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PHOSPHORIC ACID FUEL CELL STACK
AND SYSTEM DEVELOPMENT

MASTER

TECHNICAL PROGRESS REPORT

FOR THE PERIOD

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ABSTRACT

This Technical Progress Report covers the first quarterly period, through December 31, 1978. Primary emphasis during the period was on stack component development - Task I. In the fuel processing sub-system area - Task II - effort was devoted to thermodynamic analysis of the steam-reforming process and preliminary system studies directed toward fuel selection. The main system analysis and design effort - Task III - will begin after selection of a fuel. Stack component scale-up activity - Task IV - will begin in mid-1979. Electrocatalysts for the Task I program are being developed under a separate task that is wholly sponsored by Engelhard - Task V.

The bipolar plate development effort has emphasized a non-grooved, multi-element configuration. Properties tests for both the porous element and the impermeable element indicate that viable materials are now on hand. Phosphoric acid tolerance testing is proceeding, and preparations are being made for evaluation in small stacks.

The matrix program has substantially shifted from membrane-type to laminate-type configurations because of lower cell IR-losses and apparent superiority in high-temperature tolerance. Typical IR-losses are 40 mV at 150 A/ft² and 163°C. Matrix configurations that facilitate acid replenishment are being evaluated.

Electrodes utilizing 30% Pt on carbon catalysts at a nominal loading of 1.4 mg Pt/cm² have been evaluated. Recently, emphasis has been shifted to 10% Pt on carbon catalysts at a nominal loading of 0.46 mg Pt/cm². Initial performance levels have been consistent with program goals. Substrate wetproofing optimization studies are in progress.

Cyclic-voltammetric studies have indicated substantial electrocatalyst (30% Pt) surface-area loss in a 2040-hour single-cell test at 163°C (about 65% reduction) and in a 48-hour potentiostatic (0.7 V, RHE) aging test at 204°C (almost 50% reduction). Advanced catalyst development efforts are being directed toward improved resistance to sintering in phosphoric acid.

Thermodynamic equilibria have been determined for steam-reformer exit conditions over wide ranges of temperature and H_2O/C ratio. This has been completed for three candidate fuels: natural gas (methane), naphtha, and methanol. Preliminary system analysis directed toward selection of a fuel is proceeding. Sub-scale fuel processing test facilities have been designed, and laboratory preparations are underway. Flexibility was built into the design to allow accommodation of any of the candidate fuels with minor modifications.

INTRODUCTION

This program is directed toward the upgrading of phosphoric acid stack component technology in association with fuel cell system development effort. Work began in October of 1978 under the joint sponsorship of DOE and Engelhard; some activity was carried out earlier in 1978 under a preliminary spending agreement. This report covers work conducted through the first quarterly period - to December 31, 1978.

The tasks, overall objectives, and specific goals of this contract are shown in Tables I, II, and III, respectively. The general approach to Task I is shown in encapsulated form in Tables IV, V and VI.

The primary emphasis to-date has been placed on Task I - the stack component development effort. In the bipolar plate area, initial single-cell testing has utilized a carbon-sealed material that had been evaluated under a previous contract. Results have been very satisfactory, but development effort has focused on different approaches because of the projected high cost of this material.

The matrix effort in the early stages was directed toward modification of the existing membrane-type Engelhard matrix. Recently, activity has been shifted toward the laminated-type matrix because of apparent advantages in terms of high-temperature stability, cell-IR, and projected manufacturing cost.

Most of the electrode evaluations have utilized 30% Pt on carbon catalysts with a nominal loading of 1.4 mg Pt/cm². Recently, 10% Pt on carbon catalysts have been evaluated at a nominal loading of 0.46 mg Pt/cm². Catalysts are being developed under Task V, a separate program wholly sponsored by Engelhard.

Single-cell testing in sub-scale (3 inch x 3 inch) cells is being carried out as the primary diagnostic for components being developed under Task I. Two 5-KW stacks (nominal one square foot area) will be constructed and tested later in the program. Several sub-stacks (about 12 cells each) with the same cell size will be evaluated prior to the 5-KW stacks.

A 5-KW fuel processor will be constructed and tested under Task II. Initial activity consisted of thermodynamic equilibria determinations for each of three candidate fuels: natural gas (methane), naphtha, and methanol. Also, preliminary system analysis is being conducted in order to provide rationale for fuel selection for the Task II and Task III programs. Sub-scale reformer test facilities are being prepared.

