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INORGANIC ION EXCHANGE MEMBRANE FUEL CELL

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QUARTERLY PROGRESS REPORT SM-46221-Q3

INORGANIC ION EXCHANGE MEMBRANE FUEL CELL

by

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prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

April 1965

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1.0 SUMMARY AND CONCLUSIONS

The most significant accomplishments during this, the third quarterly report period, are the establishment of a fuel cell operational capability at temperatures as high as 148°C and performance levels in the range of 50 ma/cm^2 at 0.65 volts and 0.72 volts at 30 ma/cm^2. This performance held at 300 hours of continuous operation at essentially the same level of voltage and current. Fuel cell studies were performed in both the old Astropower fuel cell and a newly optimized compact unit, the design of which was based on practical experience as well as heat and mass transfer considerations.

The following results were specifically obtained:

(1) Membrane thickness had little if any effect on fuel cell performance up to about thickness levels $\approx$ 0.75 mm for the specific cell configuration employed.

(2) Fuel cell performance is relatively independent of temperature over the temperature interval of 65°C - 120°C. Steady performance is obtained at temperatures above and below this range at somewhat lower levels. An explanation for the constancy of fuel cell performance in the 65° - 120°C range is that membrane resistivity remains constant over this temperture range. This is borne out by recent independent laboratory measurements of the resistivity-relative humidity-temperature relationships for such membrane systems[2].

(3) Impregnation of platinum black into the membrane structure by a sintering technique is conducive to the stability of fuel cell performance. If the depth of impregnation of the platinum black is reduced to 10% of the overall membrane thickness, enhanced performance results.

(4) Palladium is as effective as platinum in promoting fuel cell performance.

(5) Increasing the concentration of impregnated platinum black beyond 20% does not appear to affect performance.

(6) Increase of gas exposed areas of the wafer backup plate in the compact fuel cell tests enhances fuel cell performance. The highest performance of the program to date was achieved in this manner.

(7) The relationships in (1) - (5) are essentially similar to those for the previously employed nonoptimized fuel cell.

The current and future programs concerned with attempted improvement of membrance-catalyst and membrane-catalyst-electrode configurations are presented. Waterproofing and water removal by wicking are now in progress as well.

At this point it appears that enhancement of performance to the 0.75 volt and 50 ma/cm^2 level is in the offing for the Astropower zirconium phosphate-"Zeolon H" membrane fuel cell without any membrane reformulation.

2.0 INTRODUCTION

By the mid-point of this program, it was evident that the original target performance established for this program of 25 - 50 ma/cm^2 at 0.5 v. for 300 hours could be met by the use of zirconia-phosphoric acid-"Zeolon H" membranes having transverse strengths of 5,000 psi or greater. Then, it was decided to attempt to enhance the performance by optimization of the fuel cell design as well as the membrane-catalyst-electrode configuration while keeping the membrane composition constant. Since there was no restriction on fuel cell operational temperature, it became important to determine the temperature for optimum performance.

During this report period, it was possible to achieve a 60% improvement in fuel cell performance by two independent approaches. In the first approach, a compact fuel cell was designed on the basis of practical operating conditions and substantiated by heat and mass transfer analysis. In the second approach, various exploratory tests had suggested the design of a membrane-catalyst configuration wherein the catalyst was impregnated in the membrane to only a very small depth.

Simultaneously, it has been established that hydrogen-oxygen fuel cells can be operated at temperatures as high as 148°C. Temperature does not appear to have a significant effect on fuel cell performance in the 65° - 120°C range because the conductivity of the membrane remains essentially invariant with increasing temperature under these conditions. The details of these studies are given in this report as well as indications that still higher performance levels could be attained. A new performance target level of 50 ma/cm^2 at 0.7 volts was set.

3.0 EXPERIMENTAL PROCEDURE, RESULTS AND DISCUSSION

3.1 Fuel Cell Tests

Fuel cell testing was intensified in efforts to establish the parameters for optimum fuel cell performance. The results of the most significant tests (23) performed during this report period are compiled in Table I in order of increasing test temperatures. All tests were performed at a constant current density of 30 ma/cm^2. Two types of test cells were used. The first type was the standard Astropower fuel cell described in References (1) and (2). The second type was an optimized compact fuel cell described in References (3) and (4). The results obtained will now be discussed.

3.1.1 Standard Fuel Cell Tests

All tests were performed at constant current density of 30 ma/cm^2. Test (1) was performed at 25°C in order to help establish the relative influences of temperature and membrane thickness on fuel cell performance. Tests (2) and (3) and (18) are in the same category as well. In all instances, the experimental conditions were standard, as described in References (1) and (2). The results at 65°C (Tests 2 and 3) indicate that thickness levels within the 0.2 - 0.75 mm range have little or no bearing on fuel cell performance except possibly for fuel cell longevity. The results of Test (18) are comparable to those of Tests (2) and (3), demonstrating that performance over the temperature interval of 65 to 100°C is unaffected by temperature. Howevever, the lower performance at 25°C (Test 1) definitely shows that poorer performance is related to increased fuel cell resistance. It is our belief that the higher fuel cell resistance at 25°C is due to inadequate water product removal through lower vaporization occurring at that temperature. Appropriate water proofing will probably correct this problem.

Test (4) was conducted with a membrane which had platinum deposited on it by a chemical procedure involving the reduction of chloroplatinic acid. The performance level was no better than that of Test (2) without platinum deposited on the membrane.

Test (5) was initiated during the previous quarter and mentioned in Reference (2). It had been observed in previous hydrolysis

studies that this membrane lost 5.5% of its total weight through release of unincorporated phosphoric acid upon being immersed in distilled water at 75°C for two hours. Some evidence had indicated the possibility of small gradual losses in phosphoric acid from the membrane during actual fuel cell operation. Therefore, it was of interest to establish whether or not this affected fuel cell performance. Test (5) was performed on the membrane which had been soaked for two hours at 75°C followed by a drying period for two hours at 500°C. The performance of this test is comparable to that of Test (2) and, for a longer duration. Therefore, it can be concluded that prior removal of unincorporated phosphoric acid from the membrane by soaking in water is not at all deleterious to fuel cell performance. Undoubtedly, this applies to membrane behavior during fuel cell operation as well.

Tests (9), (10), (11) and (12) were performed with platinum black being impregnated into both outer one-third layers of the membrane in the following manner. A weighed amount of platinum-bearing membrane material (10, 20, 30 or 40% platinum by weight, the remainder being C200B material) is placed in a pressing die, followed by a layer of the C200B membrane material and a second layer of the catalyst-bearing material. The top punch is then inserted and the assembly (two-inch diameter) is pressed at 15 tons total load. After pressing, the composites are placed on flat, smooth refractory plates and sintered in air for two hours at 500°C. After cooling to room temperature, the composite membrane-catalyst wafer was impregnated with 85% phosphoric acid, oven dried at 120°C and finally sintered at 500°C for two hours. The phosphoric acid treatment was repeated twice. A photograph depicting the three layers, given originally in Reference (1), is shown again in Figure 1.

Test (9) is actually a continuation of Test (2), Table I of Reference (2). Although Test (9) containing the least amount of platinum, was the most durable, its performance never reached the operational levels of the other tests. The tests with more platinum were about the same in performance level as that of Test (2), without platinum impregnated in the outer one-third layers. However, it will be shown below that platinum impregnated into the membrane does offer advantages at higher temperatures.

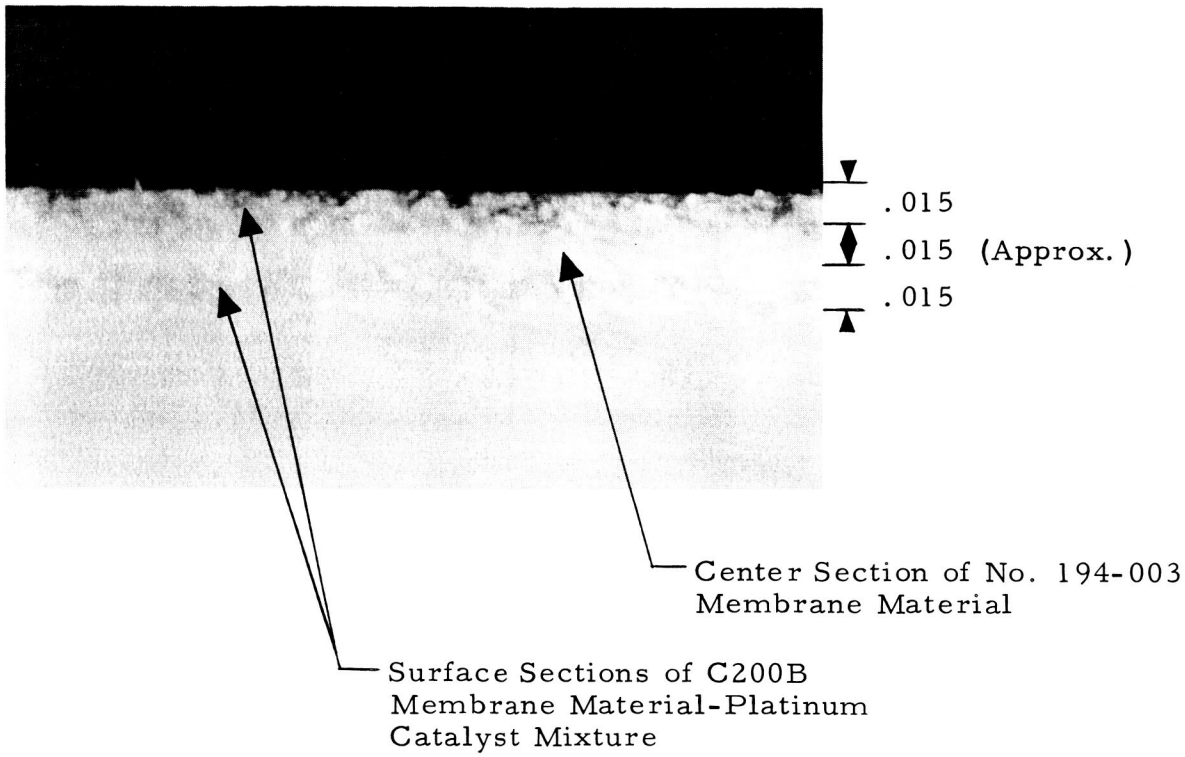

Figure 1. Fuel Cell Membrane - Catalyst Composite

It was noted at this point that the fuel cell performance level tended to remain essentially constant with increasing concentration of platinum past 20% in the outer one-third layers of the membrane. This prompted an effort with a membrane in which 20% platinum was impregnated in both outer one-tenth layers by the processing procedures described in Reference (2). Therefore, the middle layer of the membrane consisting of nonimpregnated C200B material constituted 80% of the total membrane. The results of the evaluation of this membrane are given in Test (13). It can be seen that there has been a significant enhancement in performance through this type of membrane-catalyst formulation procedure. It may be reasoned that the active area for electro-catalysis is at the surface of the membrane only. Any further penetration of the membrane by the catalyst does not provide additional sites for the appropriate catalytic reaction. In addition, the possibility of electrical short circuiting is diminished by keeping the catalyst material as close to the surface of the membrane as possible.

Test (17), (19), (22) and (23) had been performed with 20% platinum black in the outer one-third layers of the membrane over the temperature range of 93°C to 148°C. It appears that the performance remains essentially constant over the temperature range of 65 to 120°C, but appears to diminish past 120°C. In this regard, it was not possible to obtain sustained fuel cell operation at temperatures in the range of 90° to 100°C when the non-impregnated C200B membrane was used (Test No. 18). Apparently, the impregnated platinum exerts a stabilizing influence on fuel cell behavior above 65°C.

Test (21) performed with a 10% platinum impregnated membrane at 100°C, does not reach the performance level of that of Tests (19) and (23) containing 20% platinum-impregnated membranes and evaluated at comparable temperatures. A similar correlation exists at 65°C. Of further significance, it is to be noted that the performance of the 10% platinum impregnated membrane at 65°C (Test 9) is essentially equal to that of the same system at 100°C (Test 21).

