$ (i.e., buildup of KOH at the cathode) continues until the electrolyte concentration gradient and cell potential gradient balance at steady-state and cell current is carried by $OH^-$.
3. The resulting steady-state KOH concentration gradient established causes water to transfer to the cathode leaving little electrolyte at the anode.

For the membrane tested, low membrane conductivity caused a large electrolyte concentration gradient. The water transfer rate through the membrane caused by the concentration gradient established was high which resulted in cathode flooding. Since the water permeability of the membrane is actually very low, cathode flooding can be attributed primarily to low membrane conductivity although it is actually the ratio of the two membrane properties that affects the water transfer rate.

<u>Oxygen Backdiffusion</u>. An important parameter in characterizing the performance of any electrochemical device is the current efficiency. In the case of the EOC, the current efficiency is defined as the standard $cm^3/min$ of $O_2$ compressed divided by the standard $cm^3$ of $O_2$ electrochemically generated at the anode. The electrochemical $O_2$ generation rate is equal to 3.754 $cm^3$ $O_2$-$min^{-1}A^{-1}$ at 294K (70F) and 1 atmosphere. The rate of $O_2$ actually compressed is less than this amount because some $O_2$ diffuses back through the membrane.

A test was performed to measure the $O_2$ backdiffusion rate separately from the much larger electrochemical production rate. The results were used to predict actual cell current efficiencies. The backdiffusion rate was measured in the assembled test cell. The anode was pressurized with $O_2$ and the $O_2$ which diffused through to the cathode was collected and made to raise the level of water in a 1 ml calibrated pipe. Figure 19 shows the results of the test. After a few minutes the diffusion rate became steady at $1.53 \times 10^{-4}$ $cm^3/cm^2$-min at 689 $kN/m^2$ (100 psid) and $7.59 \times 10^{-5}$ $cm^3/cm^2$-min at 345 $kN/m^2$ (50 psid). Figure 20 gives projected current efficiencies for the present cell design as a function of differential pressure and current density.

