

SEVENTH QUARTERLY REPORT

On

ALKALINE BATTERY SEPARATOR
CHARACTERIZATION STUDIES

(28 April 1969 - 28 July 1969)

J. J. Kelley and S. Orenstein

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For

GODDARD SPACE FLIGHT CENTER

CONTRACT NAS 5-10418

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Prepared by
ESB INC. RESEARCH CENTER
Yardley, Pennsylvania 19067

For

GODDARD SPACE FLIGHT CENTER
Greenbelt, Maryland 20771

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ALKALINE BATTERY SEPARATOR CHARACTERIZATION STUDIES

by

J. J. Kelley and S. Orenstein

ABSTRACT

Increased emphasis was placed on the evaluation of the absorber component of conventional alkaline battery separator systems as well as the examination of the procedures used to affect the evaluation. The methods used to measure electrolyte wetting and wicking characteristics of separators were refined and comparative data was obtained. Separator samples of non-woven polyamide, polypropylene and dynel, as well as asbestos, were examined. Scouring, which removed textile processing aids and added wetting agents, made the polyamide samples unable to wick electrolyte. Samples of the scoured material treated to wet in the absence of added wetting agents only partially restored original wicking ability. The treated samples were found to contain large void areas which were not characteristic of the original material.

Battery testing of the absorber samples in sealed, nickel-cadmium cells has begun. The 90 minute earth orbit type cycle being used is at 25 percent depth of discharge.

Membrane type separators under test are: 1) a pva grafted polyethylene, RAI 191-17-1; 2) polyvinyl alcohol film - Monosol-1-001-3, and a carboxylic acid grafted cross linked polyethylene SWRI 4-359 GX. The results of characterization testing are reported.

In the membrane testing, a voltammetric method using membrane covered mercury electrode for measuring zincate diffusion rates through the membranes is described and compared to that previously used.

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1.0 INTRODUCTION

This marks the beginning of extended work on Contract NAS 5-10418. The efforts of the past six quarters have been concentrated on finding and evaluating the new separator materials for application to silver-zinc batteries. Though this is still the primary concern of this work, a major effort will be made to revise certain of the methods used previously and to develop improved, new procedures. This will hopefully lead to a better understanding of the parameters which affect the role of separators in extending cycle life and battery performance.

Progress in this area has been made during this quarter. A new method for determination of zincate permeability is reported in Section 2.1.1. Application of a membrane covered, mercury electrode with voltametric circuitry permits simultaneous measurement of the electrochemical flux and the diffusion coefficient of zincate across the test membrane. In addition, the apparatus for zinc penetration time and electrolyte wicking rate have been refined to eliminate the most frequently observed errors.

Four membrane and fifteen absorber materials have been received for evaluation. Bench screening tests are underway as are preparations for the testing of membrane samples in Ni-ZnO and Ag-CdO cells. A brief description of these materials may be found in Tables 1 and 2.

Table 1

Membrane Materials

<u>Style Number</u>	<u>Composition</u>	<u>Supplier</u>
191-17-1	polyethylene with grafted polyvinyl alcohol side chains	RAI Research
4-359 GX	radiation grafted and cross linked polyethylene	Southwest Research Inst.
1-001-3	polyvinyl alcohol	Monosol Inc.
3-001-3	polyvinyl alcohol	Monosol Inc.

Table 2

Absorber Materials

<u>Style Number</u>	<u>Composition</u>	<u>Supplier</u>
Pellon 2505WO U-Scoured	Non-woven polyamide, double washed and dried at 70°C	RAI Research
Pellon 2505WO U-Treated	Non-woven polyamide treated by unrevealed process	RAI Research
Pellon 2505WO U-Treated and Scoured	Non-woven polyamide double washed, dried, and treated	RAI Research
Pellon 2505WO E-Scoured	Non-woven polyamide double washed and dried	RAI Research
Pellon 2505WO E-Treated	Non-woven polyamide treated by unrevealed process	RAI Research
Pellon 2505WO E-Treated and Scoured	Non-woven polyamide double washed, dried and treated	RAI Research
2505 ML	Non-woven polyamide	Pellon Corp.
301	Non-woven polypropylene	Pellon Corp.
EM476	Non-woven polypropylene	Kendall Mills
E1451	Non-woven polypropylene	Kendall Mills
M1450	Non-woven Dynel	Kendall Mills
M1410	Non-woven Dynel	Kendall Mills
M1401	Non-woven Dynel	Kendall Mills
E1488	Non-woven Dynel	Kendall Mills
RD651	Non-woven asbestos	Score, Inc.

Previous attempts at evaluating absorber materials on the basis of wetting characteristics using flooded cells did not provide sufficient differentiation. Provisions have been made during this quarter to test absorber materials in "sealed", aircraft-type, Ni-Cd cells. A goal of 1000 earth-orbit cycles has been set as a temporary minimum. The design of the cell is such as to provide conditions where the absorber must wet out and wick electrolyte to the cell parts. Materials which demonstrate different wetting properties in bench screening tests should carry this behavior over to performance in operating cells.

