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AN EVALUATION OF POTENTIALLY USEFUL SEPARATOR MATERIALS FOR NICKEL-CADMIUM (NI-Cd) SATELLITE BATTERIES

H. A. Baker, et al

National Bureau of Standards

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Consumer Product Systems Section Measurement Engineering Division Institute for Applied Technology

October 1974

Final Report

Prepared for National Aeronautics and Space Administration Goddard Space Flight Center Greenbelt, Maryland 20770



U. S. DEPARTMENT OF COMMERCE, Frederict & Dent, Secretary

NATIONAL BUREAU OF STANDARDS, Richard W. Roberts, Director

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This study was undertaken for the Goddard Space Flight Center, National Aeronautics and Space Administration, to develop and improve testing procedures and testing techniques for direct electro-physicochemical evaluation of polymeric separator materials used in 6 ampere nickel-cadmium (Ni-Cd) satellite batteries.

The intent of this study was to determine which tests, including those in a proposed test method, were pertinent to the evaluation of separator materials, and to use these procedures to evaluate various types of new materials being used in experimental batteries. In addition the procedures were designed to be used to evaluate other potentially useful materials, without the need for fabrication and extensive testing of actual batteries.

It was intended that these procedures would, if necessary, provide a basis for evaluating structural changes in the materials that might result in premature failure of batteries subjected to cycling tests; and to determine the relationships between changes in properties to the mode of failure and to service life; is order to establish criteria for the selection of materials with optimum service characteristics.

After appropriate test procedures had been established using ten different separator materials, they were used to evaluate the properties of these same ten materials taken from experimental batteries after they had been subjected to simulated use conditions. Subsequently, ten additional materials were received for laboratory evaluation of their potential use as separators, based on the previously obtained data. The results obtained on all twenty of these samples were previously contained in the NBS Technical Report No. 10956, "Evaluation of Separator Materials Used in Ni-Cd Satellite Batteries," November 1972.

This report is concerned with and contains the results of the laboratory evaluation, using previously established testing procedures, of twenty-five separator materials, which had not previously been studied. Consequently, these current tests word intended to determine their potential suitability and probable efficacy if used in Ni-Cd batteries.

The methods currently used measure materials properties such as the apparent density, absorptivity of the electrolyte, electrolyte recention, porosity, electrical resistivity, wettability, wicking time, wet-out time, and air permeability.

Materials

The separator materials were in the form of sheets of non-woven fibers. The fibers used in these materials were: nylon-6 (polycaprolactam), polypropylene, grafted polypropylene, a polyethylene/polypropylene blend, and aluminum oxide. One sample consisted of a filled plastic film laminated to a non-woven asbestos substrate. The samples included in this report were submitted to this 'aboratory at various times between September 1972 and November 1973, and are listed in table 1. All of the new materials were tested in the "as-received" condition, that is in the same form in which the materials would be used to fabricate batteries. Several of the materials had been subjected to surface treatments involving the application of wetting agents. Three of the polypropylene materials had been subjected to grafting techniques in attempts to chemically attach hydrophilic components to the polypropylene fiber surfaces, in order to increase the wettability of the fibers.

Since evaluation of the preperties of materials actually used in batteries, and subsequently subjected to these test procedures, cannot be accomplished without aqueous extraction of the electrolyte, it was necessary to subject the new materials to an aqueous extraction, using a Soxhlet apparatus, in order to determine whether this process affected the properties of the materials to the extent that a substantial change due to the extraction would prevent evaluation, by these procedures, of materials taken from cycled experimental batteries.

The first group of samples submitted for evaluation in this current series were samples AA through SS. Within this group there were five pairs of samples in which the material used in each pair was identical with respect to fiber content and weight per unit area, except that one of the samples had been treated with a wetting agent. In each case there was evidence of severe bleeding of the wetting agent from the sample into the packaging materials in which they were received. Consequently, the sponsor reque. I that these, and any other samples received at a later date, of treated materials, as well as other selected samples, be subjected to two twenty-four-hour aqueous extractions prior to conducting any of the tests. This involved a variant in the normal procedure of extracting the samples for twenty-four hours. In conducting the dual extraction, the samples were allowed to air dry at room temperature between the two extraction cycles. In addition, a number of selected materials were also subjected to extraction with specified organic solvents prior to measurement of the absorption characteristics.

Procedures for Apparent Density, Absorption, Retention and Porosity 1/

These procedures involve the determination of the apparent density, based on the dimensions and weight of specimens of the separator material, the amount of electrolyte absorbed, the amount of electrolyte retained after centrifuging, and the porosity, calculated on the basis of the volume of electrolyte absorbed.

Normally these tests are conducted on ten specimens. However, on the basis of experience achieved in this laboratory on various experimental materials, it was found that some were not wetted, or incompletely wetted by the electrolyte under the conditions of tests described below. Consequently, when a new material was being subjected to a preliminary analysis, only five specimens were used. If all of these specimens wetted so that they were completely saturated, then five additional specimens were tested, and the average of all ten specimens was used to

- 2 -

characterize the material. In those cases in which the specimens were incompletely wetted, it was normally possible to obtain some data on those properties related to the absorption characteristics described in this section. However, it usually was not possible to obtain data on any of the other properties, except air permeability. In some cases, extraction resulted in the removal of all or part of the wetting agent so that specimens from these materials did not wet completely when soaked in the electrolyte. In such cases, the number of test specimens from the extracted material was also limited to five.

For these tests, the specimens are cut from the sheet materials by means of a sceel die having a diameter of 2.54 centimetres (cm) (1 in). The weight of each specimen is determined to the nearest 0.1 milligram (mg) on an analytical balance. The thickness is obtained using a dial micrometer gauge, graduated to read directly to 0.001 inch, and with a finger lever and a 1.27 cm (0.5 in) diameter stainless steel spindle. The anvil, or platform, on which the specimen is placed must be of sufficient size to accommodate the specimen. The gauge should exert a spring pressure approximately equivalent to that of a 50 gram (g) weight. To minimize the effects of compression on the separator materials by the testing device, no additional weights are used on the gauge. The nominal thickness is reported as the average of two readings, one taken within two seconds after the spindle is placed on the specimen, and the other after it has been in contact with the specimen for 3 minutes. The specimens are then placed in a plastic container with 100 milliliters (ml) of an aqueous, 31% solution of potassium hydroxide (KOH) and soaked for at least 24 hours. Plastic beakers sealed with plastic film or plastic bottles with screw lids are used. (Note: use of glass containers should be avoided since reactions between the alkali and glass may effectively dilute the electrolyte solution.) On completion of the soaking period, the specimens are removed one at a time, and both sides blotted momentarily on a fine grain ashless quantitative filter paper to remove excess KOH. Blotting is accomplished by holding the specimen with stainless steel tweezers and lightly touching each side to the filter paper. The specimen is then placed in a previously tared plastic weighing bottle, capped immediately, and the weight of KOH absorbed is determined. The specimens are placed between two stainless steel wire mesh discs in a vertical position in a centrifuge, backed by a 0.3 cm thick stainless steel ring and a piece of blotting paper, and then subjected to a centrifugal force of 25 G's for two minutes. The specimens are then immediately placed in previously tared, plastic weighing tottles, capped and weighed. The amount of absorbed KOH retained is determined. The percent porosity is determined based on the wet weight obtained just prior to centrifuging.