Test (20) was performed with 20% palladium black impregnated in both outer one-third layers of the membrane. This system

performed as well as the corresponding all-platinum composite in Tests (19) and (20) at 100°C for about 50 hours. Test (20) had been terminated because of sharply diminishing performance after 76 hours. It was observed upon opening the test cell that electrode pores were clogged with clusters of palladium black powder. Evidently, significant amounts of catalyst had become separated from the membrane structure. Since the density of palladium is one-half that of platinum, it is possible that approximately that much less palladium would be required for equivalent effectiveness. This matter is under current investigation.

Test (15) was performed in connection with the program to prepare integrated electrode-catalyst-membrane composites. The 0.1% palladium had been incorporated into the outer layer after impregnation of the membrane with platinum in 20% concentration, followed by the pressing and sintering procedure described above. Although the performance in Test (15) was not particularly outstanding, probably because of the non-porosity of the outer palladium layer, it does demonstrate that it should be possible to prepare integrated electrode-catalyst-membrane systems by this technique. Such integrated assemblies have been prepared, wherein the electrode component has been rendered porous by its being pressed through a silk screen as it is deposited on the membrane. They are to be evaluated in the fuel cell in the near future.

The various correlations of fuel cell performance with membrane thickness, concentration of impregnated catalyst and operation temperature, are depicted in summarizing tables (Tables II - VII inclusive). In these tables, the fuel cell resistances as calculated from polarization data obtained at the indicated time interval of fuel cell operation, are used for correlative purposes in the following manner.

Table II shows that for the nonimpregnated C200B membrane, the resistance is unaffected by membrane thickness up to 0.75 mm. However, beyond that point, resistance increases slightly with increasing thickness.

Table III demonstrates the effect of platinum concentration in the outer one-third layers of the platinum-impregnated C200B

membranes on fuel cell resistance at 65°C. Beyond 20% platinum concentration in the two outer one-third layers, there is no further reduction in fuel cell resistance.

Table IV shows the effect of 10% platinum with that of 20% platinum in the outer one-third layers of the platinum-impregnated membrane at 100°C. It is evident that 20% platinum affords a lower resistance than does 10% platinum, as had been noted at 65°C.

Table V shows the effect of increasing temperature on fuel cell resistance for a 20% platinum-impregnated membrane. There is no change in fuel cell resistance over the temperature range of 65° - 100°C. A slight increase occurs over the temperature range of 100°C to 125°C which becomes more significant as the temperature increases to 148°C.

Table VI shows that palladium impregnated into the C200B membrane is as effective at a similar concentration of platinum at 100°C.

Table VII shows that impregnation of the C200B membrane with platinum to the extent of 20% in the outer one-third layer compared with the nonimpregnated C200B membrane does not lower membrane resistance at 65°C; however, considerable improvement does occur at 82°C.

In connection with performance remaining essentially constant over the temperature region of 65°C to 120°C, the following explanation is offered. Figure 2 (given previously in Reference 1) shows plots of log resistivity versus percent relative humidity determined from independent experiments with the C200B membrane at 70°C, 90°C and 105°C. At higher fuel cell operating temperature the membrane dehydrates slightly causing its resistivity to remain essentially at the same level as at the lower temperature (70°C). This condition exists because resistivity changes little with variation in relative humidity at 70°C whereas, resistivity decreases significantly with increasing relative humidity at higher temperatures (105°C).

3.1.2 Compact Fuel Cell Life Tests

Two compact fuel cell units were operated during this report period, the design of which can be seen in Figure 3. A photograph of

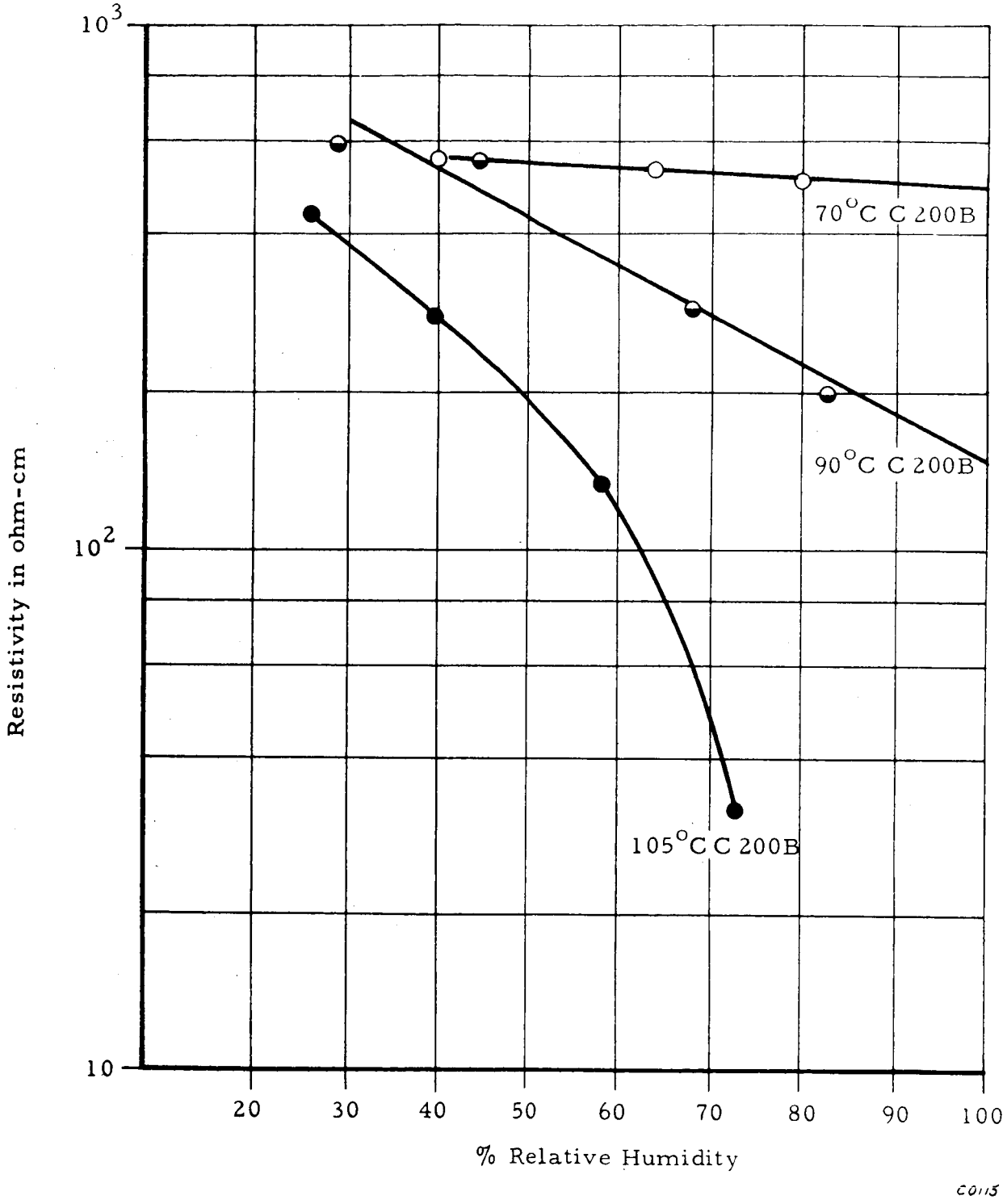

Figure 2. Log Resistivity Versus Percent Relative Humidity for the C200B Membrane at 70°C, 90°C, and 105°C