We have found previously that only partial characterization of some materials has been possible because only limited quantities of material could be obtained. A minimum of two square meters has been found necessary to complete bench screening and cell tests. It has also been found desirable to test material from more than one lot of material. We will, therefore, concentrate on those materials which are supplied in pieces 1 x 2 meter from more than one lot. This will allow us to anticipate the properties of materials which are assembled in cells for testing at NAD Crane. In addition, studies into the effect of breakdown products (inorganic and organic residues from processing) on cell performance will require considerable material.

2.0 SEPARATOR SCREENING METHODS

2.1 Membrane Materials

2.1.1 Zincate Permeability

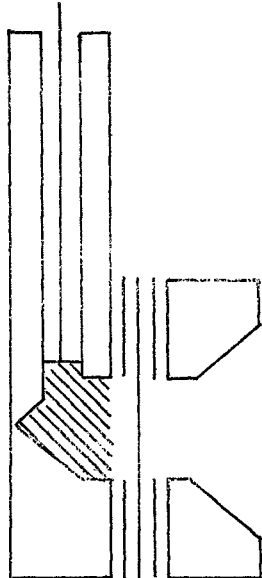
In the past, the diffusion of zincate across a membrane has been measured by the method of Lander (Ref. 6, Chapt. 11). The approach is direct, using electrolyte with and without zincate and the membrane between the two. A zinc-Hg/HgO electrode pair detects changes in zincate concentration. The reference-electrode couple is sensitive to KOH concentration and exhibits 5 to 10 mv drift. Determinations take up to two hours, including assembly of the apparatus.

Systems using a voltametric cell with a cellophane covering mercury electrode have previously been employed to measure diffusion of hydronium, cadmium, and thallos ions in dilute solutions. (1, 2). Application of membrane covered electrodes has also been made as polarographic gas detectors. (4, 5). Figures 1(a) and (b) show the sample holder, voltametric cell and associated circuitry which was used here to determine the zincate permeability of membrane samples.

FIGURE 1

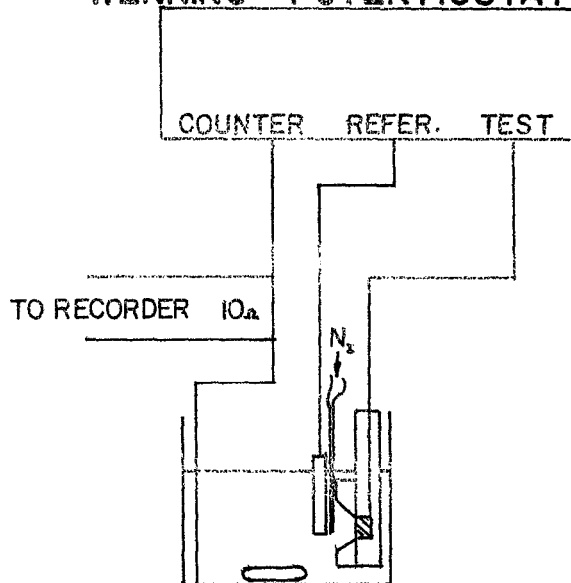
APPARATUS FOR VOLTAMETRIC DETERMINATION OF ZINCATE FLUX USING MERCURY-MEMBRANE ELECTRODE.

SAMPLE HOLDER



a.

WENKING POTENTIOSTAT



b.

The membrane is prepared, as before, by soaking the sample for 16 to 24 hours in 40% KOH. It is then assembled in the holder. The lucite cell of Figure 1(b) uses Hg/HgO and expanded zinc screen electrodes. Bubbling nitrogen is necessary to reduce the residual current to less than 150 μ ampere. The test electrode is potentiostated, with efficient stirring, at -1500 mv vs Hg/HgO, first in 40% KOH, then in 1.0 M ZnO in 40% KOH. A steady state limiting current is reached within 5 minutes. The difference between the two readings is the diffusion current i_d in amperes. The diffusion current can be expressed by:

$$i_d = n F A D \frac{C}{l} \quad (1)$$

where n is the number of equivalents per mole of reacting species, F the Faraday constant, A the geometric area of the electrode in cm^2 , D the diffusion coefficient of the species in cm^2/sec , C the bulk concentration of reacting species in moles/liter and l the membrane thickness in cm. Therefore, the diffusion coefficient of zincate across the membrane may be calculated. Alternatively, the electrochemical flux of zincate across the membrane may be defined as:

$$j = D \frac{C}{l} \quad (2)$$

with units of moles/ cm^2 sec. Rearranging equation (1):

$$j = \frac{i_d}{n F A} \quad (3)$$

for the particular electrode used here, equation (3) is reduced to:

$$j = \frac{i_d}{2.41 \times 10^5}$$

Additional extension of the diffusion current data to ionic mobility within the membrane is possible. (3). However, this will be delayed until the meaning of such data is better understood.