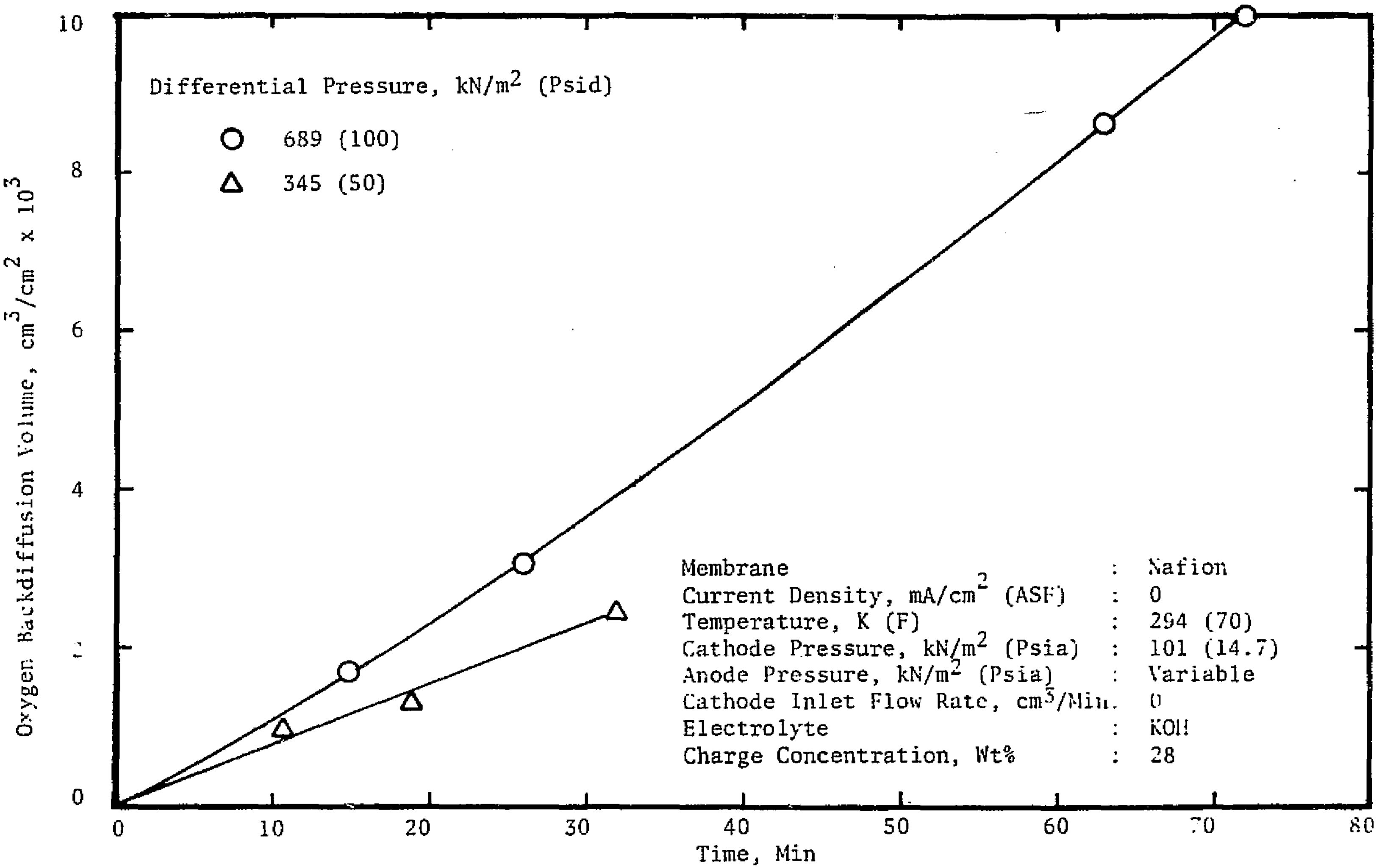


FIGURE 19 OXYGEN BACKDIFFUSION VERSUS TIME

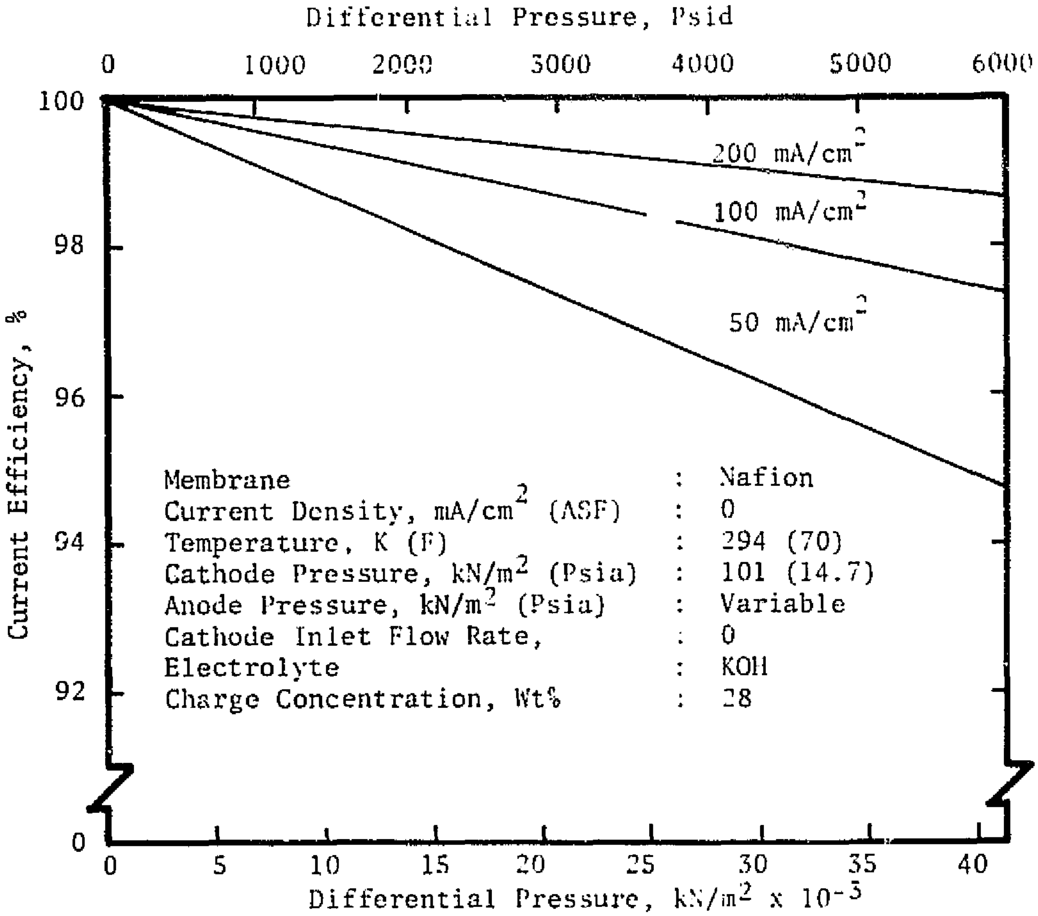


FIGURE 20 PROJECTED CURRENT EFFICIENCY VERSUS DIFFERENTIAL PRESSURE

## Feasibility Parametric Tests

The effect of current density and differential pressure on cell performance was experimentally determined for selected membranes from those fabricated specifically for EOC application.

Effect of Current Density. The effect of current density on cell voltage for membrane LSI-3 is given in Figure 21. The membrane was run with and without an electrolyte reservoir. Limiting current densities of approximately 50 mA/cm$^2$ (46 ASF) were observed for all membranes when tested without the electrolyte reservoir to maintain moisture balance. In addition, operation without the electrolyte reservoir could only be maintained for short periods of time (less than 24 hours). A comparison of the cell voltages obtained for the membranes LSI-2, 3 and 14 when operated with the anode electrolyte reservoir is presented in Figure 22. The limiting current density for the membranes exceeded the capability of the test facility, approximately 400 mA/cm$^2$ (372 ASF).

The effect of current density on electrolyte permeation rate is given in Figure 23. The rate of electrolyte transfer through membrane LSI-2 was highly dependent on current density as expected based on the initial test results obtained with Nafion.

Effect of Differential Pressure. The effect of differential pressure on cell voltage for the membrane LSI-2 is presented in Figure 24. The electrolyte permeation rate as a function of $\Delta P$ is also shown in Figure 24. Both cell voltage and electrolyte permeation rate only increased slightly with increased differential pressure.

### Design Verification Test

A 200-hour DVT was performed using membrane LSI-2. Cell voltage and electrolyte permeation rate as a function of time is presented in Figure 25. The average cell voltage was 1.4V at 100 mA/cm$^2$ (93 ASF) and the average electrolyte permeation rate was 3 cm$^3$/hr. The test was only interrupted to manually refill the reservoir tank.