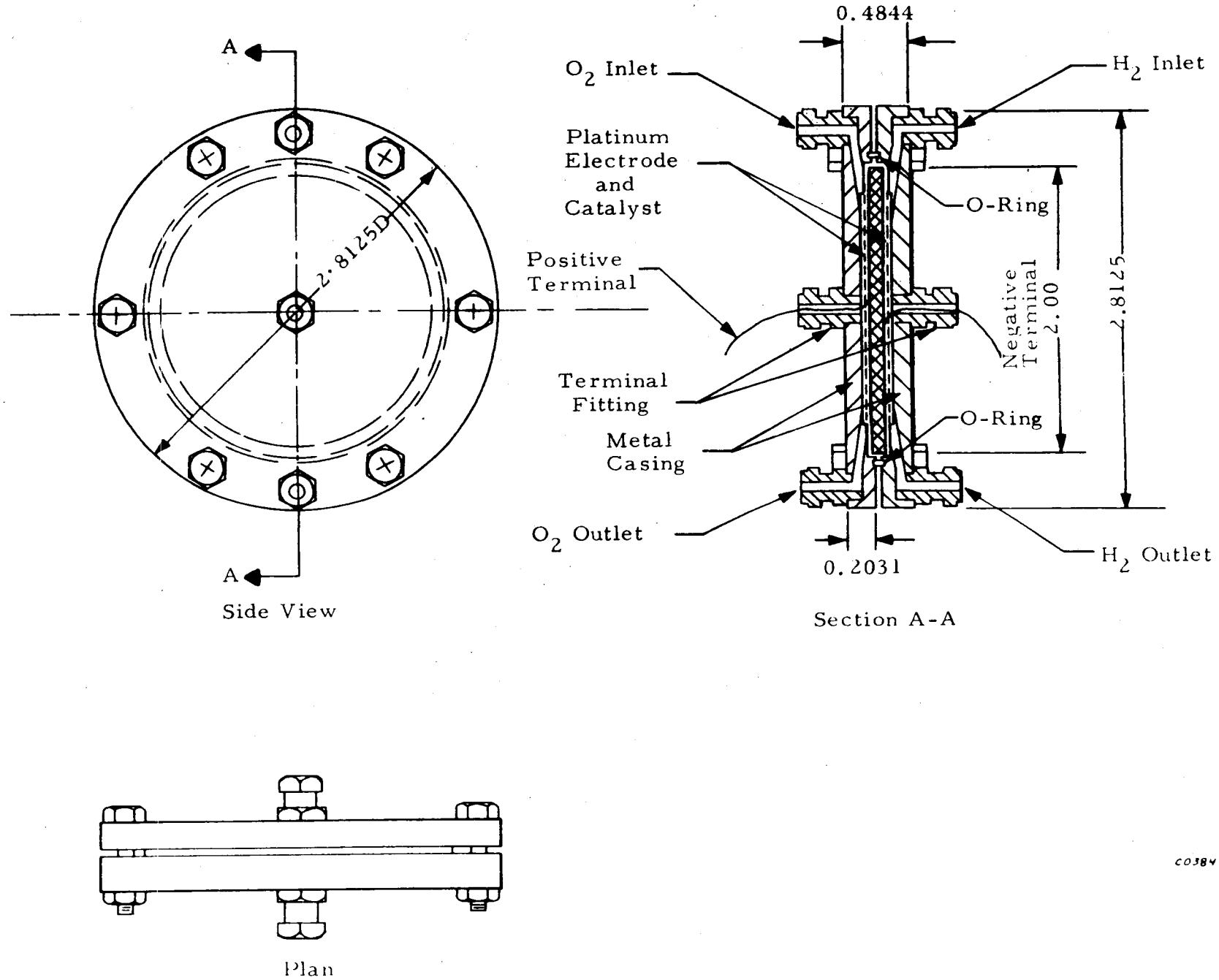

Figure 3. New Astropower Fuel Cell

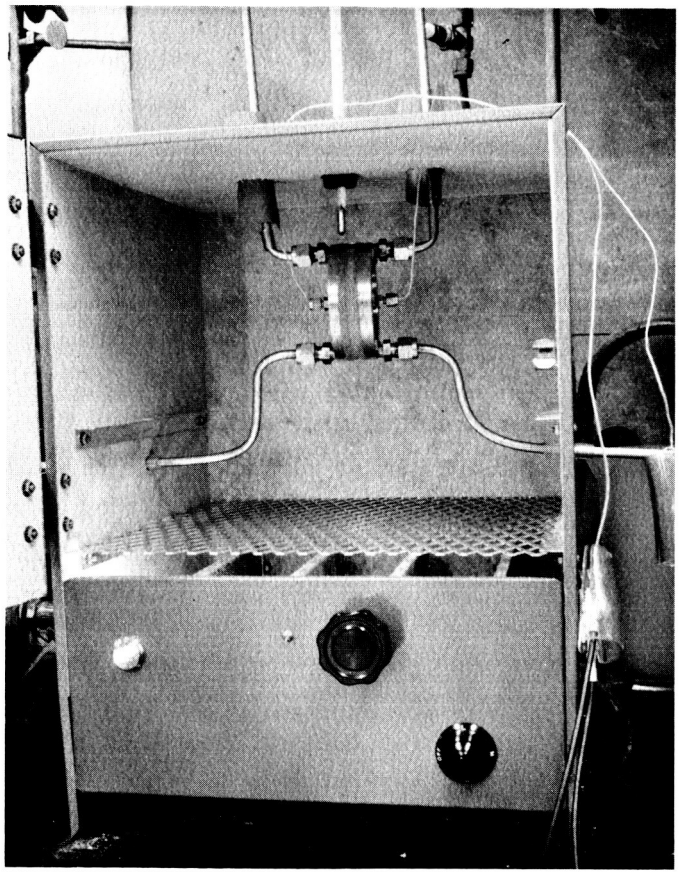

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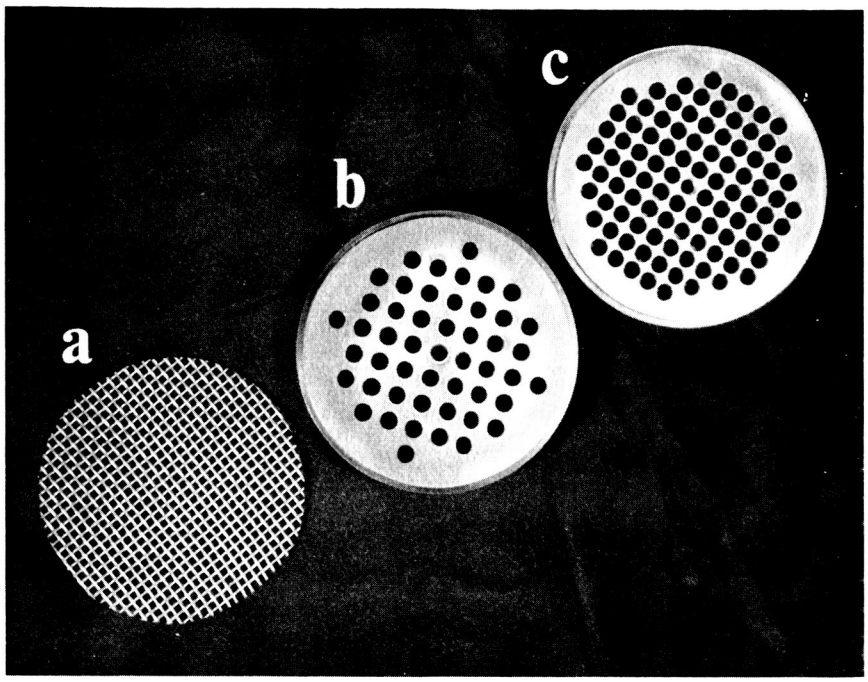
Figure 4. Compact Fuel Cell in an Oven During Operation



C0679

a. 20 Mesh Stainless Steel Screen
b. Forty-four Holes, 1/8 inch in Diameter, in Stainless Steel Back-up Plate
c. Ninety-six Holes, 1/8 inch in Diameter, in Stainless Steel Back-up Plate

Figure 5. From Left to Right, 3 Types of Back-up Plates Used in Tests 6, 7 and 8 Respectively

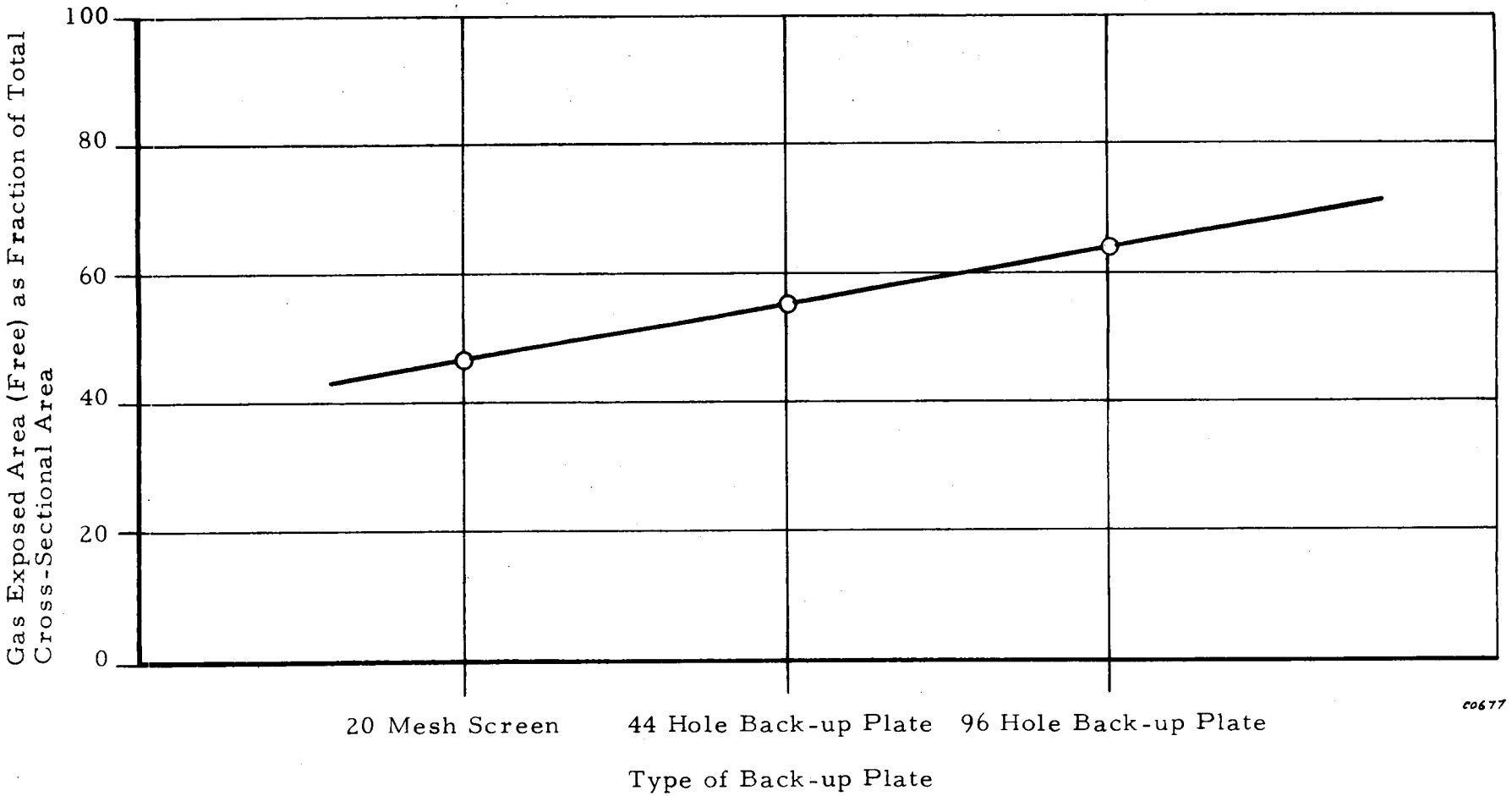


Figure 6. Relationship of Areas of Three Different Types of Back-up Plates Used in the Compact Fuel Cell Design

the compact fuel cell unit as assembled in an oven is shown in Figure 4. The results obtained for tests depicting the most significant results are repeated in Table VIII (initially listed in Table I). This fuel cell was optimally designed using mass and heat transfer analysis and engineering experience. A short description of the mathematical analysis is given in Section 3.2.3 below.

Fuel cell operating resistances are correlated in Table IX for Test (6), (7) and (8) at 65°C in which a 20 mesh screen as a backup plate, a 44 hole backup plate and a 96 hole backup plate were used. Figure 5 shows the three types of backup plates used and Figure 6 relates the gas exposed area as a fraction of the total cross-sectioned area for each back-up plate. From the results of Table IX and Figure 6, it can be concluded that as the gas exposed area was increased the fuel cell performance also increased. This phenomena will occur until contact area becomes critical and the overall cell resistance will rise again.

Test (14) has 20% platinum black impregnated in both outer one-tenth layers. It is demonstrated now that in the 60° - 70°C range, the incorporation of platinum black in a thin layer has no significant beneficial effect on performance.

Tests (17) and (22) were run in order to show how the 20% platinum black membrane in the outer one-third layers performed at the elevated temperatures of 93 ± 2°C and 128 ± 1°C respectively. Characteristically, the resistance increases relatively little with increasing temperature (from 0.43 ohms at 93 ± 2°C to 0.48 ohms at 128 ± 1°C.)

Test (14) involving the same 20% platinum-impregnated membrane affording optimum performance in the standard fuel cell (Test (13), Table I) manifests a somewhat lower performance in the compact fuel cell. There is a good chance that performance superior to that in Test (8) can be achieved if the 96 hole backup were used with this membrane. This matter will be explored further in the near future. Efforts relating to the extent of gas exposed area of the backup plate to the compact fuel cell performance will be continued as well. This, together with continued improvements in water proofing, membrane-catalyst and membrane-catalyst-electrode configurations, should lead to enhancement in fuel cell performance to the 0.70 volt level at ma/cm^2.

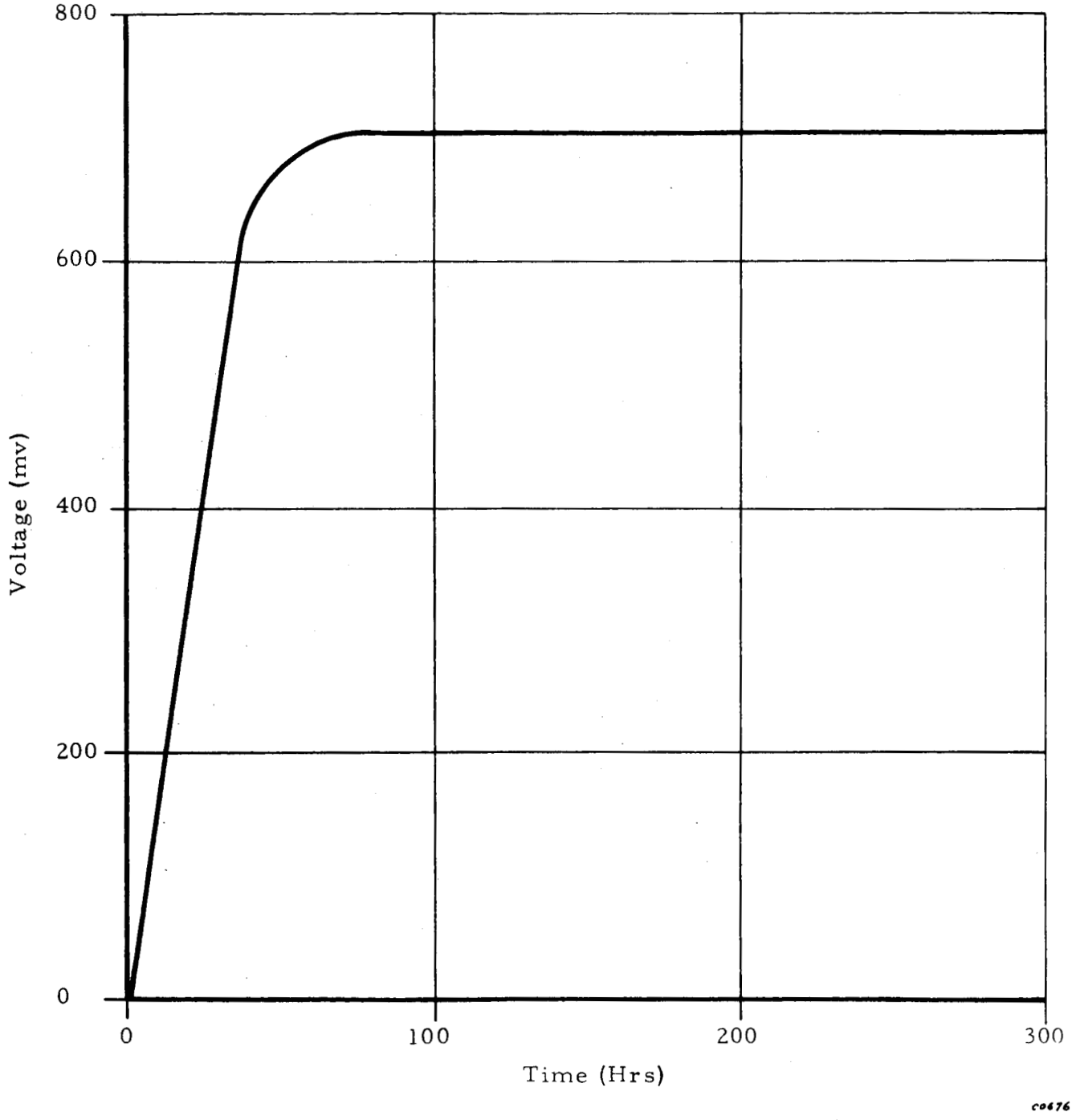


Figure 7. Synopsis of Fuel Cell Life Test at $65 \pm 1^{0}C$, Astropower Zirconium Phosphate-Zeolon-H Membrane Voltage vs Time at Current Density of 30 ma/cm^2