Calculations:

Apparent density, $g/cm^3 = \frac{dry \ weight}{dry \ volume}$

KOll absorbed, % = wet weight - dry weight x 100

KOH absorbed, $g/cm^3 = \frac{wet \ weight - \ dry \ weight}{dry \ volume}$ KOH retention, $s = \frac{weight \ after \ centrifuging - \ dry \ weight}{dry \ weight} \times 100$ s of absorbed KOH retained, $s = \frac{s \ of \ KOH \ retained}{s \ of \ KOH \ absorbed} \times 100$ $\frac{wet \ weight - \ dry \ weight}{dry \ volume} \times 100$

Note: 1.3 is average density, in gm/cm³, of an aqueous solution containing 31% KOH at ambient laboratory conditions.

When the specimens are removed from the electrolyte solution, after having soaked for at least twenty-four hours, they are visually examined and a subjective estimate is made of the approximate percentage of the surface area that is thoroughly saturated. The dry materials are white and opaque in appearance, while wetting to saturation causes their appearance to become translucent. It is the percentage of translucent area that is reported in table II, for specimens of both unextracted and extracted samples. When the specimens did not become completely saturated, the wetted areas were generally spotty and irregular, and in some cases may have been related to localized variations in the fiber density within the individual specimens.

The data on the absorption characteristics are given in table III for the "as-received" materials and in table IV for the extracted materials. It should be noted that even when the average area of the wetted portions of the specimens is rather low, the amount of KOH absorbed may be relatively high when compared to other materials.

Procedure for Electrical Resistance 2/

This procedure is used to determine the electrical resistivity of the separator materials when saturated with an electrolyte. The resistance measurements are made by connecting an electrolyte-filled test cell, in which the specimen is located between the anode and cathode of the cell, to a universal bridge operated in the internal mode (1592 Hz). The test cell is designed so that the circular area of the specimen undergoing test was 0.686 cm². Five 2.54-cm square (1 in²), or 2.54 cm diameter (1 in) specimens are used. The specimens are placed in 31% KOH solution for at least 24 hours prior to testing. On completion of the soaking period, the test cell is assembled without a specimen, filled with 31% kOll solution and the resistance of the electrolyte measured to obtain a blank reading. A similar measurement is made on completion of each set of specimens, and the average of the two measurements is used in calculating the resistance of the solution. A wet specimen is then placed in the empty cell in such a manner as to permit measurements to be performed at two different locations on each sample, for example, one

in the upper right quadrant and one in the lower left quadrant. Both sides of the cell are filled with electrolyte and the resistance measured. The average of these two measurements is used to determine the combined cell and separator resistance. Immediately after completing the resistance measurement, the thickness of the wet specimen is measured using the gauge and technique previously described in the absorption test.

The electrical resistance characteristics are reported in table VI as the specific resistivity.

Calculations:

R = cell resistance (blank), ohms

R = cell resistance-with separator, ohms

R' = separator resistance, ohms

 $R' = (R_{e} - R_{o})$

Separator specific resistivity, p , ohm-cm

 $\rho = \frac{R'}{t_W} X A$ where: $t_W = wet$ separator thickness in cm A = area in cm²

Procedure for Wettability

The universal bridge and test cell used in the electrical resistance tests are also used to determine wettability of the separator material. Wettability is measured as the time required for the electrical resistivity of the separator material to equilibrate. Ideally the specific resistivity at equilibrium should equal that obtained on the pre-soaked samples, and be achieved in a matter of minutes.

Five specimens are cut using a 2.54 cm square or 2.54 cm diameter steel die. A dry specimen is placed in the empty test cell and the cell connected to the bridge. One side of the cell is rapidly filled with 31% KOH solution, and the timing of the experiment is begun as the second side is being filled. The electrical resistivity is monitored and the time for the resistivity of the system to remain constant for one minute is observed. The time at the start of the one-minute stable period is recorded as the wettability time. In those cases where a material does not wet, the sample is allowed to remain in the cell for two hours as a means of verifying its non-wettability.

The wettability test data are also presented in table VI.

Procedure for Wicking

This procedure is used to measure the rate of rise of electrolyte solution up a vertically supported, partially immersed material. In these tests, due to the wide variation in wicking characteristics of the materials the measurements were recorded either as "time to reach 6 cm," the ideal condition, or as "cm/24 hours" depending on which event occurred first.

In order to facilitate these tests a special holder, capable of holding 5 specimens, was designed and built to provide a means for uniformly positioning and immersing the specimens, and to measure the height of the solution on the specimens. This device is illustrated in Figure 1.

The original intent of this procedure was to record the time required for the solution to rise to a height of 6 cm. It is obvious from the data given in table VII that several materials would require prolonged test periods, if the solution could indeed travel to a height of 6 cm. It was for this reason that all such experiments were discontinued after 24 hours.

Since a test of this type is sensitive to environmental conditions such as changes in temperature, relative humidity and velocity of air currents impinging on the specimen surface, the test should be conducted for a specified short period of time to reduce the effects of these variables. For example, wicking action will stop when the rate of upward movement of the solution equals the rate of evaporation of water in the electrolyte from the specimen. It is believed that a short time test, e.g., two to four hours, would provide an adequate means of assessing variations in wicking characteristics of different materials. If it is considered necessary to retain the longer test period, then it is obvious that the tests should be conducted in a totally enclosed system.

Place five specimens, $0.64 \times 8.9 \text{ cm} (0.25 \times 3 1/2 \text{ in})$, in the holder. Place the holder in a tray of suitable size and fill the tray to a depth of 10 mm above the lower ends of the specimens with 31% KOH. As an aid to accurately distinguish the leading edge of the solution on the specimen, a dye was added to the KOH solution. Phthalein purple indicator, used in a ratio of 0.167 g per 100 ml, was found to produce a suitable, stable color in the solution.

The results of these tests are given in table VII.

Procedure for Wet-Out Time

This procedure is intended to determine the time required for a drop, approximately 0.1 ml, of electrolyte solution to be absorbed when placed on the upper surface of a specimen of separator material.