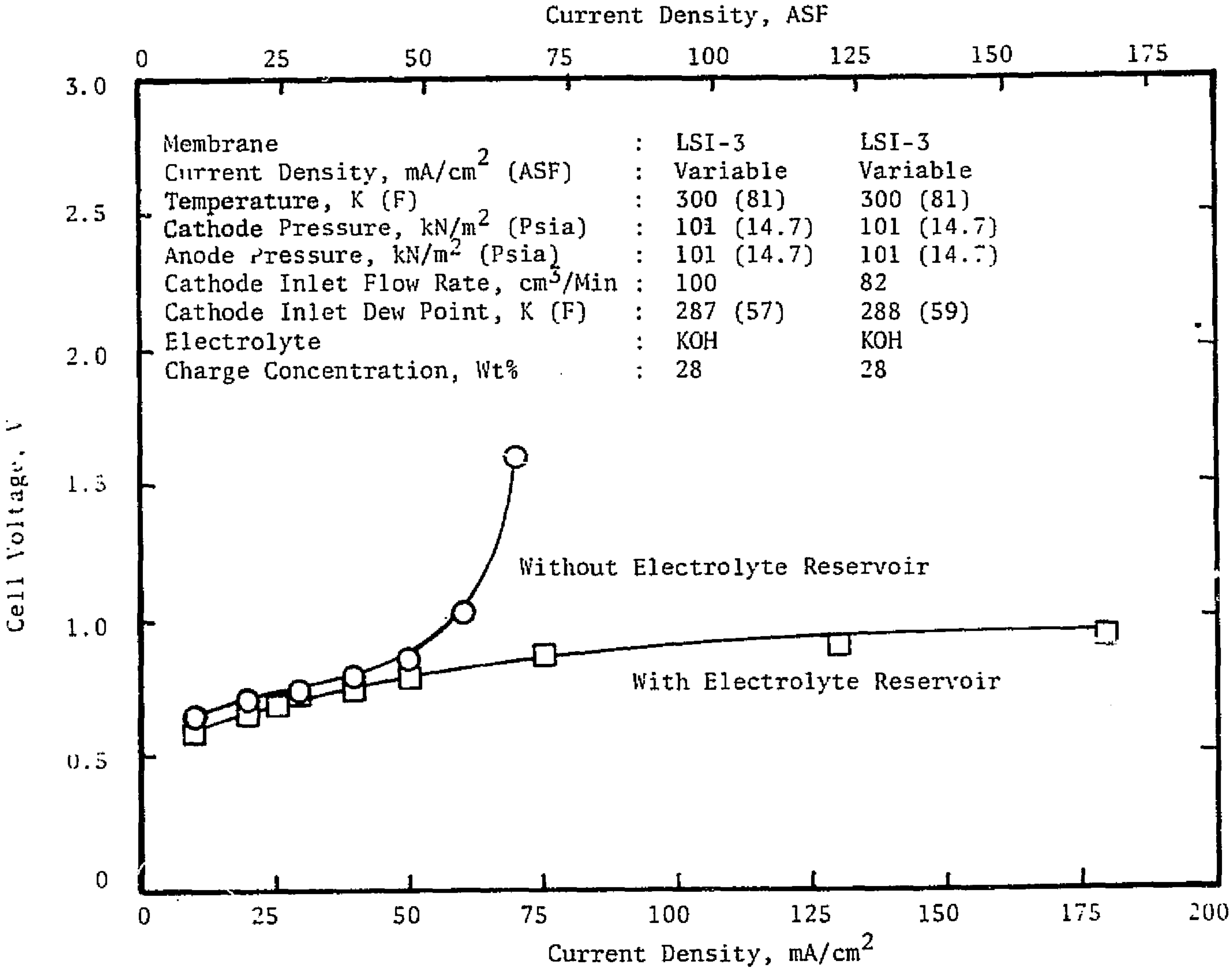
## SUPPORTING TECHNOLOGY

To support the EOC cell design, studies were completed to:

1. Identify compatible materials
2. Analyze the effect of $O_2$ backdiffusion on cell current efficiency

### Materials Compatibility

The selection of the materials used in the EOC cell and test facility was determined primarily by compatibility with high pressure $O_2$ and chemical compatibility with KOH. A summary of the materials used was presented previously in Table 7.



| | | | |
|---|---|---|---|
| Membrane | : | LSI-3 | LSI-3 |
| Current Density, mA/cm$^2$ (ASF) | : | Variable | Variable |
| Temperature, K (F) | : | 300 (81) | 300 (81) |
| Cathode Pressure, kN/m$^2$ (Psia) | : | 101 (14.7) | 101 (14.7) |
| Anode Pressure, kN/m$^2$ (Psia) | : | 101 (14.7) | 101 (14.7) |
| Cathode Inlet Flow Rate, cm$^3$/Min | : | 100 | 82 |
| Cathode Inlet Dew Point, K (F) | : | 287 (57) | 288 (59) |
| Electrolyte | : | KOH | KOH |
| Charge Concentration, Wt% | : | 28 | 28 |