A rough plot of i_d vs zincate concentration is given in Figure 2. The data was taken with PUDO 193 and zincate solutions in 45% KOH. The relationship is linear to below 0.05 M ZnO-KOH. The nature of the residual current is such that no shift in the i_d vs concentration curve is expected when changing KOH concentration. This will be demonstrated in the near future.

The utility of the method can be seen from the scatter of permeability data taken during this quarter. Table 3 gives the average of duplicate measurements of zincate permeability. With the method previously employed, it would not have been feasible to perform similar measurements in duplicate and with second decimal place accuracy. Furthermore, with the former method, the small amounts of zincate diffusing across PERMION 2291 were determined with difficulty because of drift of the zinc-Hg/HgO potentiometric couple. Nevertheless, the average zincate permeability of six lots of material was recently measured at 0.135×10^{-8} moles/cm² sec. using the Lander method. Total time for each voltammetric determination is a maximum of 60 minutes.

Application of this method to the determination of silver permeability is forthcoming. Preliminary data indicates that diffusion currents for the slightly soluble silver species are well within the measurement capability of present instrumentation.

2.1.2 Zinc Penetration

Determinations of the time for a charging zinc surface to penetrate a membrane sample will be completed at a later time. The center plates of the test cells previously used (7) are being modified. The modified plates of Figure 3(a) will allow a platinum detector grid (Figure 3(b)) to be held rigidly in place. Sharp ends from cutting the platinum were folded back over the perimeter wire and welded and cannot puncture the sample. The Teflon spacer no longer requires a channel for the platinum wire. It has been observed that many samples penetrate at the edges. For this reason, the center hole has been relieved to 9/16" to prevent premature shorting by the fixture at this area of preferential growth.

ALKALINE BATTERY SEPARATOR CHARACTERIZATION

NAS 5-10418

CONCENTRATION DEPENDENCE OF i_d USING STANDARD CELLOPHANE
45% KOH

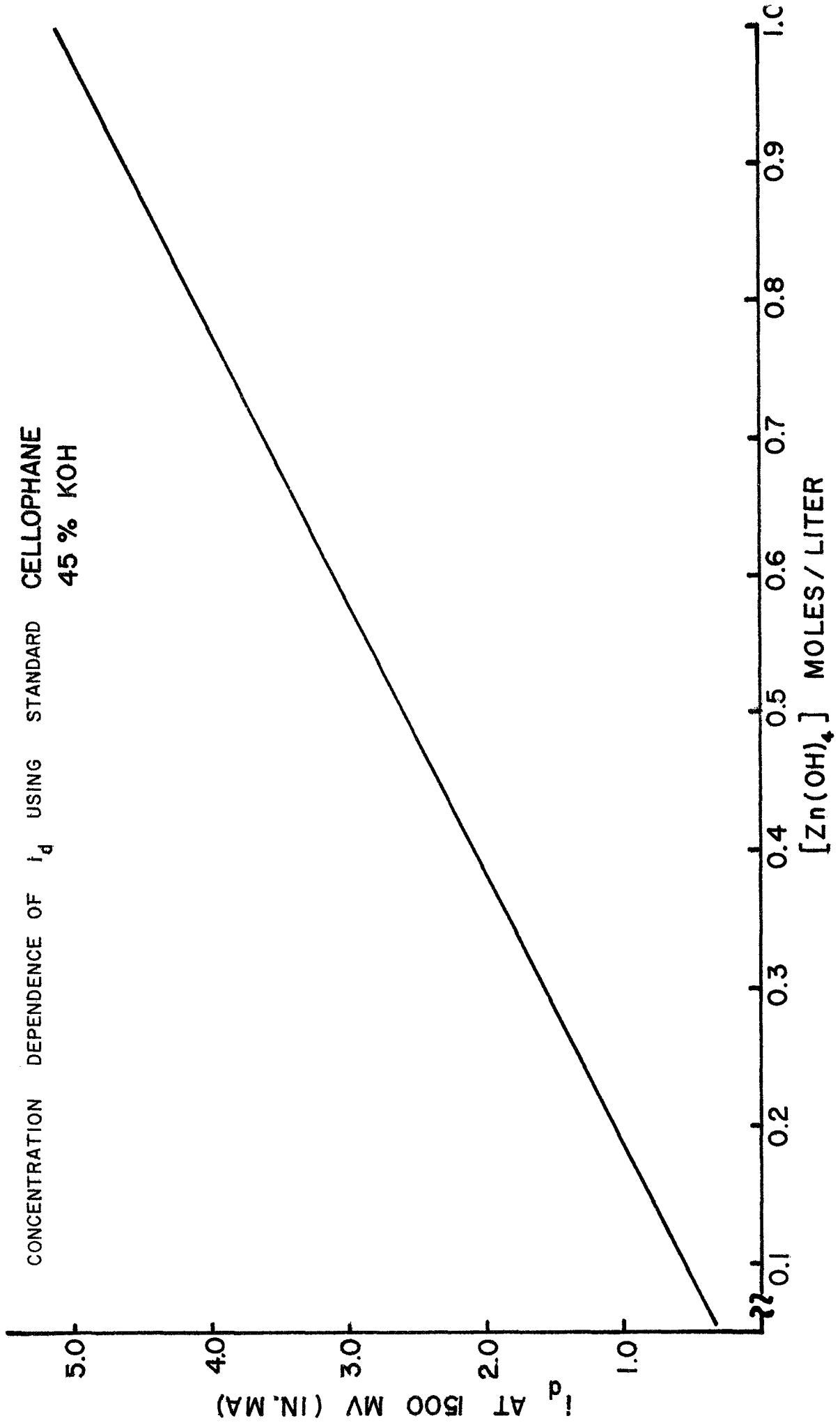
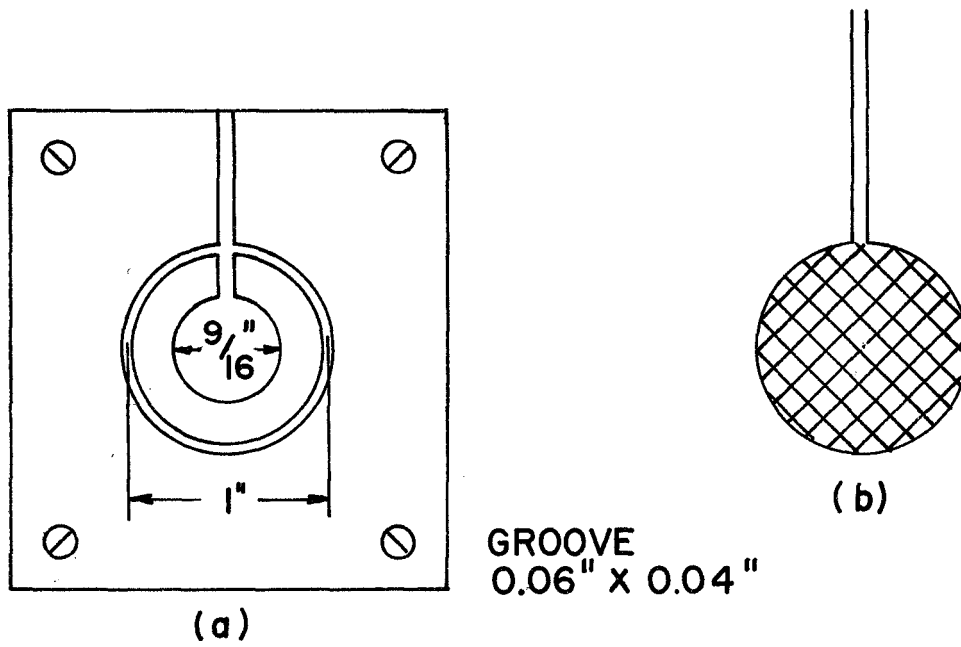