The main system analysis and design activity will begin upon selection of a fuel - Task III. A scale-up program for the phosphoric acid stack components will begin in mid-1979 - Task IV.

TASK I - PHOSPHORIC ACID STACK COMPONENT DEVELOPMENTBIPOLAR PLATES

The most extensive effort to-date in the bipolar plate area has involved multi-element plates. The initial approach is utilizing the non-grooved "ABA" configuration illustrated schematically in Figure 1. The technical goals of the ABA program are presented in Table VII. Candidate A-element and B-element processing schemes are shown in Figure 2.

Figure 3 shows a flow versus pressure-drop relationship that applies to packed beds. This correlation has been utilized in studying pressure-drop behavior in A-elements. This leads to the conclusion that a relatively large fiber diameter is required in order to achieve acceptably low air ΔP 's through non-grooved A-elements. Reticulated vitreous carbon satisfies this requirement, and various forms of this material are being evaluated.

Experimental data on reticulated vitreous carbon A-element samples upgraded to varying degrees with CVD carbon are presented in Figure 4. The flow- ΔP data were obtained in apparatus as shown in Figure 5. The results indicate that acceptably low ΔP 's can be achieved in RVC materials upgraded with CVD carbon to the point that mechanical, electrical and thermal properties meet program goals.

B-element samples based on woven precursors were evaluated for hydrogen-impermeability. Tests were conducted at approximately 200°C in the test apparatus shown in Figure 6. The results are shown in Figure 7. The hydrogen-containing chamber was equilibrated at temperature prior to sealing off the hydrogen within at a pressure of about 4 psig. These results indicate that in three of the samples hydrogen-impermeability had been attained. Treatments

had included both resin impregnation and CVD carbon deposition (see Figure 2). The slight rise in pressure with time in samples A-1 and A-3 merely reflects a slight increase in chamber temperature over the course of the test.

The through-plane electrical resistivities were determined as shown in Figure 8. This method provides for measurement of voltage-drop across a plate material by direct contact between the plate and voltage probes which penetrate the current-collecting plates through very narrow holes. In this manner, through-plane resistance readings for relatively high conductivity materials can be obtained without the complication of contact losses.

The potentially-viable A and B elements now in hand will be prepared in large enough quantities and sizes to allow small stack trials (nominal 0.5 ft² area). Edge-sealing of the A-elements will be accomplished as shown in Figure 9. Also, both A-elements and B-elements are being examined for phosphoric acid tolerance in potentiostatic aging stations as illustrated in Figure 10; the conditions are 204°C and 1.0 volt (versus RHE). The first results will be obtained in January.

In the non-ABA area several samples are now on test to evaluate phosphoric acid tolerance. These tests are being conducted under the same conditions as those for the A and B elements, above. Samples of the following materials are being examined:

- (1) Kearney Industries CO-40 (proprietary) resin impregnated graphite (found to be a potential candidate on the basis of oven tests for acid tolerance).

(2) Polyphenylene sulfide/graphite (also found to be a potential candidate on the basis of oven tests for acid tolerance).

(3) Phenolic/graphite.

The polyphenylene sulfide/graphite showed very poor compatibility at 204°C and 1.0 volt, and testing of this material has been terminated. A Kearney Industries sample had a weight loss of 0.7% after 1000 hours. This does not meet the requirements of 0.1% weight loss but testing is continuing to determine if the rate of loss decreases. Data on the phenolic/graphite will become available during January.

MATRIX

The matrix development effort includes activity directed toward modification of the existing Engelhard matrix as well as toward new formulations. Within the existing (membrane type) matrix area, modified filler materials and compositional variations were examined.

A formulation based on an aqueous gel of phosphated zirconium salt demonstrated single-cell open-circuit voltage levels and stability that were above average - see Figure 11. However, this formulation showed evidence of creep in the form of progressively increasing IR-losses. After almost 700 hours of running on load, a retorquing operation was attempted to reduce the IR. This was unsuccessful, and the open-circuit voltage was adversely affected. The properties of the gel-based membrane will be altered in an effort to alleviate the creep problem.