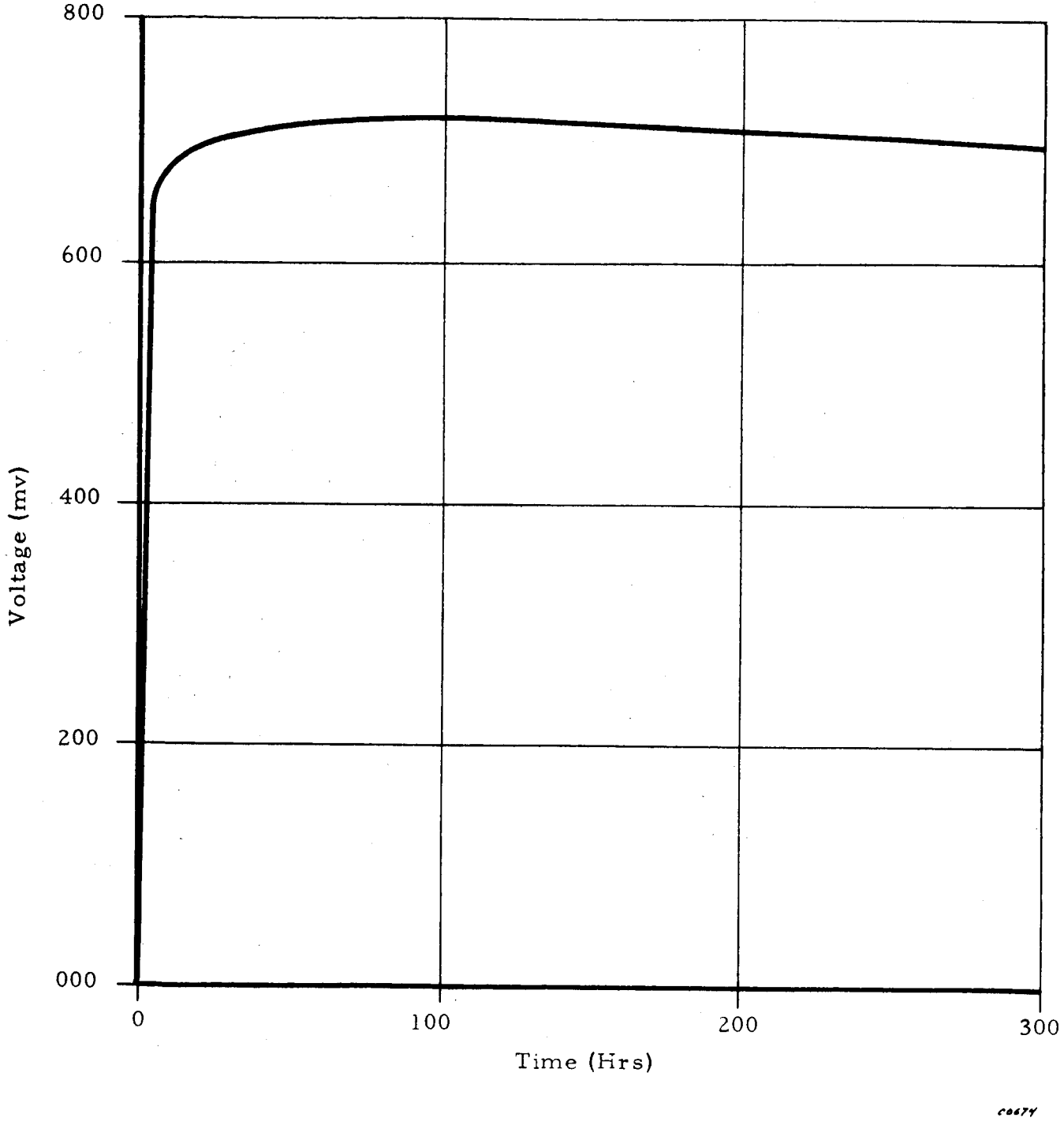


Figure 8. Synopsis of Life Tests at 66±2°C, Test 13, Table I
Voltage vs Time at Current Density of 30 ma/cm^2