FIGURE 2

FIGURE 3



a - MODIFIED CENTER PLATE

b - PLATINUM DETECTOR GRID

2.1.3 Properties of Membrane Materials

The results of bench screening tests performed on membrane materials during this quarter may be found in Table 3. One new type of material has been obtained. RAI 191-17-1 is a polyethylene containing grafted polyvinyl alcohol side chains. The electrical resistance of this material is slightly lower than some of the acrylic acid grafted. However, the zincate permeability rate is comparable to early acid grafted polyethylenes and is five times greater than some grafted and crosslinked polyethylenes from the same manufacturer (e.g. P2291).

The grafted and crosslinked polyethylene from SWRI is representative of similar materials previously tested. A comparatively low electrical resistance has been obtained at the cost of zincate permeability. The rate of transport and reduction of zincate is so great that clusters of zinc are found on the solution side of the mercury membrane electrode. Similar materials from SWRI have exhibited high zincate permeability measured by the method of Lander (Ref. 6, Chapt. 11). In addition, it is doubtful that mercury penetrated the membrane structure. It is most likely that these clusters of zinc are formed because the rate of transport and reduction of zincate has exceeded the rate of dissolution of zinc in mercury. This resulted in precipitation of the zinc. The fact that no zinc was found in the membrane structure does not rule out this mechanism. Often, during zinc penetration studies, metal can be found in the membrane only with the aid of a microscope. Further investigation into this phenomenon will be made.

The data for PERMION 2291 is taken on samples from material used in constructing silver-zinc cells for cycling at NAD Crane. Characterization tests performed, to date, agree well with determinations on other lots of material.