An increase in acid content of the membrane was studied as a means of improving open-circuit voltage stability and high-temperature tolerance. Figure 12 shows data acquired in a single-cell test using a membrane with 20% higher acid content. Open-circuit voltage was found to be superior over the first 500 hours in a single-cell test; but a progressive increase in cell-IR also occurred in this cell, and the test was terminated. No further work is presently being devoted to this approach.

New matrix development activity includes the examination of various candidate filler materials. A number of samples have been received; these are primarily inorganic carbides and nitrides. These will be screened in terms of phosphoric acid tolerance.

Work in the non-membrane - laminate-type - matrix area has involved the use of silicon carbide filler materials thus far. These new matrix elements have not yet been perfected, and pin-holes are sometimes observed. Open-circuit voltages (although acceptable) are generally lower initially than those of the membrane-type; however, these tend to be more stable, and there appears to be greater tolerance of higher temperatures. Small samples taken at random have passed a 10 in. H₂O bubble-pressure screening test, carried out in the apparatus shown in Figure 13.

Acid acceptance and wicking properties appear to be good. Matrix elements have been quite thin (0.008-0.010 inches). It has been determined that with the cell initially charged with no excess acid - so that acid deficiencies could be expected within a few days - later addition of acid tends to restore initial open-circuit voltage levels. Acid-storage configurations utilizing the A-element of an ABA bipolar plate will be evaluated for incorporation into this scheme. Initial results will be obtained during January.

This matrix appears to provide a substantial benefit with regard to cell-IR. The IR-losses at 150 amp/ft² are commonly 40 mv, as opposed to approximately 70 mv for the membrane type.

ELECTRODES

Initial electrodes utilized 30% Pt on C catalysts and had a nominal loading of 1.4 mg Pt/cm². Representative data for acceptable catalyst batches (see "Catalyst Studies," below) are shown in Table VIII. In recent testing 10% Pt on C catalysts have been used, and the nominal electrode loading (each) is 0.46 mg Pt/cm². Figure 14 shows the effect on performance of varying the platinum concentration of the catalyst while keeping the total catalyst loading (Pt + C) the same. The catalyst recipe and electrode preparation procedure had been optimized based on the 30% Pt on C catalyst. Accordingly, the performance of the 30% Pt on C cell is near average, but that of the 10% Pt on C cell is about 10-15 mv lower than typical performance of recent cells.

A representative performance plot for a 10% Pt on C catalyst cell (0.46 mg Pt/cm² each electrode) is shown in Figure 15 for a cell temperature of 163°C. Activity based on the IR-free oxygen performance at 0.9 volts is about 9 ma/mg Pt. The combined losses due to ohmic and diffusion factors are about 15 mv at 150 amp/ft².

Figure 16 shows a performance plot for a cell with similar electrodes but using a laminate-type matrix. With minor differences this performance resembles that of the cell of Figure 15 that used a standard (membrane-type) matrix. Figure 17 shows a plot of cell voltage versus current-density for the same cell. Cell voltage is somewhat higher than that of the cell using the membrane-type

matrix because of lower IR-losses. Recent cells have shown IR-losses that are substantially lower yet - averaging about 40 mv at 150 amp/ft². Consequently, cell voltages have been about 640 mv at 150 amp/ft².

Electrodes have been prepared by co-depositing catalyst and Teflon onto porous, wetproofed carbon-fiber substrates according to Engelhard-developed techniques. Most of the program has been conducted using electrode substrates of the woven-precursor type (Pfizer). Because of the substrate's large macropores the catalyst layer typically penetrates into or through this element to a certain degree, resulting in incomplete utilization of the catalyst. This is illustrated in the cross-sectional micrographs of Figure 18. Improvement of this situation is being sought through the use of finer macropore substrates.

Substrates of the non-woven precursor type (Stackpole) are presently being examined. These yield catalyst layers that have improved continuity; however, wetproofing of these substrate types has not yet been optimized. Figures 19 and 20 show the acid uptake of substrate samples as a function of fluorocarbon loading. Different trends are observed with the two respective wetproofing agents. Acid uptake decreases with increasing TFE-30 loading (Figure 19), but increases with increasing FEP-120 loading (Figure 20).

Further testing will determine acid uptake at lower fluorocarbon levels. Substrate samples will then be selected for single-cell electrode trials. Results should be obtained during January.