FIGURE 21 COMPARISON OF THE EFFECT OF CURRENT DENSITY WITH AND WITHOUT ELECTROLYTE RESERVOIR

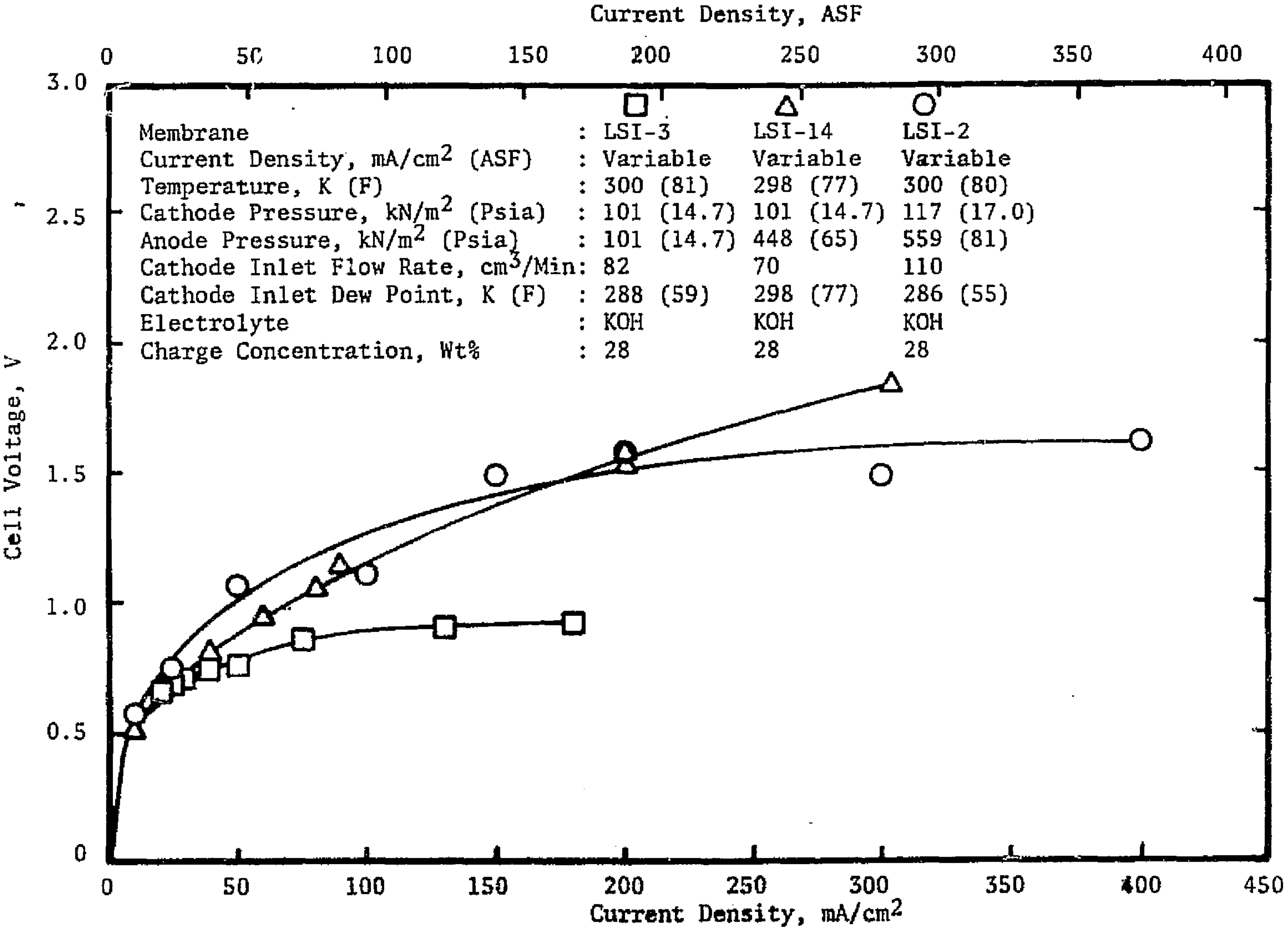


FIGURE 22 CELL VOLTAGE VERSUS CURRENT DENSITY (MEMBRANES 2, 3 AND 14)