Table 3. Properties of Membrane Separators

	RAI 191-17-1	SWRI 4-359 GX	MONOSOL 1-001-3 3-001-3		PERMION 2291	PUDO 193
<u>Separator Thickness</u>						
dry (cm x 10 ³)	3.05	3.55	3.05	3.05	3.05	-
in 40% KOH (cm x 10 ³)	3.55	3.80	3.55	3.55	3.30	7.5
% Swell	+16.4	+7.0	+16.4	+16.4	+8.1	-
<u>Zincate Permeability in 1M ZnO = 40% KOH</u>						
i _d (amp x 10 ³)	1.82	17.44	3.23	2.06	0.27	5.95
f moles/cm ² sec x 10 ⁸	0.76	7.24	1.34	0.85	0.113	2.47
D cm ² /sec x 10 ⁵	0.270	2.78	0.476	0.302	0.0373	1.85
<u>AC Resistivity</u>						
Ω-cm	62.92	40.57	22.75	23.71	151.6	-
Ω-cm ²	0.224	0.153	0.081	0.084	0.498	-

2.2 Absorber Materials

2.2.1 Wicking Rate

Wicking determinations on absorbers were done during this quarter in closed lucite chambers 3" x 3" x 8-1/2". Improved wicking rates using this apparatus are shown in Table 4 for two typical materials together with comparative determinations made in the previously described apparatus. The differences noted may be attributed to saturation of the chamber atmosphere with electrolyte vapors. This maintains uniform surface tension within the sample structure by reducing evaporation of wicking electrolyte, stabilizing total meniscus volume and assuring uniform vapor density along the height of the sample.

Table 4
Wicking in 40% KOH

Time (min.)	Maximum Height (in cm)			
	Pellon 2505 KW.		EM 476	
	Open	Closed	Open	Closed
5	0.0	0.0	1.0	2.6
15	0.05	0.06	3.0	5.6
30	0.13	0.17	4.0	> 8.4
50	2.7	4.1	-	-
70	4.4	7.0	-	-

Wicking rates for materials obtained during this quarter appear in Table 5. The data is reported with and across the roll direction in minutes/cm and maximum wicking height (in cm). Maximum height allowed by the sample holder is 8.1 to 8.4 cm. The validity of wicking data for samples which do not reach the top of the holder is doubtful. Slow wicking rates (40 min/cm or longer) are exaggerated by the time necessary for the sample to wet. Materials with poor wetting characteristics do not appear to wet as quickly in a vertical position. However, when mounted horizontally, wet-out times can be measured.

Table 5

RAI-FELTON 2505 WO

Properties of Absorber Materials in 40% KOH	E-T&S	E-T	E-S	U-T&S	U-T	U-S
Wicking Height - cm with roll	1.2	4.9	0.0	3.3	5.8	0.0
Across Roll	2.4	4.5	0.0	2.3	6.7	0.0
Wicking Rate min/cm with roll	500	32.7	DNW ¹	91.0	67.3	DNW
Across Roll	100	35.6	DNW	130.0	62.0	DNW
Wet-out Time min. rough side up	13.0	3.5	184.0	22.6	3.3	136.0
Smooth side up	12.7	3.2	184.0	19.4	4.5	153.0
Thickness cmx10 ³ Dry	34.3 ± 3.8	38.9 ± 2.3	35.8 ± 2.0	30.7 ± 3.8	34.3 ± 2.8	34.5 ± 2.8
After 20 hrs in 40% KOH	38.6 ± 3.3	42.4 ± 1.8	40.9 ± 2.5	34.3 ± 2.5	34.3 ± 2.5	37.8 ± 3.8
A.C. Resistance μ-cm ²	0.082	0.082	0.23	0.066	0.076	0.566
μ-cm	2.12	1.94	5.53	1.92	1.99	14.75
Air Permeability - sec. to pass 300cc Air	2.65	2.52	2.52	2.24	2.16	2.20

¹ DNW = did not wet

Table 5

Kendall Webril

Properties of Absorber Materials in 40% KOH	EM 476	E1451	M1450	M1410	M1401	M1488	Score RD651	Fellon 2505 ML	Fellon 301
Wicking Height - cm with roll	8.1	8.2	8.2	8.2	4.2	8.2	2.1	2.9	0.3
Across Roll	8.4	8.2	8.2	8.2	3.5	2.8	2.8	3.1	0.0
Wicking Rate min/cm with roll	2.77	1.99	7.60	3.7	42.9	20.8	42.9	2000	1000
Across Roll	5.63	4.14	5.29	11.2	51.5	75.0	87.0	2000	DNW ¹
Wet-out Time min. rough side up	3.3	2.72	3.3	2.14	1.06	37.9	61.1	40.1	15.3
smooth side up	1.2	3.17	0.63	0.34	2.72	11.8	28.9	42.8	62.6
Thickness cm x 10 ³									
Dry	11.2 ± 0.3	14.7 ± 1.5	13.7 ± 1.3	9.9 ± 1.5	6.6 ± 1.0	9.1 ± 0.8	17.0 ± 1.3	33.8 ± 3.6	13.7 ± 1.3
After 20 hrs in 40% KOH	11.4 ± 1.3	16.0 ± 0.5	12.4 ± 1.8	8.9 ± 0.5	5.1 ± 0.3	8.9 ± 1.3	13.5 ± 2.5	38.1 ± 2.5	15.2 ± 1.3
A.C. Resistance μ-cm ²	0.146	0.213	0.244	0.101	0.140	0.129	0.081	0.081	0.031
μ-cm	12.81	13.33	19.44	11.40	27.20	14.55	6.01	2.12	2.01
Air Permeability - sec. to pass 300cc Air	26.26	52.17	59.90	8.86	24.71	14.89	3354.0	1.99	0.1