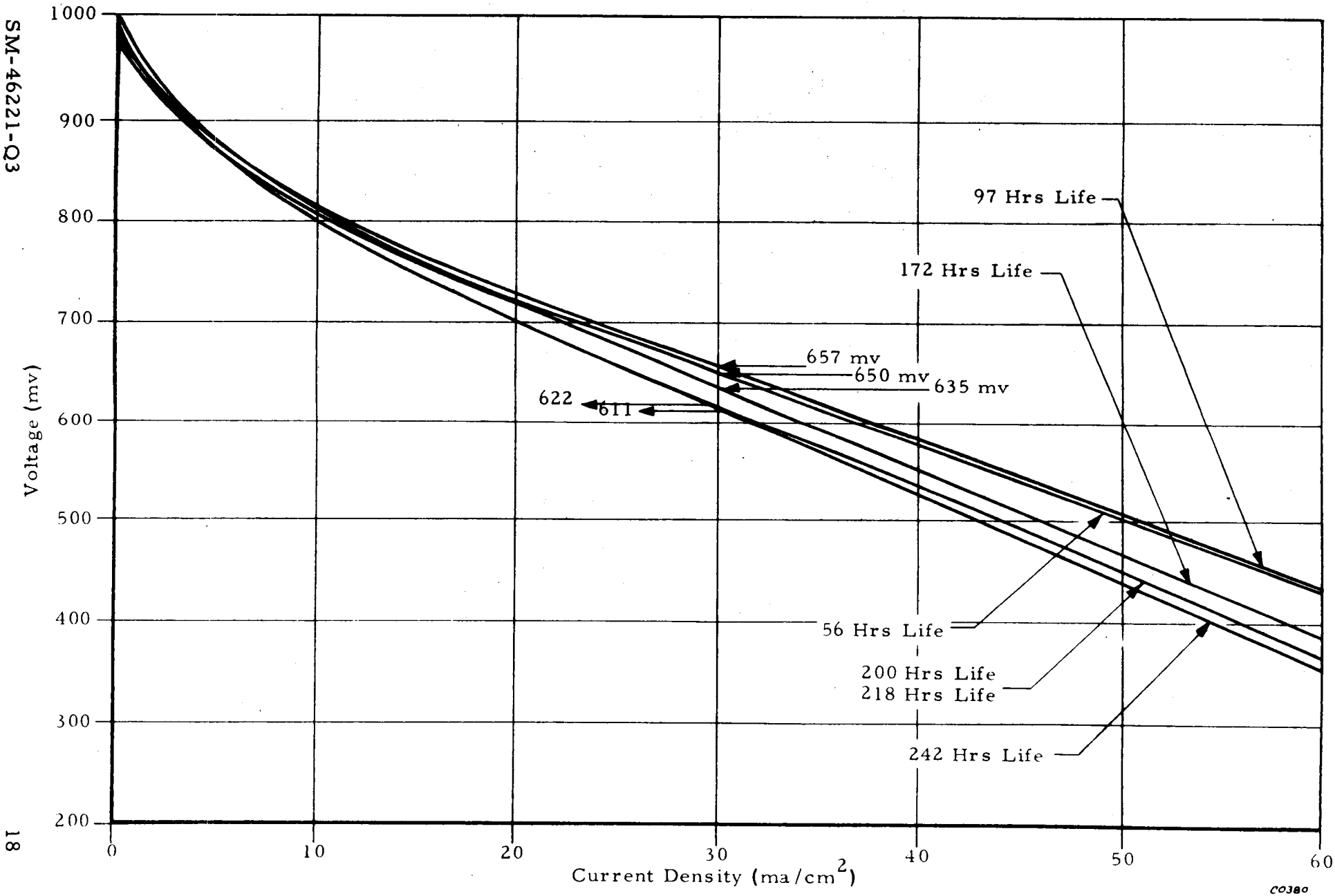


Figure 9. Polarization Curves for Inorganic Membrane Fuel Cell. Test 5, Table I

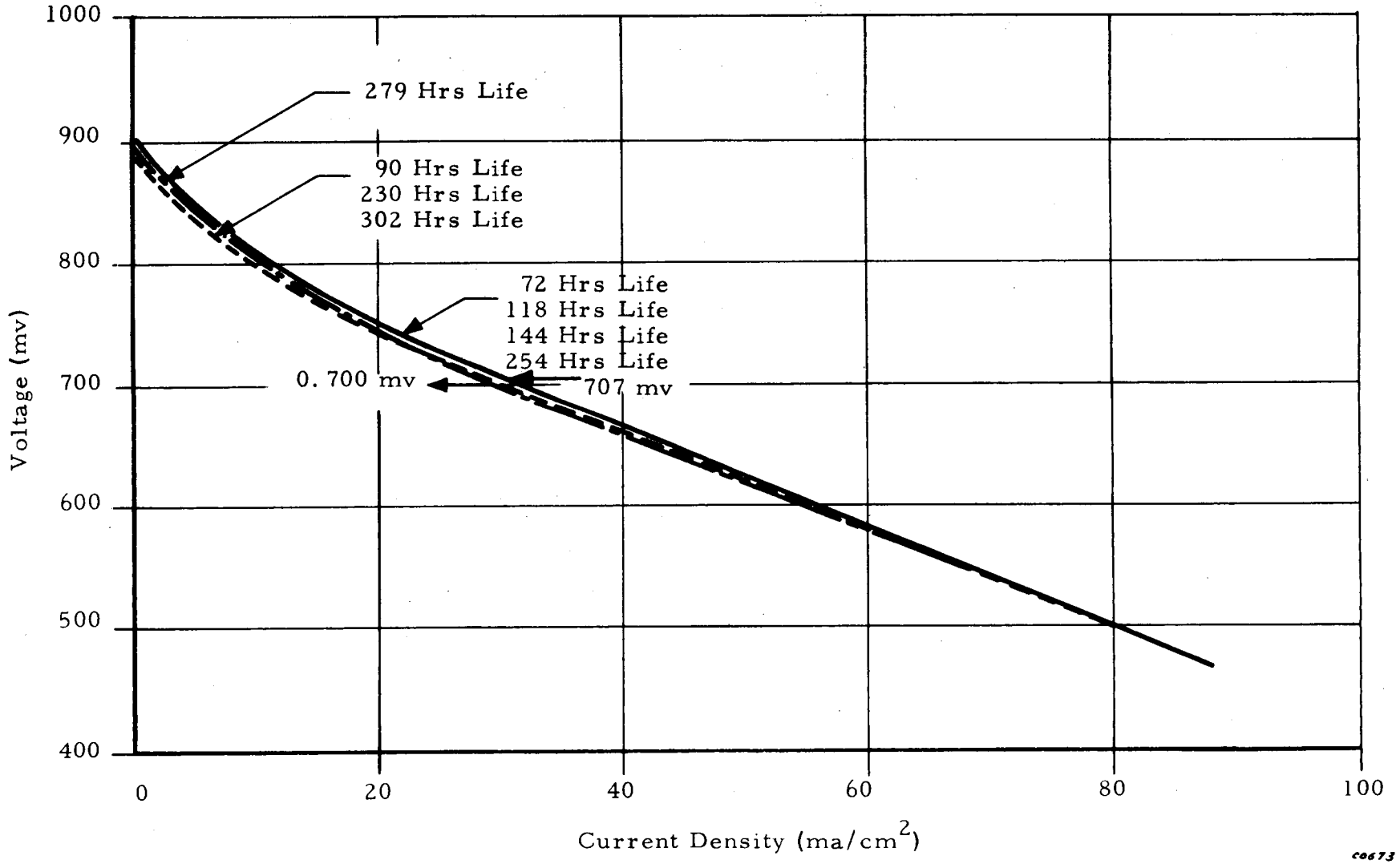


Figure 10. Polarization Curves for Inorganic Membrane Fuel Cell, Test 8, Table I

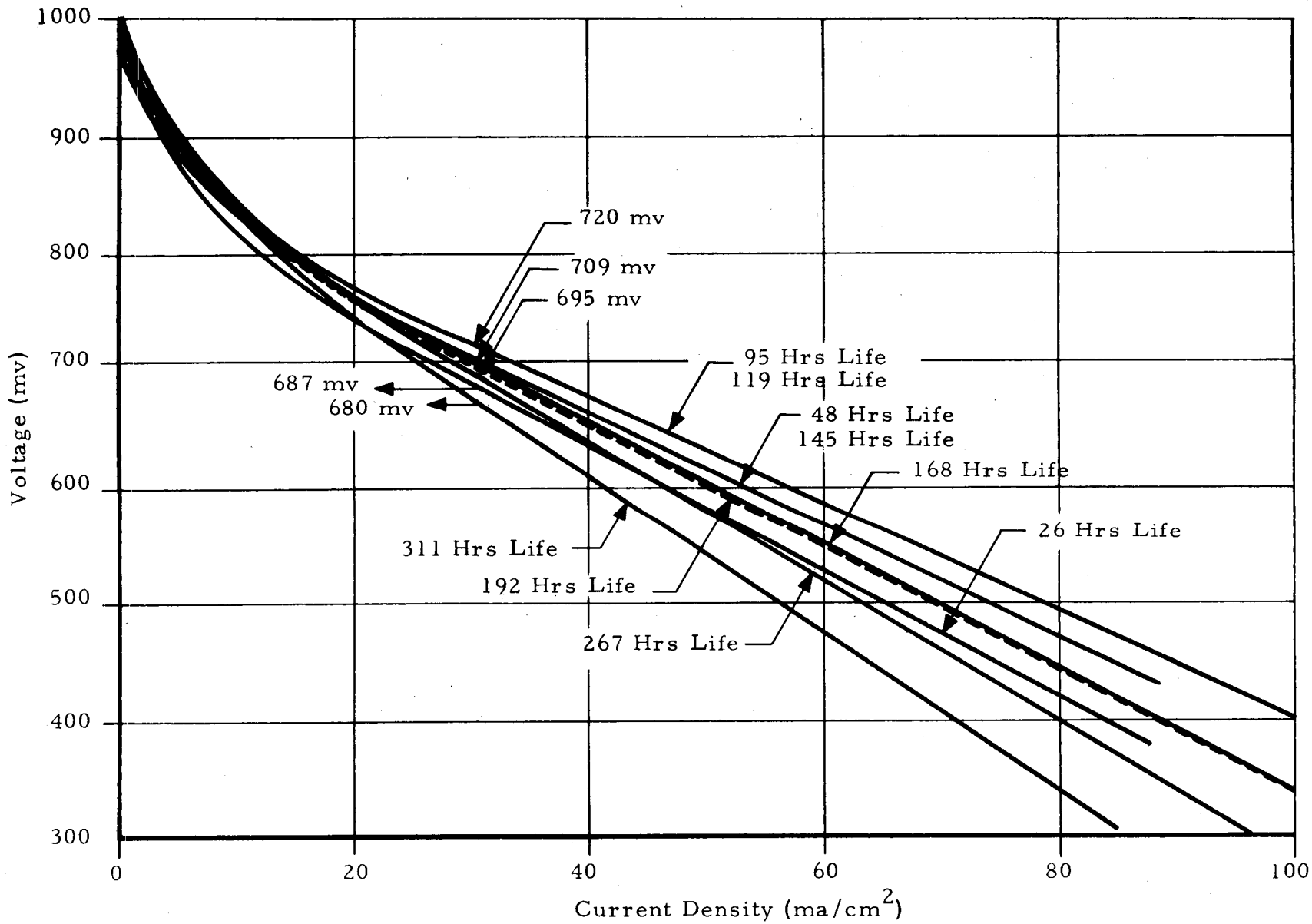


Figure 11. Polarization Curves for Inorganic Membrane Fuel Cell Test 13, Table I

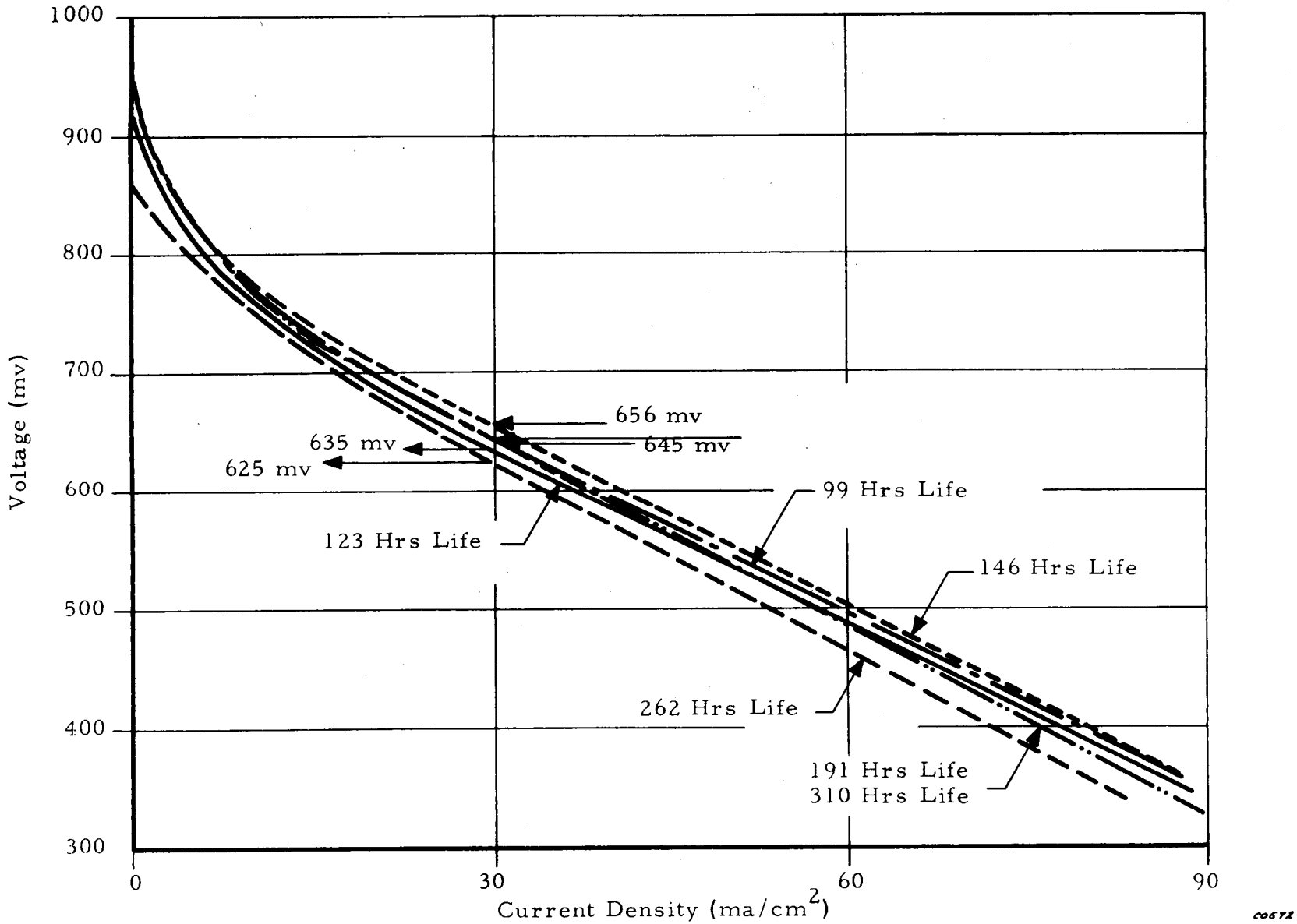


Figure 12. Polarization Curves for Inorganic Membrane Fuel Cell, Test 14, Table I

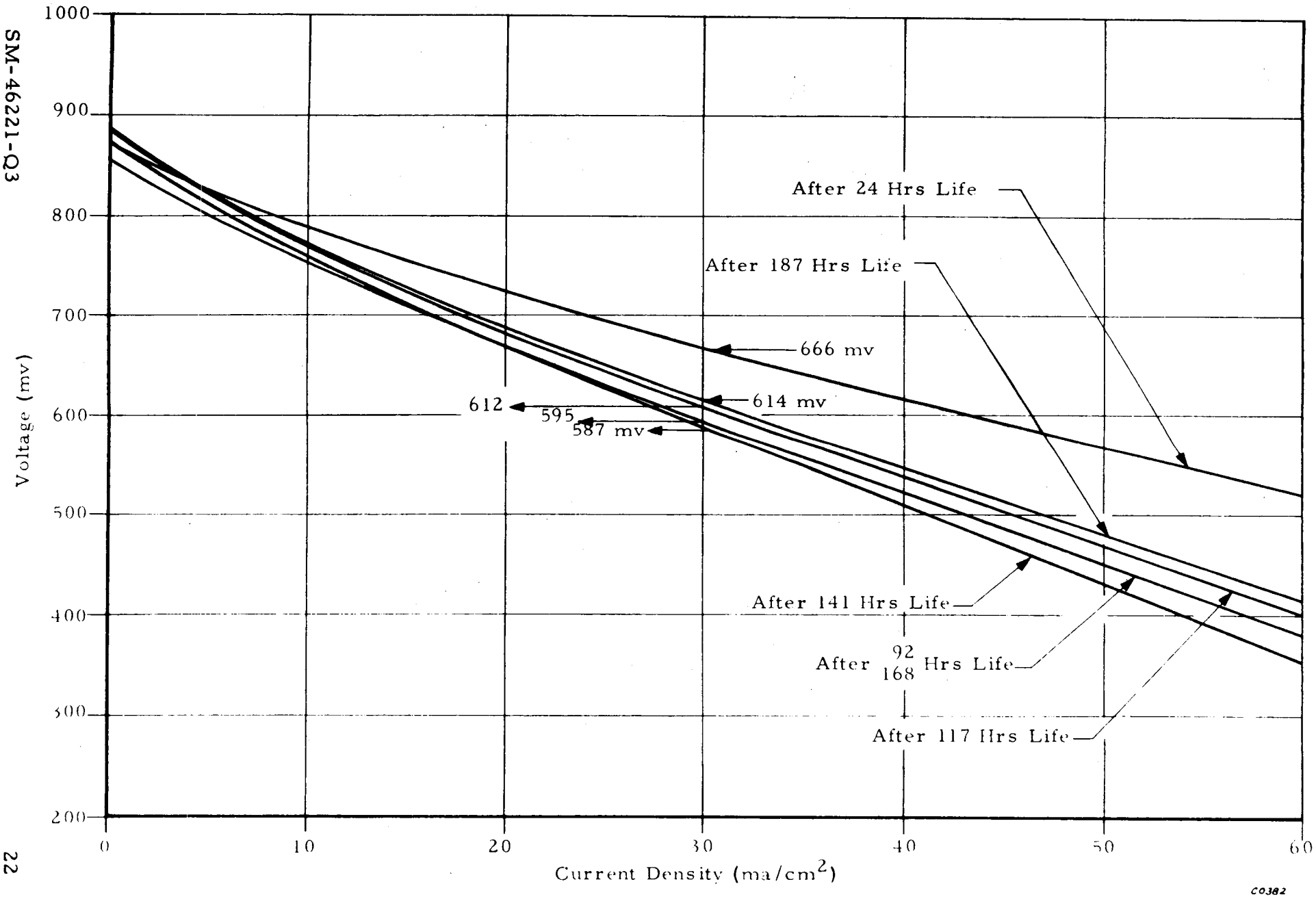


Figure 13. Polarization Curves for Inorganic Membrane Test Cell. Test 19, Table I

The synopsis of voltage-time data for Tests (8) and (13) are given in Figures 7 and 8 respectively. Polarization curves for Tests (5), (8), (13), (14) and (19) are given in Figures 9-13, respectively.