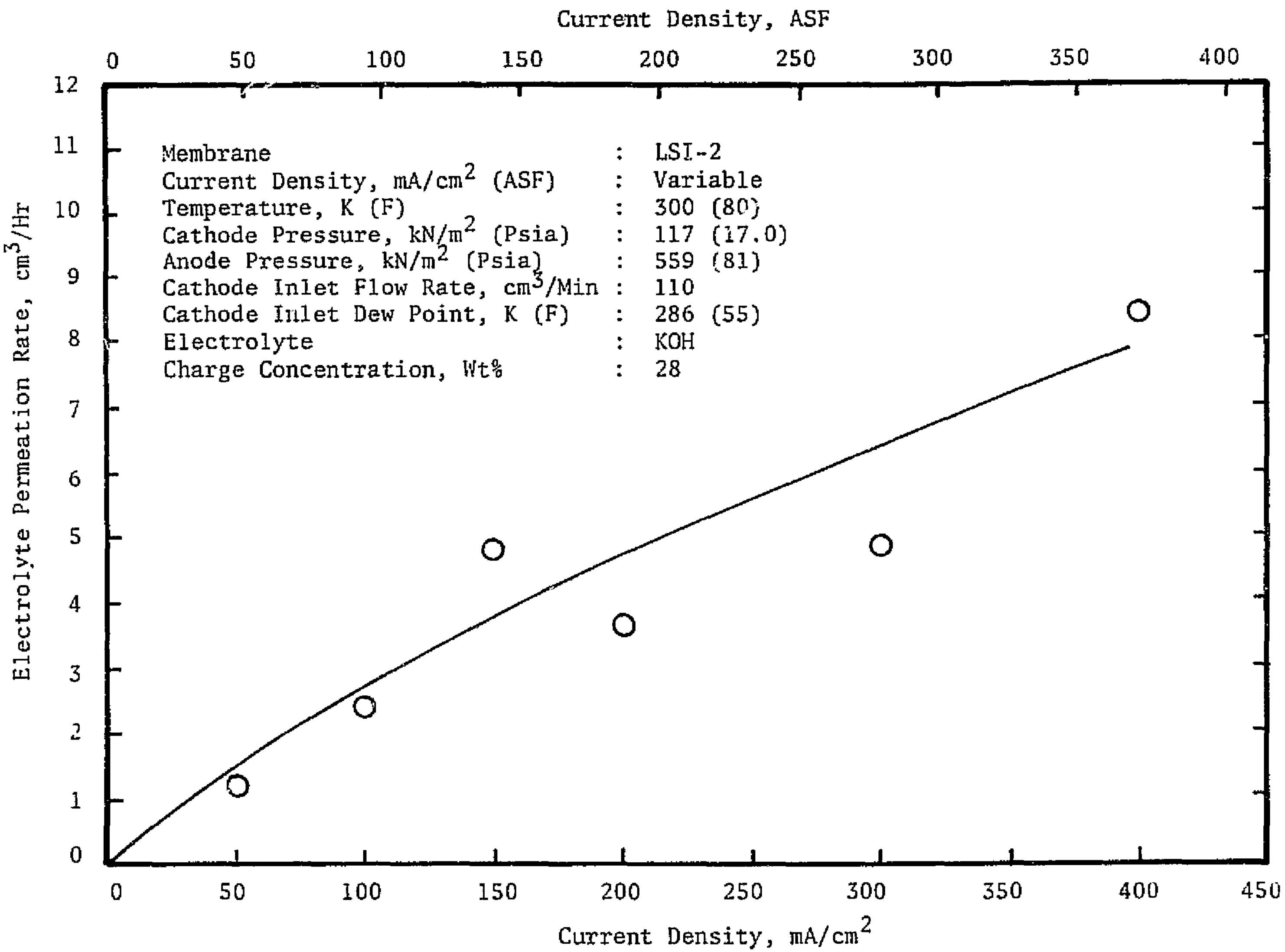


FIGURE 23 ELECTROLYTE PERMEATION RATE VERSUS CURRENT DENSITY

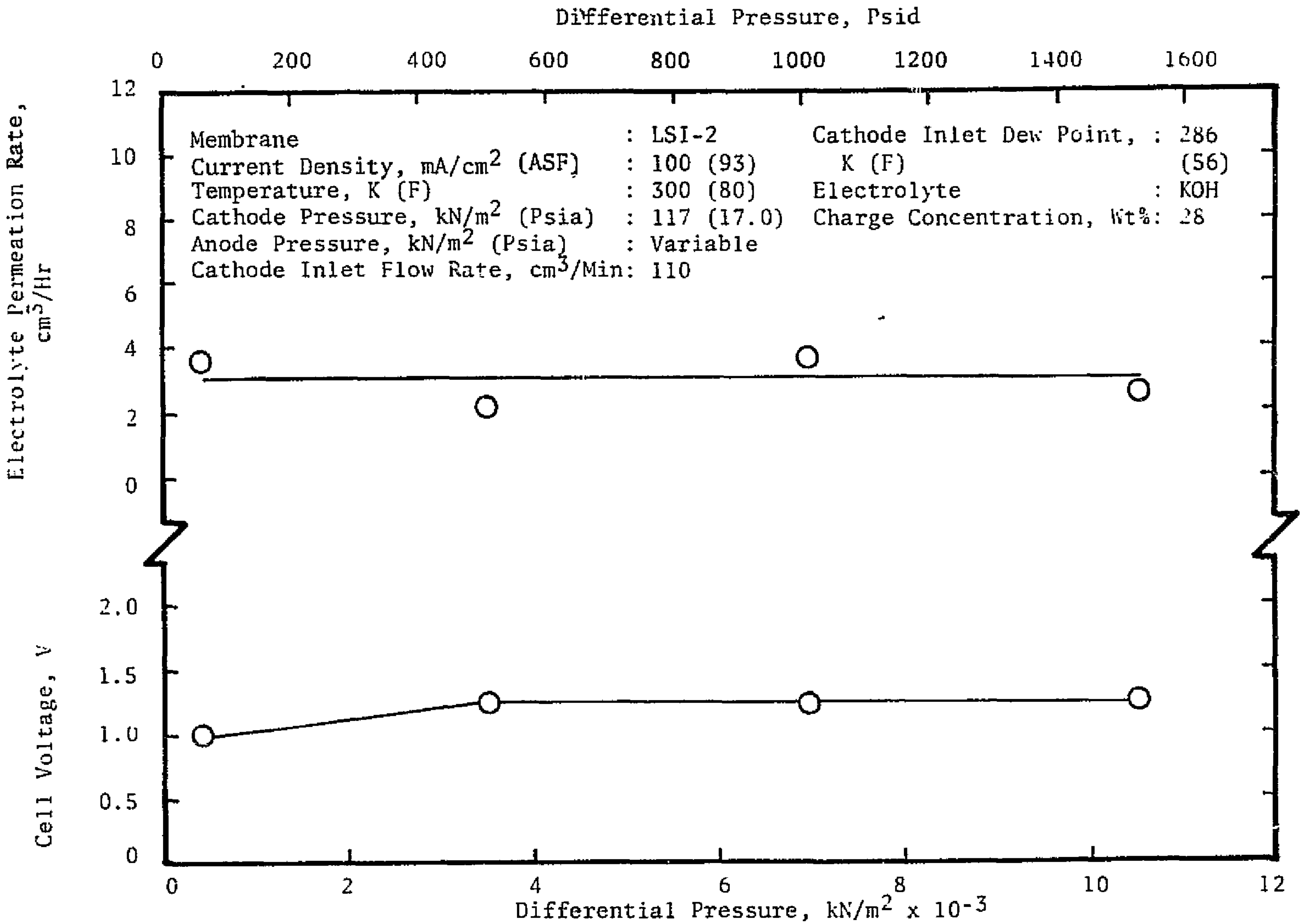


FIGURE 24 CELL VOLTAGE AND ELECTROLYTE PERMEATION RATE VERSUS DIFFERENTIAL PRESSURE

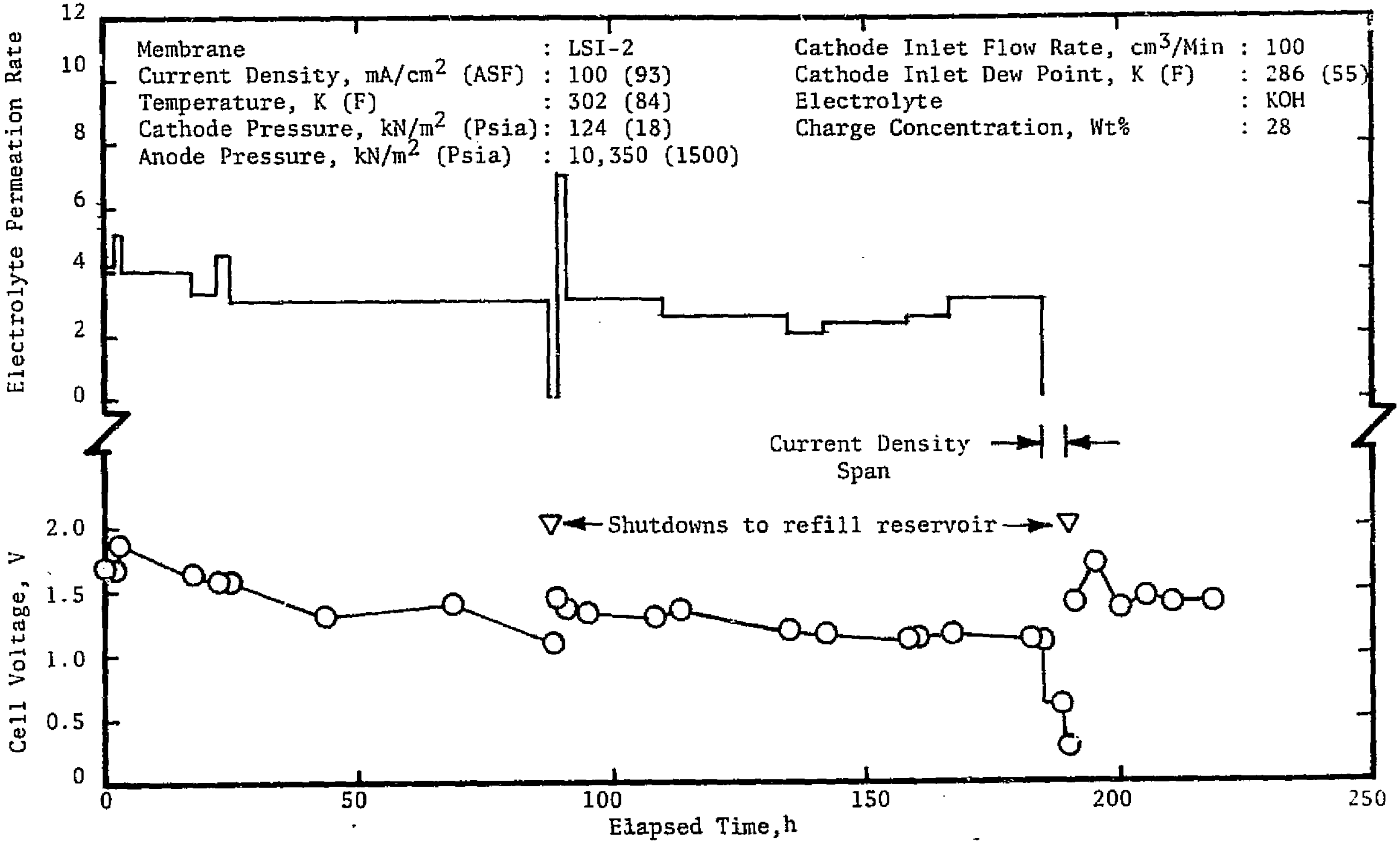


FIGURE 25 DESIGN VERIFICATION TEST (200 HOUR)

## Metallic Parts

Inconel 600, Monel 400, brass, and Ni are considered as the most suitable metals for high pressure $O_2$ systems. Stainless steel, once ignited, burns readily in a high pressure $O_2$ atmosphere and is therefore only marginally acceptable. Brass is not compatible with KOH.

All metallic parts of the cell are Inconel 600 except the anode cavity spacer, cathode support and compression ring which are Ni, and the cell housing which is 316 stainless steel. The cell housing, however, does not contact high pressure $O_2$. Test facility valves and pressure regulators are primarily stainless steel and were selected based on cost and availability considerations. All components are connected by Inconel tubing to prevent a flame from propagating to the stainless steel component from an ignition source, thereby minimizing the potential safety hazard.

## Nonmetallic Parts

Polymers and elastomers used for sealing materials are severly limited by compatibility with high pressure $O_2$ to fluorinated hydrocarbons. Teflon and other polytetrafluorethylenes are very suitable for use in both KOH and high pressure $O_2$ environments. Teflon, however, is susceptible to cold flow and therefore must be properly confined when used as a sealing material. The use of Teflon filled with a more suitable elastomer can alleviate this problem. Kel-F and other polychlorotrifluoroethylenes are also compatible with high pressure $O_2$ and KOH and are much less susceptible to cold flow. Viton is only marginally acceptable with high pressure $O_2$ since $O_2$ is highly absorbed by Viton. Absorbed $O_2$ can cause radical dimensional changes and even cracking or splitting of the material. In short-term usage and minimal exposure to high pressure $O_2$, Viton can be used since failure would result in gas leakage which represents no immediate safety hazard. Compatible lubricants used in regulators and on sealing threads are Krytox oils and greases, and Teflon tape without adhesives.

## Oxygen Backdiffusion Analysis

The mechanism of $O_2$ backdiffusion through the EOC membrane is described by Fick's Law of Diffusion:

$$N = D\frac{dc}{dx} \tag{8}$$

where

$N$ = the flux of $O_2$

$D$ = the diffusion constant for $O_2$ in the membrane

$c$ = the $O_2$ concentration in the membrane

$x$ = the distance through the membrane