¹ DNW = did not wet

2.2.2 Thickness Measurements

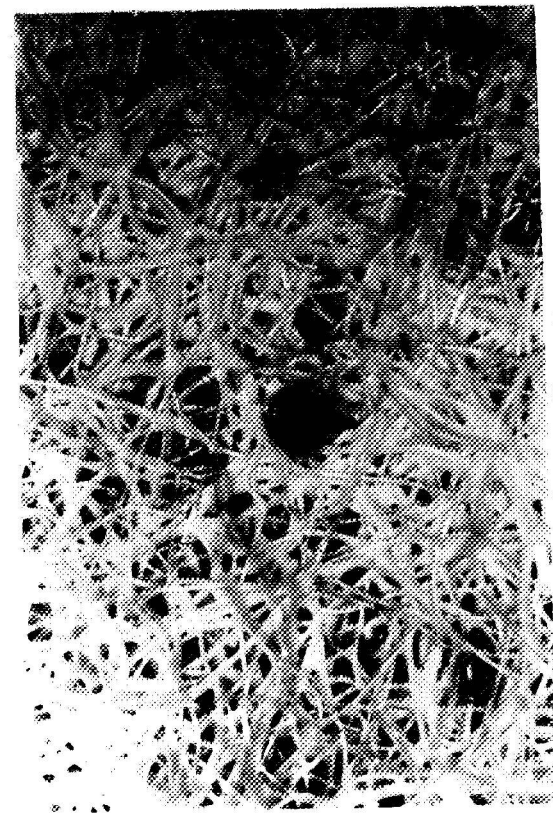
Absorber thickness is reported in $\text{cm} \times 10^3 \pm$ the maximum observed deviation from the average thickness. Measurements are made using a Federal Gauge with 1/2" diameter foot under 103 gms/cm². The calculation of percent swell is of doubtful validity since the swollen dimension is almost always within the maximum deviation from average dry thickness. Thickness measurements are taken from the 1" x 5" tensile strength samples so that values listed in Table 5 represent the averages of at least five different pieces of material.

2.2.3 Properties of Absorber Materials

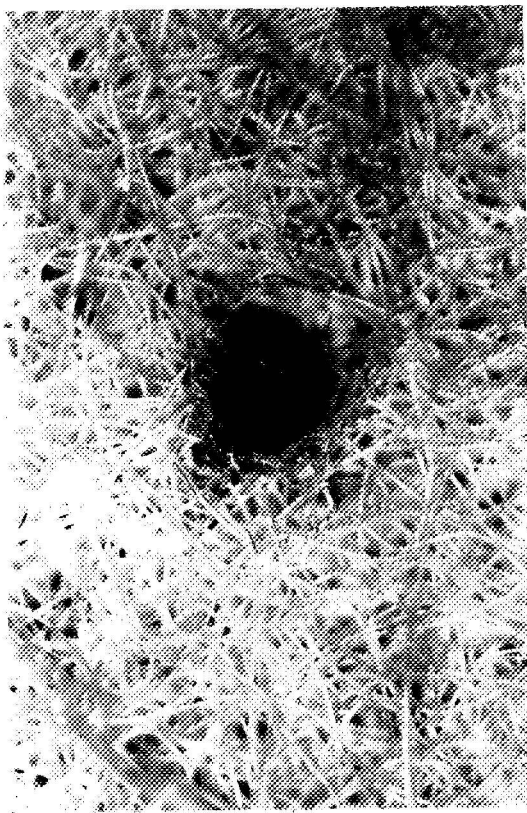
Absorber materials examined during this quarter exhibit a complete range of properties. Table 5 lists the properties measured to date.

The samples of PELLON 2505 were supplied by NASA. The material has been treated by RAI Research (Queens, New York). The scouring process consists of a 15-minute wash at 71°C in a solution of distilled water with 1% DUPONOL RA and 1% NH₄OH. DUPANOL RA is a fortified sodium ether-alcohol sulfate textile detergent of the anionic type. After scouring, the samples are water washed and dried at 60°C. The treatment process is not revealed but is directed at making the sample wettable. Control samples of the original material were not supplied.