In the process of developing this test it was noted that, as a result of the porosity of some of the materials, the solution tended to seep through the specimen and spread by capillary action between the specimen and supporting surface. It was found that placing the specimens on three layers of nylon retting eliminated the capillarity problem. The metting was held flat by affixing it to a polyethylene covered board approximately 5 X 20 cm (2 X 8 in).

Five specimens 2.54 cm (1 in) in diameter are placed on the nylon netting, and one drop of a solution of 31% KOH, containing the same amount of phthalein purple indicator used in the wicking tests, is placed in the center of the specimen, the timer started, and the time required for the entire specimen to become wetted, or until absorption is complete, is recorded. Absorption is considered to be complete on disappearance of the sheen normally visible on the surface of the liquid. In those instances in which the entire specimen is not wetted on total absorption, or on incomplete absorption after 1 hour, the area of absorption is determined by means of a supported acrylic plastic disc having etched concentric circles, which is placed over, but not in contact with the specimen. The diameters of the circles are: 0.48, 1.11, 1.59, 2.07 and 2.54 cm (3/16, 7/16, 10/16, 13/16 and 1 in), and are assigned numbers 1 to 5, respectively. In reporting the wet-out characteristics of a material, the assigned circle number or range is used.

The results of these tests are given in table VIII.

Procedure for Ai' Permeability

This procedure consists of measuring the time required for 300 ml of air to permeate a .645 sq. cm (0.1 sq in) area of a specimen under uniform pressure equivalent to 3.1 cm (1.22 in) head of water. A Gurley Densometer, model 4110, with a 141 g (5 oz) inner cylinder is used for the test. Measurements are made on ten samples from each material. These measurements are made under standard laboratory conditions required for textile fabrics, which are 21°C (70°F) and 65% R.H.

The results of the tests are given in table IX.

Discussion

Importance of Separator Properties

It must be emphasized that the properties of the separator materials in the original, "as-received" condition are of primary importance with respect to their use in batteries. Any drastic or detrimenta, change in properties as a result of aqueous extraction simply means that a realistic evaluation of similarly extracted materials removed from experimental batteries may be difficult or impossible to achieve. Measurement of materials properties after extraction was necessary since all cycled materials must be extracted to remove the electrolyte prior to retests intended to detect changes in the materials that might be useful in predicting the service life of the batteries.

The properties that appear to be most important in assessing the suitability of a material for use as a separator material are as follows: high electrolyte absorptivity, good electrolyte retention, low specific resistivity, rapid wettability, and low resistance to air permeation. Wicking characteristics and wet-out time seem to be most important with respect to the initial filling of the battery with the electrolyte. In these tests, however, one fallacy seems to nave been detected that had been deemed to be most important for a separator material, and that is the need for a high degree of electrolyte absorption. In the past, materials such as cellophane, and nylon in the form of film, woven and non-woven fabric have been used, both alone and in combinations, as Ni-Cd battery separators. Both of these materials have very high absorption capacities for many aqueous solutions due to the respective presence of hydroxyl and amide groups in the polymers. However, both materials are subject to degradation by hydrolysis when emposed to strong alkaline solutions. The direct result of such degradation is the development of electrical shorts between plates with an eventual tailure of the battery.

In this study, most of the separator materials were made using polypropylene fibers, which are very resistant to alkali hydrolysis. However, since the polypropylene fibers are basically hydrophobic, they do not wet-out when exposed to aqueous solutions. Consequently, efforts have been made to treat these fibers with various types of wetting agents.

When considerations for retest of cycled materials are concerned, it is obviously necessary that any wetting agent be resistant to removal by hot, aqueous extraction techniques. Consequently, on the basis of work done to date, it appears that the overall criteria for polypropylene materials can only be met if successful techniques can be developed for the chemical grafting of hydrophilic, alkali-resistant, materials onto the polypropylene fibers. On the basis of studies performed on these types of materials, as well as previous studies comparing new and cycled materials it appears that a high degree of electrolyte absorption may be secondary to resistance to aqueous extraction and to chemical changes during actual use, provided that other properties such as low specific resistivity, low resistance to air permeation, and rapid wettability can be achieved and maintained under actual service conditions

Previous work on cycled materials had shown that the specific resistivity and the air permeability were the properties that were most indicative of changes in the separator materials. Therefore, when there is little change in these characteristics over a period of simulated use, it appears possible to predict the potential lifetime of a battery by monitoring these two properties.

Ubservations

The rationale for use of non-woven, fibrous battery separator materials is based in part on increased ion mobility as compared to that obtainable with selid film separators. Non-woven material is particularly suited to Ni-Cd systems since dendrite formation between battery plates is not a problem.

Nylon has been used for a number of years as a separator material. However, as mentioned above it is susceptible to hydrolysis. In these tests, the nylon materials have been used primarily as standards, against which some of the more alkali-resistant materials have been compared. Polypropylene is highly resistant to alkaline hydrolysis and has excellent electrical properties. However, it is non-polar and when immersed, absorbs water to the extent of <0.05 percent. Consequently, it exhibits very low wettability and electrolyte retention characteristics in aqueous systems unless the fiber surfaces are treated, for example by oxidation, grafting, or coating with a wetting agent.

As stated previously, these samples were received at various times, with samples AA through SS included in the first group.

Sample AA was a nylon material, described as non-wettable. In the "as-received" condition, this material exhibited characteristics similar to those of other non-wettable materials. However, after aqueous extraction it exhibited all of the desirable characteristics for a separator material, and was comparable in these properties to previously evaluated nylon materials.

Samples BB through EE were described as non-wettable polypropylenes, differing from each other only in the weight per unit area. The former three samples exhibited the usual properties of non-wettable materials. Sample EE gave anomalous results in that it had all of the characteristics of a useful separator material, and data were obtained on all properties except for wet-out time on the extracted material. With the latter exception, aqueous extraction had little effect on the properties as compared to the "as-received" material. This sample. unlike the other three of the set, appeared to consist of two thin, rather stiff, surface layers, between which was a very thick, loose, and non-woven mat as the center layer. The percent absorption of electrolyte was very high on the "as received" basis, and although there was a significant decrease in absorptivity after extraction, the percentage of absorption was comparable to that noted for the nylon materials. This material, when saturated with KOH was too thick to fit into the specimen receptical in the centrifuge head without it being squaezed by the wire mesh screens, which undoubtedly accounted for the unusually low percentage of electrolyte retention. It was also noted that when the specimens were placed in the KOH solution for the absorption test, and particularly in the conducting of the wettability, wicking and wet-out tests, that the electrolyte seemed to rapidly penetrate the outer surfaces, saturating the inner layer prior to wetting of the surface layers.