CATALYST STUDIES

Characterization studies have been conducted primarily on the 30% Pt on C and 10% Pt on C catalysts. Some analysis of fresh catalysts of intermediate platinum concentration has also been performed. The basic technique used for catalyst characterization has been cyclic-voltammetry. The test cells for these studies were fabricated from Teflon blocks as shown in Figure 21. The construction of the electrode holder is shown in Figure 22. The electrical schematic for the cyclic-voltammetry apparatus is presented in Figure 23.

A characteristic cyclic-voltammogram is shown in Figure 24. A platinum black catalyst is used for this example to accentuate the current peaks that reflect the Faradaic processes occurring during sweeps between the hydrogen potential and the oxygen potential. The area of the two hydrogen deposition peaks (cathodic) is used to compute the active metal area (by way of the corresponding net surface charge as indicated in Figure 24).

Table IX shows the electrochemical area obtained by cyclic-voltammetry for catalysts over a wide range of platinum concentration. Corresponding single-cell data are also shown. A useful correlation between electrochemical area and single-cell performance is shown in Figure 25. Figure 26 shows the cyclic-voltammograms obtained on four electrodes with Pt concentrations in the catalyst ranging from 10% to 30%. Platinum loading varied proportionately since the total catalyst loading (Pt + C) was kept constant. The total platinum surface-areas - as determined from the hydrogen deposition peaks - varied in proportion to loading, as expected. However, the highest specific surface-area ($\text{m}^2/\text{g Pt}$) was obtained in the case of the lowest platinum concentration catalyst; i.e., 10% Pt on carbon.

Analysis of aged 30% Pt on carbon catalysts has indicated substantial surface-area loss. Figure 27 shows cyclic-voltammograms for a cathode before and after a 2040-hour single-cell test. The apparent surface-area loss is about 65%. Figures 28 and 29 show transmission electron micrographs of the same cathode before and after the test, respectively. These indicate a high degree of platinum sintering, in accord with the above cyclic-voltammetry results. Figure 30 shows histograms of platinum particle-size distribution for another 30% Pt on carbon cathode catalyst - both fresh and after 2000 hours of running in a single-cell test. These data, obtained from TEM analyses, indicate substantial loss of crystallites in the 0-40 Å range.

Potentiostatic aging results (see Figure 10) indicate that platinum surface-area loss of 30% Pt on carbon catalysts at 204°C and 0.7 volts takes place quite rapidly. The cyclic-voltammograms of Figure 31 show a reduction of platinum surface-area of almost 50% after only 48 hours.

Analysis to-date on 10% Pt on C catalysts indicates that platinum surface-area loss in both single-cell and potentiostatic aging tests is less severe than that of 30% Pt on carbon catalysts. This is partially due to the fact that the initial surface-areas are higher; thus, even in cases where the percentage area reduction is the same, the 10% Pt on carbon catalysts retain higher area after aging.

Considerable experimental data on corrosion and platinum sintering in 10% Pt on carbon catalysts are being accumulated, and these phenomena will be quantified accordingly as the program proceeds. Electrocatalyst development efforts are being focused toward retention of platinum surface-area under the conditions employed in this program. Characterization results for experimental catalysts developed in Task V will be reported under Task I.

TASK II - FUEL PROCESSING SUB-SYSTEM DESIGN AND DEVELOPMENT

THERMODYNAMIC ANALYSIS

A computer program suitable for acquisition of thermodynamic equilibrium compositions in the fuel processing area has been developed and checked out. APL programming was used. Random checks indicate virtual agreement with published data on thermodynamic equilibria.

The specific steam-reforming equilibrium calculations for three fuels - methane, methanol, and naphtha - have been completed. The equilibrium gas compositions were determined as a function of temperature and H_2O/C ratio. Representative results are illustrated in Figures 32, 33, and 34. Heat requirements for the endothermic steam-reforming reaction were also determined.

PRELIMINARY SYSTEM ANALYSIS

Preliminary system analysis is being conducted for the purpose of providing rationale for the selection of a primary fuel to be used in the Task II and Task III programs. A meeting was held with DOE managers at Engelhard December 12, 1978 to discuss the issue of fuel selection, specifically as this affects life-cycle cost and national energy policy. Table X shows preliminary results based on system analysis to-date for the three candidate fuels and LPG. Further system analysis is proceeding.