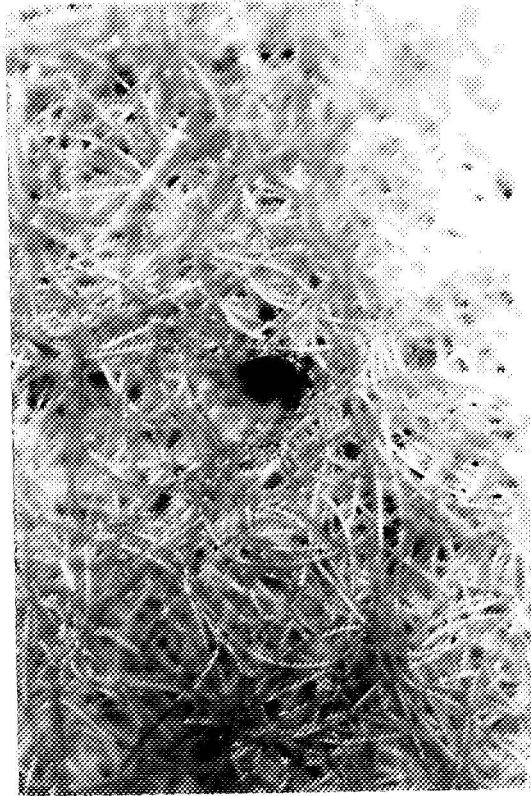
Of the six, only the two samples treated without scouring have wicking rates which are considered marginally satisfactory. The wet-out times of the four treated samples are acceptable. Unfortunately, the treated samples cannot be tested in "sealed" Ni-Cd cells. Figure 4 shows photomicrographs of pin holes which completely cover the samples. In general, there are fewer and smaller pinholes in the unscoured material. Since the untreated material has no similar pinholes, there is no doubt that these are micro-size burns resulting from the treatment process.



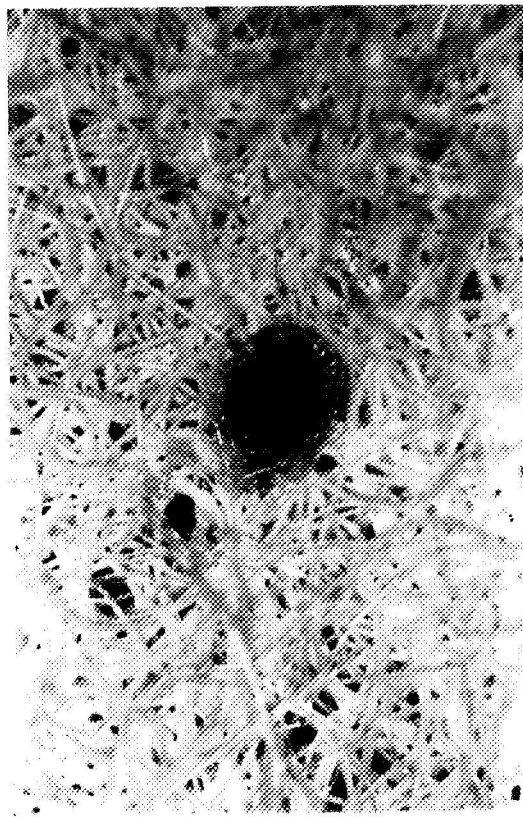
U-TREATED



U-SCOURED AND TREATED



E-TREATED



E-SCOURED AND TREATED

FIGURE 4. RAI TREATED - PELLON 2505 WO. 48X

Fusion of the fibers at the perimeter of the holes is evident. In addition, careful examination of the photographs shows that the fibers are still clear. This indicates that little surface etching has occurred. The frequency of the pinholes is partially reflected in the A.C. resistivity of these materials. Individual pinholes are approximately 300 microns in diameter.

The rates of wicking and wet-out times of the six materials from Kendall Mills are generally good. The values for air permeability and A.C. resistivity indicate a high fiber density. However, fiber density is the most variable physical property of absorber materials. If we disregard the pinholes of Figure 4, it is not unusual to find areas as tightly packed as E-Treated and as loosely spaced as U-Treated within two square centimeters of the same piece of material. Separator testing in Ni-Cd cells will be started with the Kendall materials.