Because no $O_2$ is reacted within the membrane, the flux is the same at every distance x through the membrane. The diffusion constant, D, is assumed to be constant with respect to x. Therefore, dc/dx must be constant with respect to x, and may be written as $\Delta c/\Delta x$. The concentration of $O_2$ in the membrane may be described in terms of pressure by Henry's Law:

$$p = Hc \tag{9}$$

where

p = the partial pressure of $O_2$ in equilibrium with concentration c in the membrane

H = Henry's Law constant

Substituting Equation 9 into Equation 8 to eliminate c, and writing d as Δ,

$$N = \frac{D}{H} \frac{\Delta p}{\Delta x} \tag{10}$$

indicates that the $O_2$ flux or backdiffusion is proportional to the differential pressure and inversely proportional to the thickness. The membrane selected should therefore have a low $O_2$ diffusivity. The $O_2$ backdiffusion problem for a given membrane can be minimized by increasing membrane thickness.

## CONCLUSIONS

A program to evaluate an EOC as an $O_2$ compressor for recharging high pressure EVA $O_2$ bottles has successfully been completed. Two EOC cell design concepts were evaluated for the application. The first design utilizes a customized electrolyte membrane to (a) withstand the differential pressure required to compress the $O_2$, (b) prevent $O_2$ backdiffusion, and (c) maintain cell moisture balance. The second design maintains cell moisture balance by using an electrolyte reservoir to replenish electrolyte that permeates from the high pressure side (anode) to the lower pressure side (cathode) of the cell. Both designs would compress $O_2$ in a single stage from 345 $kN/m^2$ (50 psia) to 20,700 $kN/m^2$ (3000 psia). Neither design is competitive with high pressure $O_2$ storage for resupply periods $\leq$105 days but both designs are preferrable to high pressure $O_2$ storage for resupply periods $\geq$150 days. Considerable development time and funds, however, would be required to develop either EOC design for EVA application.

The customized membrane approach represents the simplest EOC system design having lowest system weight and volume. Such a customized membrane design, however, is not compatible with present technology levels. Fifteen membranes were developed using available membrane fabrication techniques to customize a membrane for the application. None of the membranes fabricated, however, met the rigid water permeability and resistance specifications required for EOC operation. When tested, moisture imbalance would cause the anode to lose electrolyte and the cathode to flood. Cell operation was limited to short periods of time (less than 24 hours). It was concluded, therefore, that present membrane fabrication techniques were not adaptable for the application.