STEAM-REFORMER DEVELOPMENT

The design of experimental apparatus for sub-scale fuel processing studies has been completed. A location has been selected for this phase of the experimental program within the R&D Building, Menlo Park. Facilities modifications are in progress.

Equipment and parts for this experimental test station are on order. The design was tailored to provide enough flexibility to accommodate any one of the candidate fuels. (Figure 35 shows a flow schematic applicable to the use of natural gas as fuel.) Minor modifications will be required depending on the fuel choice.

TASK III - INTEGRATED SYSTEM DESIGN AND DEVELOPMENT

The main Task III effort will begin upon selection of fuel.

TASK IV - LARGE STACK DESIGN AND DEVELOPMENT

The Task IV effort is scheduled to begin in mid-1979.

TASK V - ELECTROCATALYST DEVELOPMENT

The development of electrocatalysts for the Task I program is being carried out under Task V, a separate program wholly sponsored by Engelhard. Characterization results for these catalysts will be reported under Task I.

TABLE I

PHOSPHORIC ACID FUEL CELL STACK AND SYSTEM DEVELOPMENT

DOE CONTRACT # DE-AC-03-79ET15366

TASK I.	PHOSPHORIC ACID STACK COMPONENT DEVELOPMENT
TASK II.	FUEL PROCESSING SUB-SYSTEM DESIGN AND DEVELOPMENT
TASK III.	INTEGRATED SYSTEM DESIGN AND DEVELOPMENT
TASK IV.	LARGE STACK DESIGN AND DEVELOPMENT
TASK V.	ELECTROCATALYST DEVELOPMENT (WHOLLY SPONSORED BY ENGELHARD)

TABLE II

OVERALL OBJECTIVES

TASK I : LOW-COST, HIGH-EFFICIENCY FUEL CELL STACKS

- 0.65 VOLT/CELL @ 150 mA/cm², H₂/AIR
- \$200/KW STACK COST (PROJECTED)
- 5-YEAR STACK LIFE (PROJECTED)

TASK II : DESIGN, CONSTRUCTION, AND TESTING OF FUEL
PROCESSING SUB-SYSTEM

TASK III : OVERALL DESIGN OF INTEGRATED 5-KW
HYDROCARBON/AIR PHOSPHORIC ACID
FUEL CELL SYSTEM

TASK IV : DEVELOPMENT OF COMPONENT PROCESSING PROCEDURES
FOR LARGE-AREA CELLS

TABLE IIISPECIFIC GOALSTASK I - PHOSPHORIC ACID STACK COMPONENT DEVELOPMENTBIPOLAR PLATES

- STABILITY IN CONCENTRATED H_3PO_4 @ 204°C & 1.0 VOLT
- 3 mV IR-LOSS @ 150 mA/cm²
- \$4/ft²

MATRIX

- OPERABILITY @ 204°C
- 30 mV IR-LOSS @ 150 mA/cm²
- \$3/ft²

ELECTRODES

- 0.68 VOLT/CELL IR-FREE @ 150 mA/cm², H_2 /AIR
- 1.0 mg/cm² P.M. LOADING, TOTAL CELL
- \$4/ft² NON-P.M., TOTAL CELL

COOLING UNITS

- \$3/ft² (CELL AREA)

TABLE III (CONTINUED)

SPECIFIC GOALS

TASK II - FUEL PROCESSING SUB-SYSTEM DESIGN AND DEVELOPMENT

- COST-EFFECTIVENESS (PROJECTED)
- PRODUCT QUALITY

TASK III - INTEGRATED SYSTEM DESIGN AND DEVELOPMENT

- COST/THERMAL-EFFICIENCY OPTIMIZATION
- LOAD-FOLLOWING CAPABILITY

TASK IV - LARGE STACK DESIGN AND DEVELOPMENT

- CONFORMITY WITH SMALL CELL PERFORMANCE CHARACTERISTICS
- COST GOALS OF TASK I

TABLE IV

APPROACH

BIPOLAR PLATES

- RESIN-BONDED AND RESIN-SEALED GRAPHITES
- CARBONIZED RESINS
- MULTI-ELEMENT TYPES

TABLE V

APPROACH

MATRIX

- MODIFICATIONS OF EXISTING MATRIX
- NEW FORMULATIONS (MEMBRANES, LAMINATES)
- HIGH-TEMPERATURE OPERATION
- REDUCED THICKNESS
- ACID-STORAGE MODES

TABLE VI

APPROACH

ELECTRODES

- ADVANCED CATALYSTS
- ELECTRODE STRUCTURAL OPTIMIZATION
- HIGH-TEMPERATURE OPERATION

TABLE VIITECHNICAL GOALS FOR NON-GROOVED ABA BIPOLAR PLATEA - ELEMENT

IR-DROP	< 1 MV @ 150 ASF
THERMAL CONDUCTIVITY	> 2 BTU/HR FT ² (°F/FT)
AIR PRESSURE DROP	< 2" H ₂ O @ 150 ASF, 2X STOIC.