Sample FF was described only as a polypropylene material. Its properties, as determined in the various tests, indicated that it was a non-wettable material.

There were four pairs of polypropylene materials in which each pair was identical except that one sample in each pair had been treated with a wetting agent. These pairs included samples GG and Hil, II and JJ, KK and LL, and SS and RR in which the latter sample within each pair had been treated. Similarly, samples M4 and NN were identical, with the latter having been treated, but these materials had been produced from a blend of polyethylene and polypropylene fibers. Each of the wettable samples in the five pairs exhibited good separator properties when tested in the "as-received" condition. However, after two Soxhlet extractions with water, they generally exhibited the same characteristics of the comparable non-wettable sample. The only exception noted was that sample HH still retained enough wettable characteristics to allow the measurement of the specific resistivity. However, this value was considered to be too high to be useful in evaluating changes in the material if it were used in an actual battery.

Sample PP was identified as a wettable polypropylene material of Canadian origin. Although the amount of electrolyte absorbed by this material was rather low by comparison with other wettable materials, its overall properties were excellent with respect to use as a separator material. Aqueous extraction had no significant effect on its properties, and methanol extraction did not appear to change the properties to any great extent. Chloroform extraction did result in a significant decrease in the absorption characteristics.

Of the remaining samples, all were subjected to only one 24-hour aqueous extraction, and none were extracted with organic solvents. Sample TT was produced from polypropylene to which had been grafted an acrylic-based compound in order to improve wettability. Aqueous extraction appeared to have no appreciable effect on the electrical and absorption characteristics, except in the test for wet-out time. Its properties both before and after extraction were similar to those of sample PP, including a relatively low degree of KOH absorption.

Sample UU was identified only as a polypropylene material. However, results obtained during the KOH absorption tests indicated that the material was full wettable and remained so even after aqueous extraction. In general, it behaved in a manner very similar to that noted for samples PP and TT.

Samples VV, WW, and XX were described as wettable polypropylenes. However, in the initial KOH absorption tests the "as-received" sample of WW did not wet completely as did the other two samples. All three closely resembled other non-wettable materials in their absorption and electrical characteristics after aqueous extraction.

Sample YY was the only inorganic material received. It was a ceramic material produced from non-woven fibers of aluminum oxide bonded by means of a butadlene-styrene elastomeric material. The sponsor requested that prior to conducting the evaluation tests the binder be removed by firing the material in a muffle furnace for 10 minutes at 700°C, and that in this state it should be considered as being in the "as received" condition. The fired material was rather fragile and difficult to handle. Test specimens subjected to the initial KOH absorption test partially disintegrated in the electrolyte, negating any chance of obtaining valid data on most of the subsequent test procedures. Consequently, no further tests were conducted on the material.

Sample 22 was a composite consisting of a zirconium oxide-filled poly(phenylene oxide) film laminated to a non-woven asbestos fiber substrate. Electrolyte absorption was very low and appeared to be confined essentially to the asbestos layer. The plastic film appeared to be slightly porous on the basis of a measurable electrical resistivity, although it was essentially impervious to the permeation of air. Data obtained on the wicking and wet-out tests appeared to be due principally to the presence of the asbestos layer

Sample J2 was a nylon material reputedly identical to a material previously evaluated and reported on, identified as sample J. Sample J2 had been taken from a different lot of material. This sample exhibited all of the characteristics expected of a pylon separator. However, sample J2 was approximately 35 percent thicker than sample J although both exhibited about the same weight per unit area. Consequently, a direct comparison between the two materials with respect to the apparent density, amount of KOH absorbed in g/cm³, and the percent porosity, indicates that sample J2 was somewhat inferior to its counterpart. However, all three of these properties are inversely proportional to the thickness so that comparisons made on this basis indicated that the materials were essentially identical. All other properties of the two samples were essentially comparable and any minor variations could easily be attributable to both variations in thickness and to fiber density.

After the necessary specimens had been obtained from sample KK, the remaining material was returned to the sponsor and subsequently submitted to a commercial laboratory for the purpose of grafting a hydrophilic material onto the polypropylene. The sample was saturated in a 5 percent aqueous solution of methacrylic acid and then irradiated for 41 hours with a Cobalt-60 source in an attempt to graft segments of poly (methacrylic acid) onto the fibers. The sample, when resubmitted to this laboratory for further testing, was designated as sample KK2. In the "as received" condition it exhibited excellent properties for use as a separator. However, after aqueous extraction it exhibited the same basic properties of the ungrafted, non-wettable sample KK. During the extraction, the water in the reservoir flask contained copious quantities of a white, flocculant precipitate. On completion of the extraction, the water was evaporated to dryness and in addition to the precipitate a clear film was also noted on the walls of the flask. The clear film apparently consisted of low molecular weight poly (methacrylic acid) which is water-soluble, while the precipitate apparently consisted of a higher molecular weight fraction of the polymer which is water-insoluble. It seemed apparent that the radiation had resulted in the polymerization of the sothacrylic acid, but did not provide an adequate number of free radical sites on the polypropylene fibers for sufficient grafting to occur to render the material permanently wottable. Some of the unextracted material was placed in a filute aqueous solution of KOH. The pH was monitored and found to decrease rapidly, indicating that the alkali was reacting with the free carboxylic acid groups in the polymer. This experiment indicated that if the material were used in a battery, it would effectively dilute the concentration of the electrolyte. Dilution might have some effect on the electrical characteristics of the electrolyte with respect to the functional capabilities of the battery. Assuming that suitable techniques of grafting poly (methacrylic acid) and the related poly(acrylic acid) is later achieved, the above results indicate that the grafted material should be washed in a dilute solution of KOII prior to its use in a battery.