The second design concept eliminated the strict water permeability and membrane resistance specifications by utilizing an electrolyte reservoir to maintain cell moisture balance. As the cell operates, electrolyte permeates from the high pressure side to the low pressure side. The low pressure feed $O_2$ stream leaving the cell and the high pressure $O_2$ stream are separated from the electrolyte in two gas/liquid separators. Cell performance utilizing the reservoir approach was adequate for the application and only minimal cell technology improvements are required. Cell voltages of $\leq$1.0V at 100 mA/$cm^2$ (93 ASF) and cell operation for $\geq$200 hours were obtained using the electrolyte reservoir approach. In simplifying the cell specifications, however, system peripheral components became more complex. A gas/liquid separator would be required that operates at 20,700 kN/$m^2$ (3000 psia) and is perfectly selective to $O_2$ such that no electrolyte vapor or water vapor is allowed to reach the high pressure $O_2$ bottle. Available gas/liquid separation technology is insufficient for this application and a customized gas/liquid separator would have to be developed.

## RECOMMENDATIONS

Further EOC work should be given a low priority unless the development of an EOC is critical at this time. Additional membrane work is required to develop a simple EOC system without an electrolyte reservoir. Current membrane technology, however, is not applicable and such a development would be expensive and lengthy (if even possible). Additional work on an EOC system with an electrolyte reservoir would require the development of a 20,700 kN/$m^2$ (3000 psia) $O_2$ gas/aqueous KOH, gas/liquid separator that is perfectly selective to $O_2$. The development of such a gas/liquid separator is also likely to be an expensive and lengthy development.

Prior to beginning a membrane or gas/liquid separator development specifically for EOC application, an evaluation of alternate $O_2$ compression concepts is recommended to determine the best technology basis, high pressure and cryogenic $O_2$ storage versus direct mechanical or electrochemical compression techniques, to refill spent EVA high pressure $O_2$ bottles.

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## APPENDIX 1 EQUIVALENT WEIGHT CALCULATION ASSUMPTIONS FOR EOC

1. An EVA mission is defined as two men for four hours of activity at an average metabolic $O_2$ consumption rate of 0.84 kg/day (1.84 lb/day).

2. An $O_2$ bottle recharge is averaged over the entire resupply period, i.e., continuous operation.

3. The equivalent weight penalty for using power is 268 kg/kW (591 lb/kW).

4. The equivalent weight penalty for heat rejection directly to spacecraft liquid coolant is 84 kg/J/s (184 lb/kW).

5. All heat is rejected to spacecraft liquid coolant, i.e., negligible heat loss to ambient through tubing or components.

6. Optimum mechanical and instrumentation hardware design. The EOC system weight, then, is a function of EOC performance, i.e., cell voltage, current and number of compression stages.

7. No spare $O_2$ bottles are required for comparative purposes since approximately the same number of backup $O_2$ bottles would be required for both an EOC system and simple bottle high pressure $O_2$ storage.

8. Spares, packaging, and tubing are estimated to be 100% of the basis system weight.

9. The amount of $O_2$ that remains in the bottles is greater than or equal to the current of gas required to pressurize system lines to the final bottle pressure, i.e., there is no inefficiency in pressurizing system lines.

10. Power conversion efficiency for module power is assumed to be 91%.

11. Oxygen storage pressure is 20,700 $kN/m^2$ (3000 psia).

12. Oxygen bottle weight is estimated at 1.0 kg/kg $O_2$ which includes bottle storage accessories.

13. Oxygen generation penalty is 63 kg/kg $O_2$/day for water electrolysis equivalent weight penalties for power, heat rejection, and increased weight of extra cells required.

## APPENDIX 2 TEST FACILITY AND OPERATIONAL SAFETY GUIDELINES FOR THE ELECTROCHEMICAL $O_2$ CONCENTRATOR

Aside from the safety problems of handling high pressure gases, $O_2$ creates an even more severe safety problem in that it provides, especially at high pressures, an abundant supply of oxidizer so that once an ignition source is obtained, materials (e.g., steel) not ordinarily flammable can be rapidly and violently consumed.

The three requirements for a fire are an oxidizer, fuel and ignition source. In high pressure $O_2$ systems the oxidizer is abundantly present and, as a result of the high pressures, materials such as stainless steel can fuel the oxidation reaction. Hence, the methods of preventing accidents consist of selecting materials of construction that are poor fuels in high pressure $O_2$ and elimination of sources of ignition. Five modes of ignition have been identified.

1. Electric arc - short circuit
2. Single impact - mechanical
3. Abrasion - fluid-borne particles
4. Resonance ignition
5. Fracture - new metal surfaces

The safety design criteria which follow are steps which have been designed to eliminate the ignition sources listed above.

A. <u>Safety rules to be adhered to when utilizing $O_2$ gas cylinders</u>

1. Never use oily rags or tools on $O_2$ cylinders. Wear polyethylene (or equivalent) gloves when connecting $O_2$ cylinders.

2. After removing the valve protection cap open the valve slightly for an instant in order to clear the opening of particles of dust [illegible]rt, being careful to point the valve opening away from the body. [illegible]onnect the $O_2$ pressure regulator to the $O_2$ cylinder. Never use $O_2$ from a cylinder without reducing the pressure through a suitable regulator.

3. Make certain that the threads on regulators or other unions correspond to those on the cylinder valve outlets. Never force connections which do not fit. Never interchange regulators, hoses or other appliances with similar equipment intended for use with other gases.

4. After attaching the regulator and before the cylinder valve is opened, see that the adjusting screw of the regulator is released and that the $O_2$ has been drained from the regulator.

5. Be sure that all connections are gas-tight and remain so.

6. Open cylinder valve slowly. Stand to one side and away from the regulator gauge faces when opening the cylinder valve. Never use

wrenches or tools except those provided or approved by the manufacturer. Avoid the use of a wrench on valves equipped with hand wheels. Never hammer the valve wheel in attempting to open or close the valve. If valves cannot be opened by hand, notify the supplier.

7. Never permit $O_2$ to enter the regulator suddenly. Open the cylinder valve slowly.

8. Always close the cylinder valve when work is finished. Be sure the cylinder valve is closed and all gas is released from the regulator before removing the regulator from the cylinder.

9. Oxygen cylinders in storage shall be separated from fuel gas cylinders and combustible materials (especially oil or grease) a minimum distance of twenty feet or by a noncombustible barrier at least five feet high having a fire resistance rating of at least one-half hour.