B - ELEMENT

IR-DROP	< 1 MV @ 150 ASF
THERMAL CONDUCTIVITY	> 5 BTU/HR FT ² (°F/FT)
H ₂ PERMEATION	< EQUIV. OF 0.5 ASF

COMPOSITE COST GOAL

< \$4/FT² IN QUANTITIES OF 1,000,000/YR

TABLE VIII

SUMMARY OF SINGLE-CELL RESULTS

30% Pt ON CARBON - 1.4 mg/cm²

<u>CATHODE CATALYST</u>	<u>RUN NO.</u>	<u>RUN TIME (hr)</u>	<u>O₂-GAIN(mV)*</u>	<u>VOLTAGE(mV, IR-FREE)*</u>
10864-7	283	24	77	708
		200	77	712
10864-39	344	24	94	703
		200	80	708
10914-02	353	24	97	697
		200	79	707
10914-22	399	24	84	715
		125	80	723
10914-23	402	50	82	716
		200	77	714

* At 150 AMP/FT², 163°C

TABLE IX

ELECTROCHEMICAL AREA BY CYCLIC-VOLTAMMETY

ELECTROCATALYST	ELECTRODE	Pt CONC. %	Pt LOADING (mg/cm ²)	Pt ELECTOCHEMICAL AREA		H ADSORPTION PEAKS	IR-FREE CELL VOLTAGE (mV)**
				cm ² /SAMPLE	m ² /gPt		
Pt Black		100	6.2	473	24	Very Good	
10914-16	377	30	1.4	314.6	71.5	Good	698
10914-22	397	30	1.4	330	75	Good	715
10914-23	399	30	1.4	390	88.7	Good	718
10914-25	401	30	1.4	404.8	92	Good	
10914-32	419	20	0.93	304	89	Good	692
10914-33	421	15	0.70	218	99	Good	682
10914-09	357	15	0.70	55	25	Very Poor	N.A.
10914-12	365	10	0.46	177	120	Good	671
10914-10	359	10	0.46	64.7	44	Very Poor	524
10914-11	363	10	0.46	95.5	65	Poor	
10914-15	369	10	0.46	184	125	Good	671
10914-16	369	10	0.46	176.4	122	Good	
10914-18	385	10	0.46	80.8	55	Good	628
10914-20	387	10	0.46	76.4	52	Fair	602
10914-21	389	10	0.46	169	115	Good	666
10914-21	389	10	0.46	154	105	Good	

** After 24 hours, 150A/FT², 163°C

TABLE XPRELIMINARY SYSTEM ANALYSIS RESULTSFOR VARIOUS FUELS

A. ASSUMPTIONS

	FUEL			
	<u>METHANE</u>	<u>METHANOL</u>	<u>NAPHTHA</u>	<u>LPG</u>
1. AUXILIARY POWER (%)	12	8	14	12
2. INVERTER EFFICIENCY	95	95	95	95
3. H ₂ LOSS	0	0	0	0
4. H ₂ UTILIZATION IN FUEL CELL (%)	80	88	80	80
5. VOLTAGE EFFICIENCY (% HHV)	43.9	43.9	43.9	43.9
6. REFORMER OPERATING TEMP. (°F)	1400	400	1400	1400
7. PRICE OF FUEL	25¢/CCF	44¢/GAL	38¢/GAL	35¢/GAL
<hr/>				
B. FUEL COST (¢/KW HR)	2.9	4.2	3.6	3.1
<hr/>				
C. SYSTEM CAPITAL COST (\$/KW)	646	550	659	646
<hr/>				
D. LIFE-CYCLE COST (¢/KW HR)	5.3	6.1	6.0	5.5