3.2 Catalyst-Membrane and Electrode-Catalyst-Membrane Preparations by Pressing and Sintering Techniques

Work has continued during this report period devoted to the development of composite fuel cell membrane-catalyst for components. These are prepared by pressing together three layers of material, the two outer layers being composed of mixtures of C200B membrane material and catalyst and the center section being C200B material without the catalyst addition. Each section has been approximately 0.38 mm thick resulting in a pressed composite about 1.2 mm thick. After pressing, the composites are sintered and diffusion bonded.

As described above in Section 3.1.1, there appears to be advantages in employing thinner outer catalyst layers. This is under current investigation.

Catalyst-Membrane Composites

The following membrane-catalyst composites have been prepared or are in preparation for fuel cell evaluation.

Same Catalyst Layer on Both Sides of Membrane

10& Pt - 90% C200B
20% Pt - 80% C200B
30% Pt - 70% C200B
40% Pt - 60% C200B
10% Pd - 90% C200B
20% Pd - 80% C200B
30% Pd - 70% C200B
40% Pd - 60% C200B
10% (50% Pt - 50% Pd) - 90% C200B

Different Catalyst Layer on Each Side of Membrane

10% (50% Pt - 50% Ir)	- 90% C200B	(H_2 Side)
10% Pt	- 90% C200B	(O_2 Side)
10% (50 Ag - 50% Pd)	- 90% C200B	(O_2 Side)
10% Pt	- 90% C200B	(H_2 Side)

As the maximum concentration and type of catalyst producing the best fuel cell operation is determined, the thickness of the outer catalyst bearing layers will be reduced to the minimum consistent with optimum fuel cell operation. This will results in reducing both catalyst cost and membrane thickness as well. It appears, for example, that the thickness of the catalyst layers containing at least 20% Pt, can be reduced by at least three-fold.

Electrode-Catalyst-Membrane Composites

Work is also being performed on the incorporation of electrodes into the catalyst-membrane composites. These samples are prepared by the following two different techniques:

(1) Screen Electrodes

Screen electrodes are pressed into the catalyst-membrane composite and sintered as described above.

(2) Silk Screen Electrodes

Electrode materials such as platinum, palladium, iridium and other materials are silk screened on the surfaces of the pressed catalyst-membrane composite as described above either before or after sintering. If applied after sintering the assembly is then refired to bond the electrode layers to the catalyst-membrane composite.

A. Screen Electrodes

1. Silver Mesh
2. Tantalum Mesh
3. Nickel Mesh
4. Stainless Steel Mesh
5. Gold Mesh
6. Platinum

PLATINIZED

B. Silk Screen Electrodes

1. Platinum
2. Palladium
3. Tantalum
4. Iridium

The evaluations for these materials are in progress at the present time.

3.3 Mass and Heat Transfer Analysis of Optimum Fuel Cell Design Parameters

The operation of a hydrogen-oxygen fuel cell is particularly sensitive to the proper water balance existing between the rates of water vapor removal from the electrode surface. Failure to maintain the optimum water balance about the electrodes would result in either a drowning or dehydration of the membrane-electrode composite. Consequently, a mathematical analysis was conducted to determine the optimum engineering design parameters for a flow-type fuel cell. The design will be formulated in terms of three dimensionless parameters; namely, the relative gas velocity ($\bar{v}\ b/D_v$), relative mean discharge humidity ($\bar{H}/H_s$) and the relative width of gas passage ($\frac{b}{a}$).

3.3.1 Mass Transfer Analysis

In principle, water is generated at the catalyst interface which sweats through the electrode surface. Simultaneously, the vaporization of water vapor into the gas passage serves to remove the heat generated in the electrolyte-bearing membrane, the primary mode of heat removal.

The water removal process is formulated quantitatively by writing the differential equation about a differential element of gas in the flow stream. It is given by Equation (1) below.

$$\bar{v}\ (\partial H/\partial z) = D_v\ (\partial^2 H/\partial^2 x) \qquad (1)$$

Equation (1) describes the equality between the convection of water vapor along the gas passage and the diffusion of water vapor across the flowing stream. The boundary conditions, given below, to this mass transfer problem depict the surface conditions of the electrode.

$$H = H_o \quad \text{at} \quad x = o \quad \text{and } z = o \tag{2}$$

$$H = H_s \quad \text{at} \quad x = o \quad \text{and } z = a \tag{3}$$

The solution to this system of equations for the gas humidity profile in the plane or the discharge port is given below.

$$(H - H_s) / (H_o - H_s) = \text{erf}\ (\bar{v}\, b^2 / 4\, a\, D_v)^{1/2} \tag{4}$$

By integrating Equation (4) across the width of the gas passage, the average discharge humidity $\bar{H}$ of the gas leaving the fuel cell is given as follows:

$$(\bar{H} - H_s) / (H_o - H_s) = (\bar{v}\, b^2 / 4\pi\, a\, D_v)^{1/2} \tag{5}$$

Equation (5) is presented graphically in Figure 14.

The optimum engineering design parameters are obtained graphically by plotting the relative gas velocity ($\bar{v}\, b/D_v$) versus the relative width of the gas passage (b/a) at constant values of the relative mean discharge humidity ($\bar{H}/H_s$). Figure 15 is based upon ($\bar{H}/H_s$) = 0.95. For this discharge humidity, the optimum design parameter (b/a) is 0.208. This is essentially the prevailing cell geometry for the Astropower compact fuel cell, described in Section 3.1.2. Exceedingly lower values would result at increased saturation of flowing gas.

In conclusion, the mathematical analysis resulted in the determination of the engineering design parameters pertinent to the optimum operation of a hydrogen-oxygen fuel cell. In general, design curves similar to those of Figure 14 can be used to evaluate the discharge gas humidity, $\bar{H}$ as a function of the gas velocity, $\bar{v}$ and the width of the gas passage b. The optimum dimensionless design parameters ($\bar{H}/H_s$) ($\bar{v}\, b/D_v$) and (b/a) evaluated in this analysis can be determined from curves similar to those of Figure 15.

3.3.2 Heat Transfer Analysis

A mathematical analysis was conducted to determine the heat rejected from the electrodes of a fuel cell. The heat generated over the surface of the electrodes due to the electrochemical reactions and I^2R