10. Gas shall not be transferred from one cylinder to another or mixed with another gas in a cylinder.

11. If cylinders are found to have leaky valves or fittings which cannot be stopped by closing of the valve, the cylinders shall be taken outdoors away from sources of ignition and slowly emptied. Leakage through a valve outlet may be temporarily stopped by attaching a regulator. The owner of the cylinder should be advised so that it may be repaired when returned to him. It is advisable to tag the cylinder in such a way as to indicate the nature of the trouble.

12. High-pressure $O_2$ cylinders shall be used only with pressure-regulating devices approved and marked for use with $O_2$.

13. Cylinders should never be allowed to reach a temperature exceeding 325K (125F) because of the rise in pressure in the cylinder with increasing temperature. Cylinders should, therefore, never be stored near furnaces, radiators or any other source of heat.

14. Oxygen cylinders should be protected from abnormal mechanical shock which is likely to damage the cylinder, valve or safety device.

15. Valve protection caps, when provided, should always be in place, hand tight, except when cylinders are in use or connected for use.

16. Cylinders should be protected from the tampering of unauthorized individuals.

17. Storage should be planned so that cylinders may be used in the order in which they are received from the supplier.

18. Empty and full cylinders should be stored separately. Empty cylinders should be marked to avoid confusion.

19. Cylinders should be stored in a dry, well-ventilated location.

20. Cylinders should not be exposed to continual dampness and should not be stored near salt or other corrosive chemicals or fumes. Rusting will damage the cylinders and may cause the valve protection caps to stick.

21. Valves should be closed on all empty cylinders in storage.

22. Oxygen cylinders should be secured in an upright position so they cannot be knocked over.

B. Safety rules for system assembly and operation

1. Do not store components or parts to be used in the system near flammable material, especially oil, grease or any other readily combustible substance.

2. Never permit oil, grease or any other readily combustible substance to come in contact with $O_2$ valves, regulators, gauges and fittings. Oil on $O_2$ may combine with explosive violence if ignited. Degrease all tools before using on $O_2$ components.

3. Never lubricate $O_2$ valves, regulators, gauges or fittings with oil or any other combustible substance. The only acceptable lubricant for $O_2$ systems is Krytox 240.

4. Do not handle $O_2$ apparatus with oily hands or gloves.

5. Oxygen piping and fittings for pressures in excess of 4830 $kN/m^2$ (700 psig) shall be stainless steel or nonferrous metal. Inconel or Monel are recommended.

6. Piping systems shall be tested and proved gas-tight at 1-1/2 times the maximum operating pressure. The material used for testing $O_2$ lines shall be oil-free and nonflammable. Flames shall not be used to detect leaks. Oil-free $N_2$ or oil-free Ar are recommended.

7. Test facilities shall be located in a clean, well-ventilated area with sufficient space for testing.

8. All systems shall be constructed such that they are fail-safe whenever feasible, that is, interruption of supply gases, fluids or voltages should not by itself cause a critical condition.

9. All wiring and insulation and accessories in a system utilizing an $O_2$-rich atmosphere shall be of teflon composition. Care shall be exercised in the design, fabrication and installation of wire runs in bundles to avoid crimping, abrasion and mechanical pressure.

10. Specific attention shall be directed to providing adequate fusing and/or current limiters to electric circuits to minimize fire hazards.

11. Systems designed for use with $O_2$-rich atmospheres shall be free of hydrocarbon contamination.

12. All components in the high pressure $O_2$ lines must be cleaned for $O_2$ service. The vendor performing the cleaning operation will certify that the parts have been cleaned and meet the specification cited. The cleaned parts will be shipped and stored in sealed polyethylene bags.

13. Avoid venting or disposal of $O_2$ near electrical systems or other ignition sources capable of initiating a fire.

14. Ensure controlled valve operation of $O_2$ valves to limit generation of heat due to gas compression.

15. Verify that $O_2$ line pressures have been relieved before disconnecting $O_2$ gas lines.

16. Maintain a maintenance log for the high pressure $O_2$ test stand so that the operations performed can be evaluated for their impact on safety.

17. The high pressure $O_2$ system must be electrically grounded.

18. Flame restrictors must be utilized in stainless steel high pressure $O_2$ lines.

19. Avoid high flow rates of $O_2$ gas which may accelerate contaminant particles and cause localized temperature rise when the particle bombards a tube or component wall.

20. Do not open sealed polyethylene bags containing system parts until just prior to incorporating into the system. Take special precautions to keep the individual components capped and the system closed off from the atmosphere while assembling.

21. System assembly shall be conducted in a clean lab area where there are no drafts or excessive activity.

22. Assemble the system in such a way that the high pressure $O_2$ parts are remote from the control area and the electronics.

23. Minimize the closure force on valves or other operational devices in high pressure $O_2$ to avoid ignition by impact. Motor-driven ball valves are acceptable, solenoids are not.

24. Strive to eliminate all dead end cavities in the $O_2$ system. These could be exposed to rapidly fluctuating pressure fields generated by unstable systems of shock waves. The temperature can climb to dangerous levels in the gas near the end of such cavities.

25. Select materials of construction that are compatible with high pressure $O_2$.

26. The system will be filled with oil-free Ar and sealed off when not in operation. This will prevent airborne contaminants from entering the system when not in use.

## C. Personnel protection features

Features and procedures which are required for personnel protection include:

1. Safety inspections to ensure that the safety guidelines listed in A and B above are in compliance.
2. All employees working in the area while the high pressure $O_2$ system is in operation will be required to wear the safety equipment designated by the Safety Engineer.
3. The system design will incorporate protection features such as rupture discs and instrumentation to shut off the $O_2$ supply in the event high pressures or temperatures are sensed. The instrumentation provided for personnel safety will be redundant.
4. All monitoring of system performance during operation at $O_2$ pressures $\geq$13800 kN/$m^2$ (2000 psia) will be accomplished from a remote location or the system will be appropriately barricaded.
5. All equipment will be grounded to protect individuals from electrical shock and to minimize possible ignition sources.
6. An inert gas purge will be included to dilute the $O_2$ in the event a system shutdown occurs.