FIGURE 1

A - B - A

CONSTRUCTION

PT
10/24/78

FIGURE 2
CONVERSION PROCESS OUTLINE -
PRECURSOR TO FINISHED ELEMENT

PK
12/5/78

PRESSURE DROP CORRELATION

$$p_1^2 - p_2^2 = \frac{2zRG^2T}{g_c M} \left(\frac{2f_m L (1-\epsilon)^{3-n}}{\phi_s^{3-n} \epsilon^3 D_p} \right)$$

- p = PRESSURE, PSF
 z = COMPRESSIBILITY FACTOR, 1.0034 (AIR @ 500K, 1 ATM)
 R = GAS CONSTANT, 1546 FT LB_f / LB MOLE °R
 G = FLOW IN EMPTY CHAMBER LB / SEC FT²
 T = TEMPERATURE, °R
 g_c = GRAVITATIONAL CONSTANT, 32.17 FT LB / LB_f SEC²
 M = MOLECULAR WEIGHT, 30
 f_m = FRICTION FACTOR
 L = LENGTH OF BED, FT
 ϵ = VOID FRACTION (≈ 0.8)
 n = 1.0, SEE BELOW
 ϕ_s = SHAPE FACTOR (≈ 0.4)
 D_p = AVERAGE PARTICLE DIA

Fig. 3-49. Friction factor for beds of solids. (Lena, "Fluidization," p. 49, McGraw-Hill, New York, 1959)

PF
 2/5/78

FIGURE 4

A-ELEMENT IN-PLANE AIR PRESSURE-DROP,
3 INCH x 3 INCH SAMPLE

ENGELHARD MINERALS & CHEMICALS
CALCULATIONS

ENGELHARD

SUBJECT _____

SHEET NO. _____ OF _____

FILE _____

DATE _____

BY _____

FIGURE 5

FLOW VERSUS ΔP APPARATUS

127 10 30 69

FIGURE 6

HYDROGEN PERMEABILITY TEST APPARATUS

Chamber Pressure - PSIG

SAMPLE AVG. TEMP. (°C)

+	(A-1)	215
X	(A-3)	220
○	(B-2)	208
Δ	(B-4)	190

FIGURE 7

HYDROGEN LEAKAGE TEST

Time - Hours

R.A. SMITH 12/6/78

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CALCULATIONS

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DATE _____

BY _____

FIGURE 8

ELECTRICAL CONDUCTIVITY MEASUREMENT APPARATUS

V_{A-D} = VOLTAGE DROP ACROSS SAMPLE

TEST CHECK

$$V_{B-C} = (V_{A-C}) + (V_{B-D}) - (V_{A-D})$$

PT
10/24/78

FIGURE 9

ABA EDGE-SEALING

RT
10/24/78

FIGURE 10
POTENTIOSTATIC AGING TEST CELL

AC
10/24/78

FIGURE 11

Open-Circuit Voltage Stability for Experimental Matrix Using Aqueous Gel (#25)

Temp: 325°F (163°C)

MF
10/24/1

FIGURE 12

Temp: 325°F (163°C)

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FIGURE 13

BUBBLE-PRESSURE TESTING APPARATUS

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FIGURE 14

AS 8014-01
10 X 10 TO THE CENTIMETER
VOLTAGE
(IR-FREE)
AT 150 ASF

VARIATION OF FUEL CELL VOLTAGE WITH PLATINUM LOADING

TOTAL CATALYST LOADING - 4.6 mg/cm^2

TEMP: 163°C

50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
950
1000
1050
1100
1150
1200
1250
1300
1350
1400
1450
1500
1550
1600
1650
1700
1750
1800
1850
1900
1950
2000
2050
2100
2150
2200
2250
2300
2350
2400
2450
2500
2550
2600
2650
2700
2750
2800
2850
2900
2950
3000
3050
3100
3150
3200
3250
3300
3350
3400
3450
3500
3550
3600
3650
3700
3750
3800
3850
3900
3950
4000
4050
4100
4150
4200
4250
4300
4350
4400
4450
4500
4550
4600
4650
4700
4750
4800
4850
4900
4950
5000
5050
5100
5150
5200
5250
5300
5350
5400
5450
5500
5550
5600
5650
5700
5750
5800
5850
5900
5950
6000
6050
6100
6150
6200
6250
6300
6350
6400
6450
6500
6550
6600
6650
6700
6750
6800
6850
6900
6950
7000
7050
7100
7150
7200
7250
7300
7350
7400
7450
7500
7550
7600
7650
7700
7750
7800
7850
7900
7950
8000
8050
8100
8150
8200
8250
8300
8350
8400
8450
8500
8550
8600
8650
8700
8750
8800
8850
8900
8950
9000
9050
9100
9150
9200
9250
9300
9350
9400
9450
9500
9550
9600
9650
9700
9750
9800
9850
9900
9950
10000