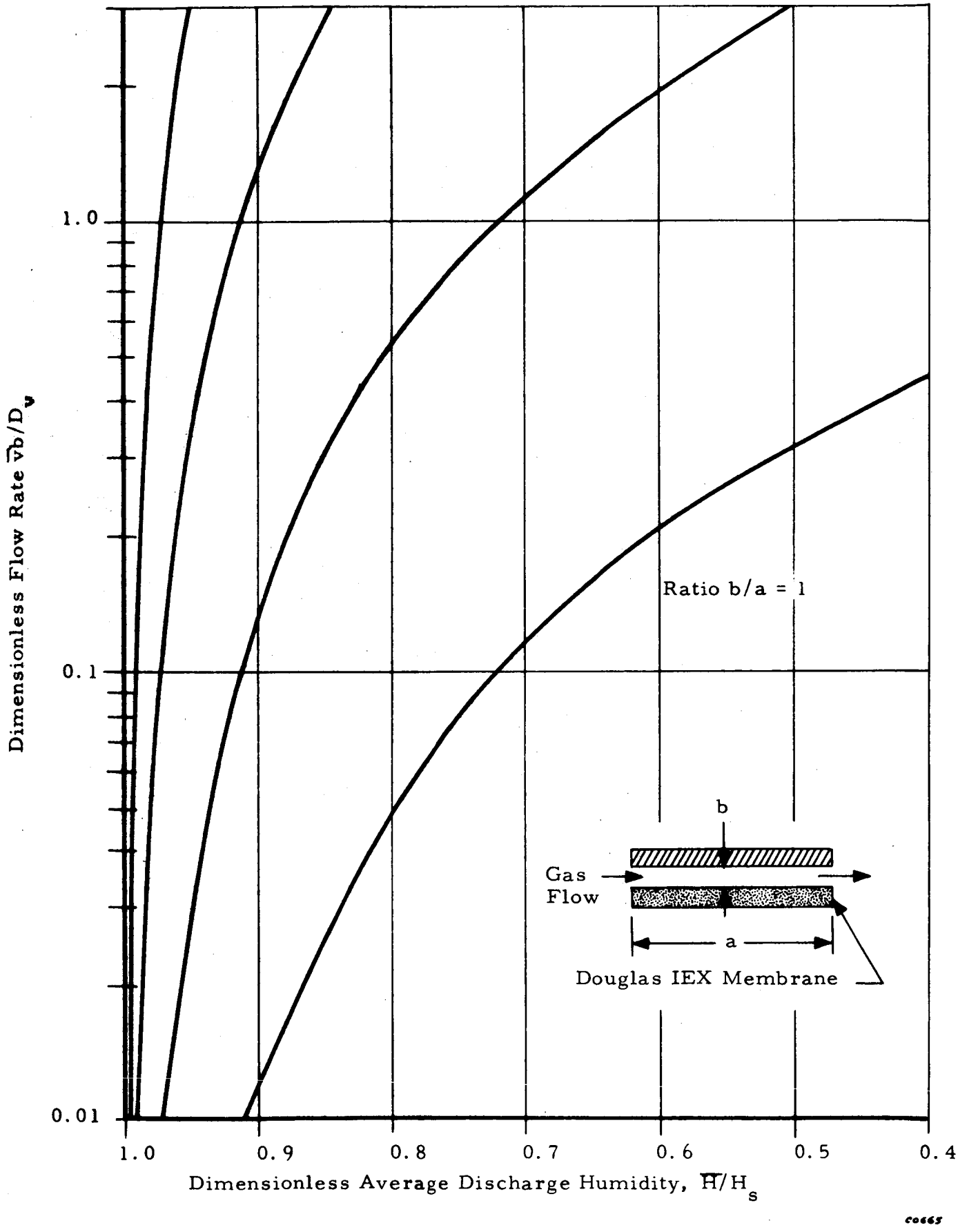


Figure 14. Discharge Gas Humidity vs Fuel Cell Geometry

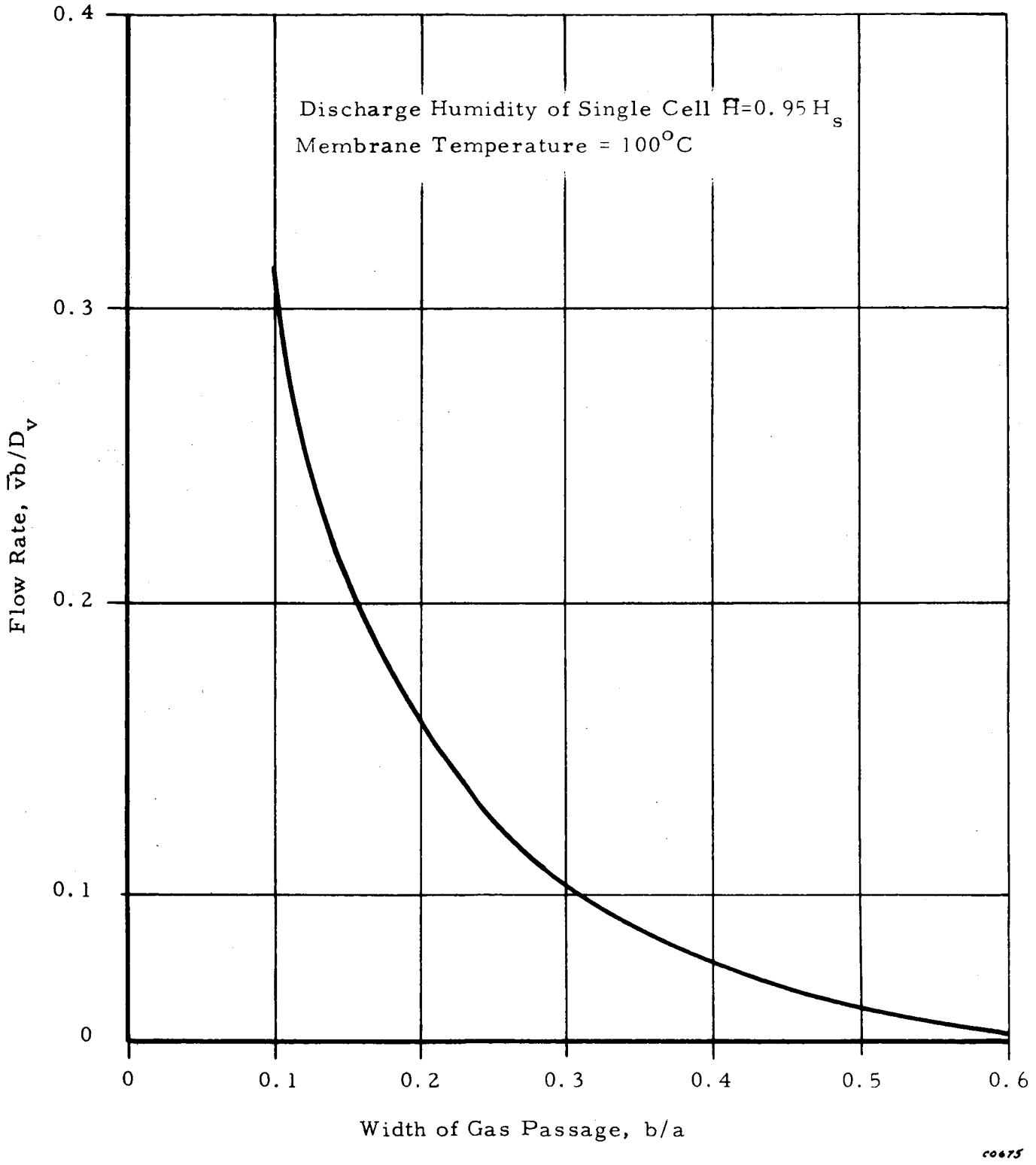


Figure 15. Optimized Fuel Cell Design

losses is assumed to be uniform over the entire area. It is dissipated by the process of water vaporization to the flowing gas stream, thereby, increasing the discharge gas temperature for a specific gas velocity.

To describe this thermal behavior of the flow type fuel cell, an energy balance was written across a differential volume of flowing gas. Under steady flow conditions, the resultant heat transport equation is given below.

$$\rho C_p \bar{v}_z (\partial T/\partial z) = k (\partial^2 T/\partial x^2) \qquad (6)$$

Equation (6) represents the balance between the convective transport of energy along the flow path and the thermal transport of energy across the flowing gas stream by conduction.

The boundary conditions, being of the homogeneous type, are formulated on the basis that the heats of electrochemical reaction are generated at a constant rate, q_1 over the entire electrode surface. The equivalent expressions are given below.

$$T = T_o \quad \text{at} \quad z = 0 \quad \text{between} \quad (o < x < b) \qquad (7)$$

$$(\partial T/\partial z)_{x=o} = q_o/k \quad \text{at} \quad x = 0 \quad \text{between} \quad (o < z < a) \qquad (8)$$

The analytical solution to Equations (6), (7) and (8) in terms of dimensionless parameters is given below.

$$T/T_o = 1 + (q_o/kT_o)(4\pi ka/\rho C_p \bar{v})^{1/2} \operatorname{erf} (\bar{v} x^2 \rho Cp/4k a)^{1/2} \qquad (9)$$

Equation (9) represents the temperature profile across the flowing gas stream in the plane of the discharge port. Although the minimum thermal variation will occur at gas rates in excess of the stoichiometric limits, the maximum increase in the gas temperature at the electrode surface is attained at high cell efficiencies for a fuel cell operating under adiabatic conditions at the stoichiometric ratio of the reactants. Under these conditions, the thermal ratio (T_s/t_o) at the electrode surface could exceed the bulk gas stream value by 120 percent.

NOMENCLATURE

ρ = gas density, lb mass/ft^3

C_p = heat capacity, Btu/(lb mass) (^oR)

$\bar{v}$ = gas velocity, ft/sec

T = Temperature, ^oR

z = Position along gas flow, ft

k = Thermal conductivity, (Btu) ft/(sq ft) (sec) (^oR)

x = Position across gas flow, ft

b = Width of gas passage, ft

a = Length of gas passage, ft

q_o = Heat flux at electrode surface, Btu/(ft^2) (sec)

T_o = Inlet gas temperature, ^oR

H = Humidity of gas stream, lb mass water/lb mass gas

D_v = Molecular diffusivity, sq ft/sec

$\bar{H}$ = Average gas humidity, lb mass water/lb mass gas

H_s = Saturated gas humidity at electrode, lb mass water/lb mass gas

H_o = Inlet gas humidity, lb mass water/lb mass gas

erf = Probability integral

erfc = Complement of probability integral

3.4 Additional Studies – New Catalyst and Water-Proofing Studies

Several pairs of electrodes were fabricated using tantalum screen and a variety of catalysts and water-proofing agents. Platinum was used as the catalyst. It was applied to the two-inch diameter screens by either of two methods: electrodeposition or application of a "paste" or slurry. Electrodeposition was carried out in 3% H_2PtCl_6(aq). Up to 40% by weight (total weight of screen) of platinum was deposited in this fashion. The platinum pastes consisted of platinum black suspended in a toluene solution of silicone

resin (G.E. SR-224). After application of the paste, the toluene was evaporated and the resin cured at 90°C.

Other catalysts were also applied to tantalum screens by the "pasting" technique. Silicone resin was invariably used as the water-proofing binder. The catalysts employed were palladium, nickel boride, and cobalt boride. The finished screens contained from 30% to 40% by weight catalyst.

Several of the successful electrode screens are being evaluated in working fuel cells. A few of the above electrodes are currently being evaluated.

4.0 FUTURE WORK

(1) Studies involving new catalyst-membrane composites and electrode-catalyst-membrane composites as indicated herein will be continued.

(2) Studies with the compact fuel cell will be continued. A larger cell will be constructed capable of handling a four-inch diameter mamebrane. Tests will be performed with this unit.

(3) Efforts will be directed toward the water-proofing of electrodes. It is planned to attempt to improve fuel cell performance by providing the compact unit with a wicking arrangement.

5.0 REFERENCES

(1) "Inorganic Ion Exchange Membrane Fuel Cell" NASA-Lewis Center Contract NAS 3-6000, Quarterly Progress Report SM 46221-Q1, Period Ending 10 October 1964.

(2) Ibid, Quarterly Progress Report SM 46221-Q2, Period Ending 10 January 1965.

(3) Ibid, Monthly Progress Report for Period Ending 10 February 1965.

(4) Ibid, Monthly Progress Report for Period Ending 10 March 1965.

6.0 PROJECT PERSONNEL

The following Astropower staff personnel were connected with the program during this reporting period:

Dr. C. Berger, Principal Investigator
Dr. M. P. Strier
Mr. F. C. Arrance
Dr. L. O. Rutz
Mr. G. Belfort
Mr. A. G. Rosa
Mr. R. Hubata

TABLE I

SUMMARY OF MORE SIGNIFICANT FUEL CELL TESTS ON PRESINTERED ZIRCONIUM DIOXIDE – PHOSPHORIC ACID – "ZEOLON H" MEMBRANE C200B TESTS CONDUCTED AT CONSTANT CURRENT DENSITY OF 30 ma/cm²

Fuel Cell Test No.	Membrane Description	Membrane Thickness, mm	Temperature °C	Current Density at 0.5 v, ma/cm²	Voltage at 30 ma/cm², volts	Fuel Cell Resistance, ohms	Open Circuit voltage, volts	Time of Measurement from Start of Run, hours	Time of Run[b]
1	No special treatment	0.51	25 ± 1	27.5	0.464	0.66	0.953	98	
				26.5	0.453	0.66	0.962	140	
				26.0	0.444	0.69	0.967	166	
				26.0	0.444	0.69	0.983	231	
2	No special treatment	0.88	65 ± 1	69.5	0.706	0.26	1.020	46	126
				61.7	0.682	0.29	1.020	70	
				52.0	0.647	0.33	0.992	97	
3	No special treatment	0.20	65 ± 2	60.0	0.678	0.29	0.985	3	23
4	Membrane was chemically platinized, then, resintered at 500°C	0.86	65 ± 1	52.2	0.650	0.33	0.968	24	48
5	Hydrolyzed for 2 hours at 75°C, then, sintered at 500°C for 2 hours	0.53	65 ± 1	50.5	0.650	0.36	0.978	56	345
				51.0	0.657	0.36	1.000	97	
				45.0	0.635	0.38	0.990	172	
				43.6	0.622	0.42	0.980	200	
				43.5	0.622	0.42	0.980	218	
				40.2	0.611	0.42	0.988	242	
				–	0.605	–	–	266	
				–	0.605	–	–	314	
6	No special treatment	0.75	65 ± 1	33.0	0.525	0.62	0.889	47	118[c-1]
				37.0	0.550	0.55	0.893	78	
7	No special treatment	0.72	65 ± 1	60.0	0.655	0.26	0.885	16	144[c-2]
				62.5	0.668	0.26	0.943	42	
				57.5	0.652	0.27	0.928	90	
				51.0	0.635	0.31	0.830	119	
8	No special treatment	0.50	65 ± 1	80.0	0.707	0.20	0.905	72	307[c-3]
				80.0	0.700	0.20	0.900	90	
				80.0	0.707	0.20	0.908	118	
				80.0	0.707	0.20	0.905	144	
				80.0	0.700	0.20	0.900	230	
				80.0	0.707	0.20	0.900	254	
				80.0	0.700	0.20	0.892	279	
				80.0	0.700	0.20	0.892	302	

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TABLE I (CONT.)

SUMMARY OF MORE SIGNIFICANT FUEL CELL TESTS ON PRESINTERED ZIRCONIUM DIOXIDE – PHOSPHORIC ACID – "ZEOLON H" MEMBRANE C200B TESTS CONDUCTED AT CONSTANT CURRENT DENSITY OF 30 ma/cm^2

Fuel Cell Test No.	Membrane Description	Membrane Thickness, mm	Temperature °C	Current Density at 0.5 v, ma/cm^2	Voltage at 30 ma/cm^2, volts	Fuel Cell Resistance, ohms	Open Circuit voltage, volts	Time Measurement from Start of Run, hours	Time of run[b]
9	Impregnated with platinum black – 10% in both outer one-third layers	0.84	65 ± 1	26.9	0.460	0.62	0.933	97	362
				26.9	0.460	0.62	0.965	115	
				31.0	0.510	0.57	0.952	195	
				33.0	0.533	0.53	0.952	199	
				35.0	0.552	0.51	0.965	291	
				–	0.570	–	–	316	
				–	0.548	–	–	350	
10	Impregnated with platinum black – 20% in both outer one-third layers	1.15	65 ± 3	37.6	0.570	0.45	0.975	7	49
				49.1	0.658	0.41	1.100	24	
				53.7	0.658	0.33	0.988	31	
11	Impregnated with platinum black – 30% in both outer one-third layers	1.22	65 ± 1	47.7	0.649	0.41	1.080	27	263
				54.7	0.657	0.31	0.995	44	
				52.0	0.652	0.32	1.065	70	
				55.7	0.667	0.32	1.090	93	
				55.7	0.667	0.32	1.075	118	
				50.3	0.650	0.36	1.075	166	
				51.3	0.652	0.35	1.100	194	
				46.3	0.631	0.41	1.090	217	
12	Impregnated with platinum black – 40% in both outer one-third layers	1.19	65 ± 2	52.0	0.624	0.28	0.885	32	168
				58.6	0.650	0.26	0.889	46	
				42.6	0.595	0.27	0.885	70	
				37.5	0.556	0.37	0.860	101	
				40.6	0.578	0.36	0.873	123	
13	Impregnated with platinum black – 20% in both outer one-tenth layers	0.78	66 ± 2	64.7	0.687	0.26	0.987	26	311
				73.5	0.709	0.24	0.995	48	
				78.1	0.720	0.23	1.000	95	
				78.1	0.720	0.23	1.010	119	
				73.5	0.709	0.24	1.015	145	
				69.0	0.709	0.27	0.992	168	
				68.6	0.709	0.27	1.010	192	
				62.7	0.695	0.31	1.005	267	
				57.0	0.680	0.53	1.015	311	

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TABLE I (CONT.)