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Table

Type a/ d/d d/d 2 GP 0 0. 2 -4 NASA Designation WEX - 1242, grafted with acrylic, Permion 3073 - 23, non-wettable, 1.46 oz/sq yd 3073 - 37, non-wettable, 1.77 oz/sq yd 3074 - 24, non-wettable, 2.05 oz/sq yd 3074 - 18, non-wettable. 0.72 oz/sq yd 2711 - 55, non-wettable, 1.5 oz/sq yd 2711 - 54, non-wettable, 1 oz/sq yd 2711 - 56, non-wettable, 2 oz/sq yd 2711 - 85, non-wettable, 4 oz/sq yd 3073 - 35, non-wettable, 1 oz/sq yd 3073 - 23, wettable, 1.46 oz/sq yd 3074 - 24, wettable, 2.05 oz/sq yd 3074-18, wettable, 0.72 oz/sq yd 3073 - 35, wettable, i oz/sq yd WEX-1242, wettable, Canadian 3073 - 22, non-wettable 73020 (#123), wettable 3073--22, wettable T-21124, Pelion 3073 - 22 NBS Sample Code 183 23 Y 0a 99 ÷ Z Ξ 3.7 N.K. 1 2 e. SS = 2 E RA 27

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Table I. Continued

Code	NASA Designation	Type a/
×	1972 - 31 (#123), wettable	4
XX	3073 - 32 (#123), wettable	4
YY	Alumina, non-woven ceramic, synthetic rubber binder	
1	11D2172, zirconium oxide in poly(phenylene cxide) film laminated to an asbestos substrate	
2	Pellon - 2505 ML, (lot 8, roll 3)	N
KK2	Sample KK, after grafting in 5% methacrylic acid solution	GP

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N - nylon
P - polypropylene
P/P - polycthylene/polypropylene blend
GP - grafted polypropylene

	As Re	eceived	Ext	racted
NBS Sample Code	Hours in KOH	Saturated Area, %	Hours in KOH	Saturated Area, %
AA	91	50	24	100
BB	91	60	24	20
сс	91	50	24	10
DD	91	25	24	0 <u>a</u> /
EE	24	100	24	• • 95
FF	94	• 75	24	20
GG	94	60	24	20
HH	24	100	24	40
11	67	50	24	10
JJ	24	100	24	25
JJ			162	80
КК	67	40	24	10
LL	24	100	24	25
LL			162	55
MM	67	45	24	50
NN	24	100	- 24	\$8
NN		-	162	100
PP	24	100	24	100
RR	24	100	24	60
SS	24	10	24	15
SS			162	70
TT	24	100	24	100
UU	24	100	24	100
w	24	100	24	25
WW	24	50	24	25
			1	

Table II. Degree of Electrolyte Saturation of Battery Separator Materials

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	As R	eceived	Extra	acted
NBS Sample Code	Hours in KOH	Saturated Arca, %	Hours in KOH	Saturated Area, %
XX	24	100	24	20
ZZ	24		24 .	-
J2	24	100	24	100
KK2	24	100	24	5

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a/ Some solution held on surface, sample still opaque

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Retention	ved
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Table III. Thickness, Apparent Density. Absorption, Retention	of Rattory
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Porosity	-	27.6	36.5	21.6	15.8	27.6	. 7.8	6.9	1.11	4.5	2.8	4.5	1.2	86.3	95.0	75.4	29.0	47.4	13.7	27.2	47.8	14.9
KOH Retained after centrifuge		36.8	93.3	. 0.96	94.7	98.1	91.3	95.2	99.5	6.16	\$6.2	99.4	92.3	23.2	30.6	12.4	. 97.0	2.99	94.0	96.8	98.4	94.7
_	SD	51.6			94.6			44.7			14.4			60.1			155.5			116.5		
KOH Absorption	0 ¹⁰	215.0	301.1	169.4	184.6	339.3	82.0	95.4	171.7	63.2	31.9	48.6	13.6	1028.0	1120.0	925.1 .	280.0	495.5	134.8 .	. 48.2	478.7	135.9
KOII	g/cm3 c/	.359	.475	.281	.205	. 359	.102	050.	.144	.058	.037	.059	.016	1.122	1.123	0.980	.376	.616	.179	354	623	.194
ent ity	SU <u>b</u> /	10.1			.010			.010			.017			.003			.008			900		
Apparent Density	8/cm3	.167	.178	.158	.113	.124	.100	.095	.109	.084	.118	.140	.093	109	115	.105	135	146	.124	147	341	.132
Thickness	inch	1133	0140	.0127	. 0123	.0132	.0118	0610	.0230	.0154	.0259	.0280	.0237	0565	0640	.0487	1000	2000	.0036	2000	1110	0600.
Avg. a/		(5)	12	:	(5)	£ =	: -1	(5)	E	: :	(2)	-		(01)	in H	:	(6)	(c) =	:	101	(c) :	
NBS Sample Code			2		BB	2		J	3.		a	1		83	1		Ľ	-		5	3	

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Table III. Continued

Porosit, 25.3 16.4
24.5
10.2 85.6 93.3 78.4 36.354.8 71.2 82.1 92.0 74.3 81.4 89.7 76.3 after centrifuge **KOII** Retained 97.7 98.8 94.4 97.8 99.5 96.0 95.1 96.8 95.9 96.7 97.2 98.5 95.8 98.9 95.2 96.7 93.7 95.1 • 17.9 21.5 94.6 51.0 63.1 98.4 22.1 SD KOII Absorption 688.6 723.4 237.2 320.3 103.2 145.6 224.2 88.7 283.2 439.9 191.0 650.8 740.3 823.9 626.2 492.1 519.9 460.1 654.4 618.1 g/cm3 c/ 1.196 1.166 .319 .112 1.212 1.019 .713 .926 1.067 .328 .451 .144 1.058 .133 la ns .005 .006 .004 .007 500. .002 .003 Apparent Density g/cm3 .155 .139 .148 .142 .151 .162 .199 .141 .141 .136 .155 .167 Thickness inch 1600 .0169 .0144 .0194 .0179 1710. 0066 .0068 .0078 0157 0183 .0064 .0061 1700. Avg. a/ (10) (16) (10) (01) Г (S) E (2) (2) = -= .. -NUSS Sample Code 2 III 3 KK LL. NN