720
700
680
660
640
620
600

0 5 10 15 20 25 30 % Pt on Carbon

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FIGURE 15

SINGLE-CELL PERFORMANCE OF 10% Pt on CARBON ELECTRODES

IR-FREE
CELL VOLTAGE

ENGELHARD

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FIGURE 16

SINGLE-CELL PERFORMANCE OF 10% Pt ON CARBON ELECTRODES

IR-FREE
CELL VOLTAGE

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FIGURE 17

SINGLE-CELL PERFORMANCE FOR 10% Pt ON CARBON ELECTRODES

CELL
VOLTAGE

Cathode: 0.46 mg Pt/cm²
Anode: 0.46 mg Pt/cm²
Matrix: Laminated

Temp: 325°F (163°C)
Time: 24 Hours

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(x 50)

(x 200)

FIGURE 18

CROSS-SECTION OF FUEL CELL ELECTRODE SHOWING CATALYST LAYER AND WOVEN SUBSTRATE

FIGURE 19
ACID UPTAKE OF NON-WOVEN SUBSTRATE
 Wetproofing: T-30 Teflon
 Temperature: 400°F (204°C)
 Time: 24 Hours

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FIGURE 20

ACID UPTAKE OF NON-WOVEN SUBSTRATE

Wetproofing: FEP-120
 Temperature: 400°F (204°C)
 Time: 24 Hours

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FIGURE 21

CYCLIC-VOLTAMMETRIC TEST CELL (TEFLON)

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FIGURE 22

ELECTRODE HOLDER SHOWING THE ASSEMBLY OF THE TEST ELECTRODE

FIGURE 23

ELECTRICAL ARRANGEMENT FOR CYCLIC VOLTAMMETRY

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FIGURE 25

Single Cell Voltage vs. Electrochemical Area Of Pt

IR-Free Cell Voltage (mV at 150 A/ft², 163°C)

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FIGURE 26

Cyclic Voltammogram of Electrocatalysts with Various Pt Concentrations

Sweep Rate: 20 m V/s
25% H_3PO_4
Room Temp.

FIGURE 27

Cyclic Voltammograms of 30% Pt on Carbon Cathodes (Catalyst 10794-9)

Sweep Rate: 10 mv/s

25% H_3PO_4

Room Temp.

1: Virgin Electrode: $63 \text{ m}^2/\text{g}=S_o$

2: Electrode after 2040 Hrs in
single-cell ($150 \text{ A}/\text{ft}^2$, 163°C):

$22 \text{ m}^2/\text{g}=S_t$

$$\frac{S_o - S_t}{S_o} = 65\%$$

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FIGURE 28

CATHODE #178, FRESH (x246,000)

FIGURE 29

CATHODE #178 AFTER 2040 HRS. (x246,000)

NO.
OF
PARTICLES

HISTOGRAM OF PLATINUM PARTICLE SIZE DISTRIBUTION

40

30

%

20

10

0

FRESH ELECTRODE: CATHODE 184

Pt LOADING: 1.4 mg/cm^2

30% Pt on Carbon

40

%

30

20

10

AGED ELECTRODE: CATHODE 184

TIME: 2000 HOURS

TEMPERATURE: 325°F (163°C)

CURRENT DENSITY: 75 AMP/FT^2

0 20 40 60 80 100 120 140 160 180 200 220 240 Å

PLATINUM PARTICLE SIZE

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FIGURE 31

Cyclic Voltammogram of 30% Pt on Carbon Cathode

Sweep Rate: 10 mv/s

25% H_3PO_4

Room Temp.

Catalyst: 10914-23

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FIGURE 33

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METHANOL STEAM REFORMING
EQUILIBRIUM COMPOSITION
 $H_2O/CH_3OH=1.5$, 1ATM

JW
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FIGURE 35