SUMMARY OF MORE SIGNIFICANT FUEL CELL TESTS ON PRESINTERED ZIRCONIUM DIOXIDE – PHOSPHORIC ACID – "ZEOLON H" MEMBRANE C200B TESTS CONDUCTED AT CONSTANT CURRENT DENSITY OF 30 ma/cm^2

Fuel Cell Test No.	Membrane Description	Membrane Thickness, mm	Temperature °C	Current Density at 0.5 v, ma/cm^2	Voltage at 30 ma/cm^2, volts	Fuel Cell Resistance, ohms	Open Circuit voltage, volts	Time Measurement from Start of Run, hours	Time of Run[b]
14	Impregnated with platinum black – 20% in both outer one-tenth layers	0.74	69 ± 1	59.2	0.645	0.25	0.965	99	310[c-2]
				57.2	0.645	0.26	0.965	123	
				60.5	0.656	0.26	0.965	146	
				57.7	0.635	0.24	0.935	191	
				53.1	0.625	0.26	0.800	262	
				57.7	0.625	0.24	0.953	310	
15	Impregnated with platinum black – 20% in both outer one-third layers followed by impregnation with 0.1% palladium	0.89	80 ± 5	29.2	0.492	0.54	0.912	174	318
				30.2	0.502	0.56	0.942	192	
				–	0.460	–	–	240	
				–	0.440	–	–	272	
				28.9	0.486	0.57	0.922	294	
16	Impregnated with platinum black – 20% in both outer one-third layers	1.06	85 ± 5	54.4	0.631	0.27	0.860	4	72
				55.0	0.646	0.29	0.800	22	
				–	0.639	–	–	48	
				–	0.615	–	–	72	
17	Impregnated with platinum black – 20% in both outer one-third layers	1.31	93 ± 2	36.0	0.545	0.43	0.940	21	50[c-1]
18	No special treatment	0.66	97 ± 3	–	0.672	–	–	1	8
				–	0.680	–	–	2	
				–	0.635	–	–	4	
				–	0.668	–	–	5	
				–	0.635	–	–	8	
19	Impregnated with platinum black – 20% in both outer one-third layers	0.93	100 ± 3	65.0	0.666	0.24	0.872	24	312
				42.4	0.595	0.36	0.855	92	
				44.8	0.612	0.35	0.888	117	
				40.9	0.587	0.39	0.872	141	
				42.4	0.595	0.36	0.883	168	
				45.8	0.614	0.34	0.882	187	
				–	0.620	–	–	216	
				–	0.580	–	–	264	
				–	0.550	–	–	271	
				–	0.500	–	–	312	

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TABLE I (CONT.)

SUMMARY OF MORE SICNIGICANT FUEL CELL TESTS ON PRESINTERED ZIRCONIUM DIOXIDE – PHOSPHORIC ACID – "ZEOLON H" MEMBRANE C200B TESTS CONDUCTED AT CONSTANT CURRENT DENSITY OF 30 ma/cm^2

Fuel Cell Test No.	Membrane Description	Membrane Thickness, mm	Temperature °C	Current Density at 0.5 v, ma/cm^2	Voltage at 30 ma/cm^2, volts	Fuel Cell Resistance, ohms	Open Circuit voltage, volts	Time Measurement from Start of Run, hours	Time of Run[b]
20	Impregnated with palladium black – 20% both outer one-third layers	1.27	100 ± 2	32.5	0.520	0.41	0.818	22	76
				36.5	0.545	0.36	0.830	28	
				40.3	0.570	0.33	0.833	46	
				47.0	0.614	0.33	0.840	52	
				42.5	5.80	0.35	0.880	72	
21	Impregnated with platinum black – 10% in both outer one-third layers	0.92	103 ± 2	28.8	0.490	0.40	0.785	45	307
				20.4	0.389	0.56	0.850	189	
				18.8	0.385	0.49	0.752	255	
				15.8	0.343	0.51	0.710	284	
22	Impregnated with platinum black – 20% in both outer one-third layers	1.52	128 ± 1	30.0	0.500	0.48	0.900	24	31[c-2]
23	Impregnated with platinum black – 20% in both outer one-third layers	1.43	102 ± 2	47.5	0.624	0.34	0.965	21	146
			102 ± 2	45.5	0.615	0.37	0.942	45	
			102 ± 2	42.2	0.597	0.40	0.935	51	
			120	36.2	0.605	0.42	0.952	54	
			148	20.9	0.364	0.72	0.928	99	

(a) All tests were run at constant current densities of 30 ma/cm^2 with membranes having cross sectional areas of 20-2 cm^2, except test No. 7 which had cross sectional area of 13.38 cm^2.

(b) Tests run for less than 300 hours were terminated because of diminishing fuel cell performance.

(c) Tests run in the compact design fuel cell, with (1) 20 gauge stainless steel screen, (2) 44-hole back-up plate, (3) 92-hole back-up plate.

TABLE II

COMPARISON OF RESISTANCE FOR STANDARD NON-IMPREGNATED MEMBRANE AT VARIOUS TEMPERATURES

Temperature °C Thickness mm	Resistance - (Ohms) 25°C Fuel Cell Test No.	65°C Fuel Cell Test No.
0.00 - 0.50		0.29/3
0.50 - 0.75	0.66/1	0.29/(b)
0.75 - 1.00	0.74/(a)	0.33/2

(a) From Reference (2) - Fuel Cell Test No. 1

(b) From Reference (2) - Fuel Cell Test No. 5

TABLE III

RESISTANCE COMPARISON AT 65± 2°C AND VARIOUS TIME INTERVALS FOR CONCENTRATIONS OF PLATINUM BLACK IMPREGNATED IN THE OUTER ONE-THIRD LAYERS

		Resistance (OHMS) at 65± 2°C					
Time (Hours)		0 - 50	50 - 100	100 - 150	150 - 200	200 - 250	250 - 300
%-Pt	Test No.						
10	9		0.62	0.62	0.55		0.51
20	10	0.33					
30	11	0.36	0.32	0.32	0.35	0.41	
40	12	0.27	0.27	0.36			

TABLE IV

COMPARISON OF RESISTANCE AT 100± 2°C AND VARIOUS TIME INTERVALS FOR 10 AND 20 PERCENT PLATINUM BLACK IMPREGNATED IN THE OUTER ONE-THIRD LAYERS

		Resistance (OHMS) at 100± 2°C					
Time (Hours)		0 - 50	50 - 100	100 - 150	150 - 200	200 - 250	250 - 300
%-Pt	Test No.						
10	21	0.40			0.56		0.50
20	19, 23	0.24	0.36	0.37	0.35		

TABLE V

COMPARISON OF RESISTANCE FOR 20 PERCENT PLATINUM BLACK MEMBRANE IMPREGNATED IN THE OUTER ONE-THIRD LAYERS AT VARIOUS TEMPERATURES

Temperature °C	Resistance - (OHMS)				
	65 ± 2	85 ± 2	100 ± 2	125 ± 3	148 ± 2
(a) Resistance (ohms)	0.33	0.30	0.35	0.45	0.72
Membrane Thickness (mm)	1.15	1.06	0.93 1.43	1.43	1.43
Fuel Cell Test Number	10	16	19 23	23	23

(a) After approximately 50 hours

TABLE VI

COMPARISON OF RESISTANCE AT 100 ± 2°C AND VARIOUS TIME INTERVALS FOR 20 PERCENT PLATINUM AND PALLADIUM-BLACK CATALYST IMPREGNATED IN THE OUTER ONE-THIRD LAYERS

Time (Hours) / Catalyst		Resistance (OHMS) at 100 ± 2°C					
		0 - 30	30 - 40	40 - 50	50 - 60	60 - 70	70 - 80
% Pt.	Test No.						
20	19, 23	0.34		0.37	0.40		
% Pd.							
20	20	0.41		0.33	0.33		0.35

TABLE VII

COMPARISON RESISTANCE[a] OF STANDARD MEMBRANE AND MEMBRANE WITH 20 PERCENT PLATINUM BLACK IMPREGNATED IN THE OUTER ONE-THIRD LAYERS AT 65 ± 1°C AND 82 ± 2°C

	65 ± 1°C			82 ± 2°C		
Membrane Composition	Fuel Cell Test No.	Resistance Ohms	Membrane Thickness mm	Fuel Cell Test No.	Resistance Ohms	Membrane Thickness mm
Standard C200B	2	0.23	0.88	(c)	0.36	0.74
	(b)	0.31	0.67			
20% Impregnated Pt-black in Outer one-third layer	10	0.33	1.15	16	0.30	1.06

(a) Noted after approximately 50 hours.

(b) From Reference 2 - Fuel Cell Test No. 5

(c) From Reference 2 - Fuel Cell Test No. 12 (122 hours)

TABLE VIII

SUMMARY OF MORE SIGNIFICANT FUEL CELL TESTS ON PRESINTERED ZIRCONIUM DIOXIDE – PHOSPHORIC ACID – "ZEOLON H" MEMBRANE C200B OPERATED IN THE COMPACT FUEL CELL DESIGN

Fuel Cell Test No.	Membrane Description	Membrane Thickness	Temperature °C	Current Density at 0.5 v, ma/cm²	Voltage at 30 ma/cm², volts	Fuel Cell Resistance, ohms	Open Circuit Voltage, volts	Time of Measurement from Start of Run, hours	Time of Run
6[a]	No special treatment	0.75	65 ± 1	33.0	0.525	0.62	0.889	47	118
				37.0	0.550	0.55	0.993	78	
7[b]	No special treatment	0.72	65 ± 1	62.5	0.668	0.26	0.943	42	144
				57.5	0.652	0.27	0.928	90	
				51.0	0.635	0.31	0.880	119	
8[c]	No special treatment	0.50	65 ± 1	80.0	0.700	0.20	0.900	72	307
				80.0	0.700	0.20	0.900	90	
				80.0	0.707	0.20	0.908	118	
				80.0	0.707	0.20	0.905	144	
				80.0	0.700	0.20	0.900	230	
				80.0	0.707	0.20	0.900	254	
				80.0	0.702	0.20	0.892	279	
				80.0	0.700	0.20	0.892	302	
14[b]	Impregnated with Platinum Black – 20% in both outer one-tenth layers	0.70	69 ± 1	59.2	0.645	0.25	0.965	99	310
				57.2	0.645	0.26	0.965	123	
				60.5	0.656	0.26	0.965	146	
				57.7	0.635	0.24	0.935	191	
				57.7	0.625	0.24	0.953	310	
17[b]	Impregnated with Platinum Black – 20% in both outer one-third layers	1.31	93 ± 2	36.0	0.545	0.43	0.940	21	50
22[b]	Impregnated with Platinum Black – 20% in both outer one-third layers	1.52	128 ± 1	30.0	0.500	0.48	0.900	24	31

(a) Involved use of wire mesh screen back-up plate.

(b) Involved use of 44-hole back-up plate.

(c) Involved use of 96-hole back-up plate.

TABLE IX

(a)

COMPARISON OF RESISTANCE FOR THE COMPACT FUEL CELL DESIGN USING VARIOUS BACK-UP PLATE CONFIGURATIONS

Fuel Cell Test Number	Type of Back-up Plate	Temperature °C	Resistance Ohms
8	20 gauge stainless steel screen	65 ± 1	0.62
9	44-Hole stainless steel 1/8" thick disc	65 ± 1	0.26
10	92-Hole stainless steel 1/8" thick disc	65 ± 1	0.20

(a) Noted after approximately 50 hours.

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