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Table III. Continued

NBS Sample Code	Avg. a/	Thickness	Apparent . Density	sity	KOH	KOH Absorption		after cent.ifu	ketained cent.ifuge	Porosit)
		inch	g/cm3	SU b/	g/cm3 c/		SD	*		*
4	1013	0002	289	600	.584	202.3	27.5	87.6		44.
	(or)	0106			.685	241.8 .		97.0		52.
	:	.0084	.272		.445	151.7		66.0		34.2
2	1013	000	14.7	010	978	602.9	23.8	98.3		75.2
XX .	(or)	2000.	176		160.1	637.4	-	59.2		84.0
	:	.0080	.144		.857	567.6		97.8		66.0
55	(2)	.0087	.133	.004	.181	136.0	27.8	86.9		14.0
3	-	1010	.138	•	.221	164.7		90.2		17.0
	:	.0081	.129		.128	39.4		83.3		9.8
ŧ	(01)	125	.221	.019	.487	222.7	29.1	. 98.4		37.5
		0132	244		.521	291.3		9.66		.0.
	:	.0116	.178		.429	190.2		97.4	1	33.0
	1017	0300	100	010	.536	173.2	13.1	. 98.8		41.2
8	(01)	¥500	328		.626	192.7		8.00	-	48.1
	: _>	.0082	.294		.473	155.2		97.8		36.
~~~	(01)	1210	.152	900.	. 606 .	397.9	14.6	7.70		46.6
	1000	0136	.162		.649	423.8		98.9	-	50.0
•	:	.0109	.136		.552	374.6		95.7		42
WM	(10)	.0194	.141	.004	.696	492:9	85.7	2.00		53.6
	-	.0224	.146		.8.13	604.6		9.60		64.8
	1	.0165	.133		.474	339.3		98.2		36.

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NBN Sample Lode	Avg. a/	Thickness	Appa	Apparent Density	KOII	KOll Absorption	u	KOII Retained after centrifuge	Porosity
		inch	g/cm3	No by	g/cm3 c/		SD		-
XX	(10)	.0151	.139	.004	.952	683.3	23.9	98.8	73.2
	=	.0161	.150		1.043	734.2		0.00	80.2
	1	. 0140 .	.133		.894	646.9		98.2	68.8
22	(10)	.0157	1.446	.043	126.	67.3	7.2	98.1	74.7
	=	.0162	1.528		1.123	79.3		98.9	86.4
	-1	.0149	1.393		. 844 .	57.2		97.4	64.9
J2	(20)	.0166	.138	.004	.786	569.6	32.7	98.4	60.4
	H	.0180	.150		.850	621.0		99.4	65.4
	-1	.0141	.132		.729	510.3		92.6	56.1
KK2	(10)	.0227	. 202	.014	.872	434.1	41.1	. 99.3	67.1
	=	.0239	.236		.934	488.5		99.8	6.17
	1	.0210	.187		.821	347.7		98.9	63.1

Number in parentheses represents the number of specimens used to compute the average values. H = highe value, L = lowest value. 10

Standard deviation 21 Grams of KOH absorbed per cm³ of separator material. 21

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		Table IV.		Thickness, A Retention and Materials Af Water	Thickness, Apparent Density, Absorption, Retention and Porosity of Battery Separa Materials After Extracting for 24 hours Water	ity, Absorl of Battery S ng fer 24 h	serption, ery Separator 24 hours in		· 
NBS Sample Code	Avg. a/	Thickness	Anpa	Anparent vensity	NON	KOII Absorption	3	KON Retained after centrifuge	Porosity
		inch	g/cm3	SI) b/	8/cm3 c/		SD	c.9	
IP VV	(10)	.0151	.159	.007	966.	628.1	25.2.	98.5	76.9
•	=	.0167	.166		1.075	666.7 .		99.4	82.7
	1	.0131	.144		924	591.4		98.4	71.1
38	(5)	.0134	.108	.008	.253	237.8	60.2	98.2	19.4
	=	.0143	.119		.315	. 307.8		8.00	24.3
	-	.0126	.100		.206	173.2		97.5	15.8
8	(2)	.0254	.095	.007	.155	163.4	38.7	98.2	11.9
	=	.0267	.105		.208	221.1		98.5	16.0
	1	.0243	.036		.119	126.2		98.0	9.2
00	(2)	.9302	112	110.	.061	55.9	19.3	. 58.1	4.7
	=	.0316	.126		.069	81.7		98.9	6.1
	-1	.0280	.093		.043	34.4		97.6	3.4
IE d/	(2)	.0447	.131	.014	.774	608.4	186.4	27.0	59.6
•	=	.0470	.145	•	.962	835.8		35.7	74.0
	7	.0431	.115		.657	451.6		17.9	50.5
Ŧ	(2)	.0105	.126	.002	.304	240.4	29.8	92.2	23.4
	=	.0119	.128		.353	279.5		93.8	27.1
	1	.0095	.123		246	196.4		88.9	19.0
99	(2)	.0105	.128	.005	. 297	231.3	30.8	94.2	22.9
	=	.0115	.134		.348	254.3		95.7	20.8
	1	2600.	.120		.240	189.8		93.3	18.5

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inch $g/cm^3$ SU $y/cm^3$ $y/cm^$	NUS Sample Code	Avg. a/	Thickness	Apparent Density	rent sity	KOH	KOH Abscrption	u	KOII Retained after centrifug	Retained centrifuge	Porosit
			inch	8/cm3	SU b/		0.5	SD	-		
	/P III	(2)	.0092	.337	.004	.612	447.4	112.1	96.7		47.1
	1	=	.0098	.143		.786	571.9		98.0		60.5
		1	.0078	.134		.379	264.0		95.3		29.2
$ \begin{bmatrix} 1. & .0163 & .156 & .342 & 2311 & .96.7 \\ L & .0138 & .132 & .168 & .115.2 & .95.7 \\ (5) & .0144 & .140 & .02 & .517 & 368.6 & 68.5 & 95.7 \\ L & .0129 & .1138 & .434 & 315.7 & .95.9 \\ 1 & .0140 & .147 & .002 & .933 & 647.2 & 34.4 & .99.0 \\ 1 & .0140 & .147 & .022 & .133 & 547.2 & 34.4 & .99.0 \\ 1 & .0103 & .147 & .022 & .136 & .93.1 & 9.7 & .99.4 \\ 1 & .0175 & .1138 & .1146 & 1022 8 & .93.1 & 9.7 & .99.7 \\ 1 & .0175 & .1138 & .1146 & 1022 8 & .93.1 & 9.7 & .99.7 \\ 1 & .0175 & .1138 & .1146 & 1022 8 & .93.1 & 9.7 & .99.0 \\ 1 & .0175 & .1138 & .116 & .002 & .116 & 82.3 & .94.6 \\ 1 & .0175 & .133 & .106 & .377 & 264.2 & 88.3 & .97.2 \\ 1 & .0192 & .113 & .006 & .377 & 264.2 & 88.3 & .97.2 \\ 1 & .0208 & .147 & .006 & .377 & 264.2 & 88.3 & .97.2 \\ 1 & .0208 & .147 & .006 & .377 & 264.2 & 88.3 & .97.2 \\ 1 & .0208 & .147 & .006 & .377 & 264.2 & 88.3 & .97.2 \\ 1 & .0208 & .147 & .006 & .377 & 264.2 & 88.3 & .97.2 \\ 1 & .0200 & .142 & .006 & .377 & 264.2 & 88.3 & .97.2 \\ 1 & .0200 & .147 & .002 & .567 & .584 & .97.1 & .96.7 \\ 1 & .0200 & .147 & .533 & .38.4 & .97.1 & .97.8 \\ 1 & .0200 & .147 & .553 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0200 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0000 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0000 & .147 & .563 & .388.4 & .97.8 & .97.8 \\ 1 & .0000 & .147 & .444 & .444 & .444 & .4444 & .4444 & .4444 & .4444 & .4444 & .44444 & .44444 & .4444 & .4444 & .4444 & .44444 & .44444 & .44$	11	(2)	.0151	.144	600.	.253	173.8	49.7	95.2		19.4