2.3 Cell Testing

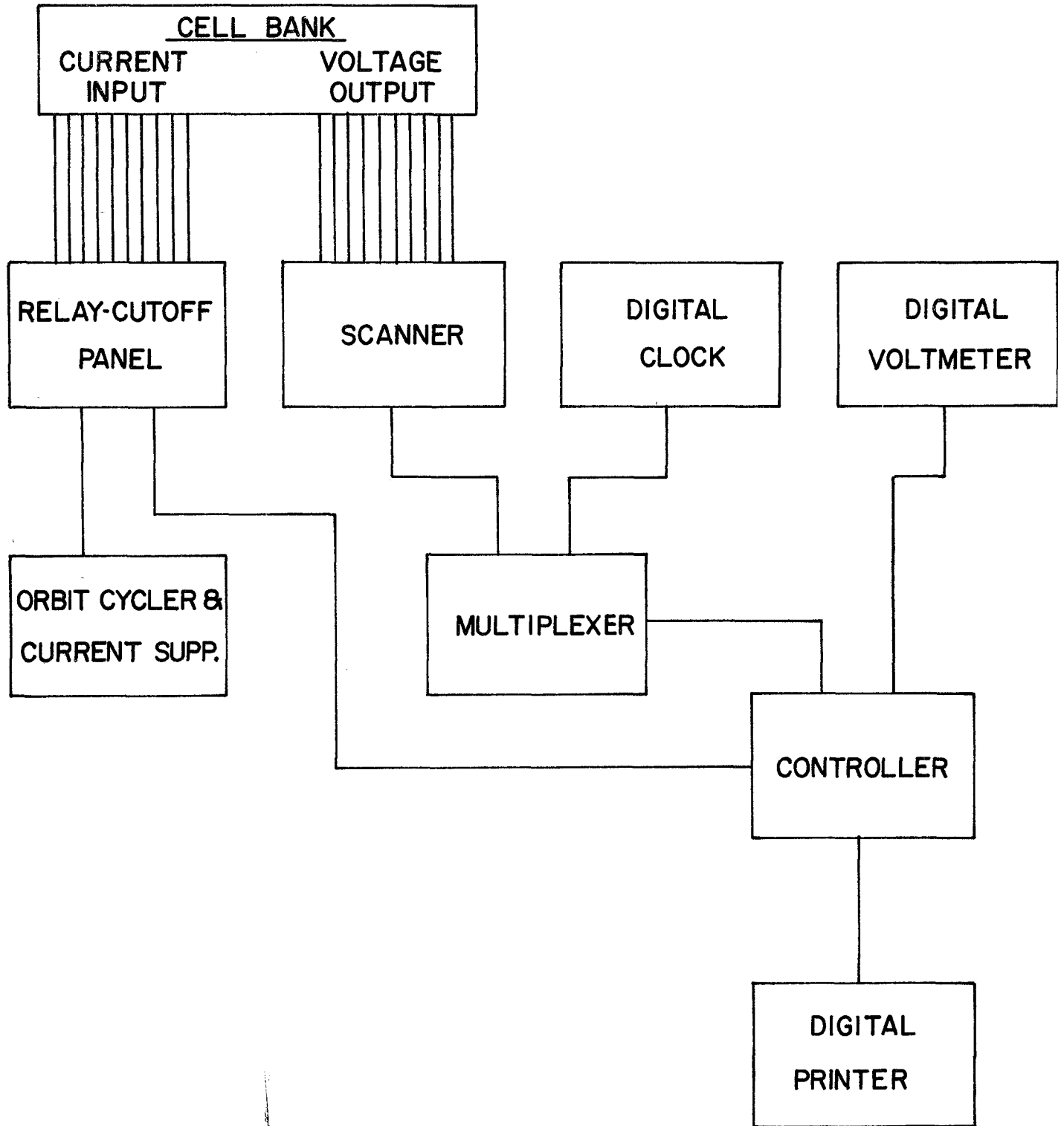
2.3.1 Sealed, Nickel-Cadmium Test Cells

In addition to silver-cadmium oxide and nickel-zinc oxide cell testing, provisions have been made for testing absorber materials in sealed, nickel-cadmium cells. Nylon cell cases with nickel plated hardware have been obtained. A three-plate 0.5 A.H. cell was assembled from 4.0 A.H. cells supplied by NASA. The three plates and six polysulfone shims of the same thickness as the plates, were wrapped with an S-fold of absorber material. Additional polysulfone shims were used to fit the nylon case. The six "dummy" electrodes were wrapped to aid in saturating the space above the plates with electrolyte vapors. The S-fold of material is cut one-half inch longer than the cell plates. The lower one-quarter inch of material was immersed in an electrolyte reservoir in the bottom of the cell case. The absorber material was, therefore, required to wet-out and wick sufficient electrolyte to supply the cell. Provisions were made for the inclusion of a nickel reference electrode.

A ten-position digital recorder has been modified to impose upper and lower limits to cell voltage. The cells will be subjected to a 90-minute cycle at 25% depth of discharge and 27% overcharge. The two hour discharge rate and three hour charge rate are to be employed. At the present time, 1000 cycles will be considered satisfactory performance. A block diagram of the cycling apparatus is given in Figure 5.

FIGURE 5

TEN POSITION -ORBIT CYCLING APPARATUS



3.0 FUTURE WORK

The major part of the next quarter will be taken up with assembling and cycling three-plate test cells. As in the past, membrane samples will be tested in Ni-ZnO and Hg-CdO cells. Performance is considered satisfactory, if duplicate cells complete thirty 100% depth of discharge cycles with 20% overcharge each cycle. Performance on the thirtieth cycle must be at least 50% of initial capacity. Normal plateau voltages are 1.60-1.68 volts for Ni-ZnO and 1.02-1.09 volt for Ag-CdO.

Absorber materials will be tested in the Ni-Cd cells described in 2.3.1. Satisfactory performance has been set at 1000 cycles. Further qualifications to end of discharge, plateau, and end of charge voltages will be made when characteristic cell performance can be defined.

The effects of surfactants on wet-out and wicking properties will be studied to determine the difference between cationic, anionic and non-ionic tenside. In addition, analysis of residual processing aids, both organic and inorganic, will be performed with the goal of identifying these materials. Finally, the effect on cell performance of breakdown products of polyamide and polypropylene materials in an O_2 -KOH environment will be studied. Plans have been made to introduce these products into cell electrolyte as well as to determine the effect of degradation on the structure of the material.

Present practice by absorber material manufactureres is to adjust wetting properties to user requirements with surfactants. Surface modification of the individual fibers should yield the enhanced wetting properties without the introduction of soluble and possibly impurities. Experiments into more than one surface treatment will be made with measurement of resultant changes in wet out and wicking rates.

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