L 0138 132 168 115.2 93.7 (5) 0144 140 002 517 568.6 68.5 95.7 L 0129 1133 02 696 487.3 95.0 95.0 L 0129 1147 02 151 716.5 99.0 99.4 L 0148 147 02 151 716.5 94.4 99.0 99.4 L 0148 147 1031 716.5 94.4 99.0 99.4 (5) 0193 148 146 102 1531 716.5 94.4 99.4 99.4 L 0113 148 146 102 131 9.5 99.4 99.4 99.4 L 0113 148 146 102 148 99.4 99.4 99.4 L 0113 148 148 92.3 94.6 99.4 99.4 L 0113 148 148 92.3 94.6 99.4 99.4 L 0119 148 148 92.3 94.6 99.4 99.4 L 0119 148 148 92.3 94.6 99.4 99.4 L 0119 148 149 1071 94.6 99.4 1877 94.6 99.4 L 0119 148 147 96.7 99.6 99.9 94.6 99.5 148 197.1 96.7 99.5 148 197.1 96.7 100 148 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 96.7 100.5 146 197.1 100.5 146 197.1 96.7 100.5 146 197.1 100.5 146 197.1 96.7 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197.1 100.5 146 197		H	.0163	.156		.342			2.90		26.3
		1	.0138	.132			115.		93.7		12.9
H         .0155         .143         .696 $487.3$ 96.9           L         .0129         .138         .454 $315.7$ 95.0           L         .0129         .138         .454 $315.7$ 95.0           L         .0140         .147         .002         .933 $647.2$ $54.4$ 99.0           L         .0140         .147         .002         .933 $647.2$ $54.4$ 99.0           L         .0140         .147         .002         .133 $573.4$ $99.0$ $99.7$ L         .0175         .147         .002         .130 $95.1$ $99.7$ L         .0175         .138         .116 $102.8$ $95.1$ $99.7$ L         .0175         .138         .012 $95.1$ $99.7$ $99.7$ L         .0195         .142         .006 $377$ $264.2$ $88.3$ $97.2$ L         .0195         .147         .006         .377 $264.2$ $88.3$ $97.2$ L         .0195         <	/p ff	(2)	.0144	.140	0.02	.517	368.6	68.5	95.7		39.8
L         .0129         .138         .434 $315.7$ 95.0 $L'$ (5)         .0145         .1147         .002         .933 $647.2$ $54.4$ 99.0           L         .0140         .1147         .002         .933 $647.2$ $54.4$ 99.0           L         .0140         .1147         .002         .933 $647.2$ $54.4$ 99.0           L         .0193         .1147         .002         .130         95.1         99.4           (5)         .0175         .1183         .115 $87.3$ 94.6           L         .0103         .1142         .006         .377 $264.2$ $88.3$ 97.2           L         .0103         .1142         .006         .377 $264.2$ $88.3$ 97.2           L         .0103         .1142         .006         .571         264.2 $88.3$ 97.8           L         .0103         .1147         .022         .584.4         197.1         96.7           L         .0203         .1147         .563         .588.4         97.8           L	1	H	.0155	.143		.696	487.3		96.9		53.5
e/       (5)       .0145       .144       .002       .933       647.2       54.4       99.0         L       .0151       .147       .002       .933       647.2       54.4       99.0         L       .0140       .147       .002       .821       573.4       99.4         (5)       .0185       .140       .002       .130       93.1       95.7         (1       .0175       .118       .002       .136       93.1       95.7         1       .0175       .118       .002       .136       93.1       95.7         1       .0175       .118       .006       .377       264.2       88.3       97.6         1       .0208       .149       .006       .377       264.2       88.3       97.2         1       .0208       .149       .006       .377       264.2       88.3       97.2         1       .019:       .133       .0208       .147       .006       .584       97.2         1       .0208       .147       .0203       .147       .097.1       96.7         1       .0208       .147       .002       .671       464.4       68.9		ſ	.0129	.138		.434	315.7		95.0		.33.4
II         .0151         .147         1.051         716.5         99.4           L         .0140         .147         .821         573.4         98.7           (5)         .0185         .140         .002         .130         93.1         95.1           (5)         .0175         .140         .002         .130         93.1         95.1           (5)         .0175         .138         .115         82.3         94.6           L         .0103         .142         .006         .377         264.2         88.3         97.2           (5)         .0208         .149         .006         .377         264.2         88.3         97.2           1         .0208         .143         .006         .377         264.2         88.3         97.2           1         .0208         .143         .0204         .95.1         96.7           1         .0208         .143         .0204         .95.1         96.7           2/         (5)         .0203         .147         .032.9         97.2           1         .0203         .147         .032.9         .97.3         96.7           1         .0203	JJ d/ c/		.0145	.144	.002	.933	647.2	54.4			71.8
L .0140 .142 .821 573.4 98.7 (5) .0185 .140 .002 .130 93.1 9.° 95.1 L .0175 .138 .146 102.8 95.7 L .0175 .138 .115 82.3 94.6 94.6 95.7 94.6 115 82.3 94.6 95.7 94.6 115 82.3 94.6 95.7 94.6 116 102.8 .149 .006 .377 264.2 88.3 97.2 98.0 110 .0208 .149 .609 418.7 99.0 98.0 24 (5) .0203 .145 .002 .671 464.4 68.9 98.9 117 .0200 .142 .531.2 95.7 95.7 95.7 95.7 95.7 95.7 95.7 95.7	1		.0151	.147		1.051	716.5		5.99.4		6.68
		1	.0140	.142		.821	573.4		98.7		63.1
II     .0193     .145     .146     102.8     95.7       L     .0175     .138     .115     82.3     94.6       L     .0102     .138     .115     82.3     94.6       (5)     .0202     .142     .006     .377     264.2     88.3     97.2       II     .0208     .149     .006     .377     264.2     88.3     97.2       II     .0208     .149     .006     .377     264.2     88.3     97.2       II     .0208     .149     .006     .377     264.4     98.3     98.0       IL     .019:     .133     .002     .671     464.4     68.9     98.9       L     .0203     .147     .002     .671     464.4     68.9     98.9       L     .0200     .147     .020     .147     99.3     97.8	KK	(2)	.0185	.140	.002	.130	93.1	9.6	95.1		10.0
L     .0175     .138     .115     82.3     94.6       (5)     .0202     .142     .006     .377     264.2     88.3     97.2       11     .0208     .149     .609     418.7     98.3     97.2       11     .0208     .149     .609     418.7     98.0       12     .019:     .133     .284     197.1     96.7       1     .0103     .147     .002     .671     464.4     68.9     98.9       2/     (5)     .0208     .147     .002     .671     464.4     68.9     98.9       1     .0208     .147     .020     .147     .022     .563     .671     464.4     68.9     98.9       1     .0200     .1147     .022     .1147     .032.3     .563     531.2     99.3       1     .0200     .1147     .563     .581.4     99.3     99.3			.0193	.143		.146	102 8.		95.7		11.2
(5)       .0202       .142       .006       .377       264.2       88.3       97.2         II       .0208       .149       .006       .377       264.2       88.3       97.2         II       .0208       .149       .006       .377       264.2       88.3       97.2         II       .0208       .149       .006       .571       264.2       88.3       98.0         L       .019:       .133       .284       197.1       96.7       96.7         L       .0203       .147       .002       .671       464.4       68.9       98.9         L       .0200       .1147       .022       .1147       .563       531.2       99.3         L       .0200       .1142       .563       531.2       99.3       97.8		1	.0175	.138		.115	82.3		94.6		8.8
II         .0208         .149         .609         418.7         98.0           L         .019:         .133         .284         197.1         96.7           L         .019:         .133         .284         197.1         96.7           L         .0203         .145         .002         .671         464.4         68.9         98.9           L         .0208         .147         .002         .671         464.4         531.2         99.3           L         .0208         .147         .563         531.2         99.3         99.3	/P TT	(2)	.0202	.142	.006	.377		88.3	97.2		29.0
L .019: .133 .284 197.1 96.7 96.7 15019: .147 .284 197.1 10.0203 .147 .002 .671 464.4 68.9 98.9 10.03 1147 .164 531.2 99.3 1147 .142 .1563 338.4 99.3 97.8 1142 .142 .142 .1563 338.4 1147 1142 .1563 1147 1147 1147 1147 1147 1147 1147 114	1		.0208	.149		.609	418.7		98.0		46.8
(5)         .0203         .145         .002         .671         464.4         68.9         98.9           II         .0208         .147         .764         531.2         99.3           L         .0200         .142         .563         388.4         97.8		ľ	:610.	.133		.284	197.1		96.7		21.8
II         .0208         .147         .764         531.2         99.3           L         .0200         .142         .563         388.4         97.8	LL d/ c/		.0203	.145	.002	.671	464.4	08.9			51.6
.0200 .142 .563 388.4 97.8	1		.0208	.147		.764	531.2		99.3		58.7
		L	.0200	.142	•	.563	388.4		97.8		43.3

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Table IV. Continued

	Avg. a/	Thickness	· Density	Density	KOII	KOIL Absorption	u	after centrifuge	Porosity
		inch	g/cu3	19 05	g/cm3 c/	0.0	SD	-	-
TIM	(2)	.0071	(4).	:509	.415	278.1	40.0	89.9	31.9
	I	.0078	.159		.516	339.8		32.0	39.8
	r	.0065	.135		.349	244.5		86.3	26.8
/P NN	(2)	.0067	171.	.006	177.	450.3	25.4	96.5	59.3
1	II.	.0071	.177		.813	169.7		97.1	62.6
	ŗ	.0064	.162		669.	406.4		96.3	53.8
NN d/ e/	(2)	.0059	.172	.005	. 855	497.5	12.1	98.4	65.8
1		.0074	.177		. 896	506.4		99.0	69.0
	ŗ	.000.	.166		.804	479.3		97.7	61.9
/P dd	(2)	r087	.289	.018	.711	246.2	15.7	. 91.5	54.7
•	н	0600.	314		.782	253.1		94.5	62.2
	-1	.0083	.268		.660	220.8		. 88.3	50.5
RR d/	(2)	.0095	.140	.004	.643	460.3	101.9	96.3	49.5
•	11	.0100	.146		061.	581.8 .		97.2	60.7
	-1	.0086	.136		.437	514.1 -		24.1	33.6
SS d/	(10)	.0098	.135	.004	.551	407.1	107.8	98.3	42.4
1	H	.0106	.141		.780	594.6		99.5	60.0
	7	2600.	.130		.396	295.9		90.6	50.5
SS d/ e/	(2)	\$600.	.132	.004	.297	224.3	44.2	90.9	22.9
1		.0104	.138		.396	287.2		\$3.9	30.5
	L	1600.	.126		.217	163.0		86.5	15.8

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Table IV. Continued

Table IV. Continued

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NBS Sample Code	Avg. a/	Thickness	Apparent Density	rent sity	KOH	KOH Absorption	E	KOH Retained after centrifuge	fuge	Porosity
		inch	g/cm ³	<u>SU b/</u>	g/cm3 c/	ole	SD	-		
J.1.	(01)	1210	.211	.018	.526	252.5	40.7	. 97.8		40.5
	6	0138	.230		.621	360.6		5.02		47.8
	:	. 0108	.172		.487	223.0		95.8		37.4
	(01)	0002	.291	.008	.585	201.2	24.9	89.9		45.0
2		5010	300		.719	247:4		7.76		55.3
••		.0086	.279		494	169.7		66.7		38.0
N	(2)	.0120	.138	.014	.350	255.9	47.9	97.6		26.9
		.0130	.152		.417	336.7		98.9		32.1
	. <b>_</b> 1	.0112	.123		.318	215.1		95.4		24.5
MX	(2)	.0205	.138	.010	.375	273.3	62.8	0.02	•	28.9
	-	.0237	.148		.419	345.1		99.3		36.5
	:	.0189	.121		. 292	205.9		. 98.6		22.4
· XX	(2)	.0143	.140	400.	.316	225.4	4.3	97.7		24.3
	-	.0158	.148		329	230.7		98.6		25.3
	: <b>-</b> ]	.0115	.135		.306	219.4		97.1		23.6
77	(5)	0163	1.370	.024	.927	67.6	3.0	98.0		71.3
:	È=	.0170	1.390		.975	70.2		98.8		75.0
	. 1	.0161	1.331		.836	62.8		90.6		64.3

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inch $g/cm^3$ SU $y/m^3$ <	NBS	N35 Sample Code	Avg. a/	Thickness	Apparent	upparent Density	НОХ	KOH Absorption		KOII Retained after centrifuge	Porosity
J2       (10)       .0168       .133       .006       .918       689.4       29.0       97.4         KK2       (5)       .0203       .137       .003       .925       731.8       90.2         KK2       (5)       .0202       .154       .003       .024       15.3       2.5       80.7         KK2       (5)       .0202       .154       .003       .029       15.3       2.5       80.7         KK2       (5)       .0202       .157       .003       .029       15.3       2.5       80.7         KK2       (5)       .0202       .159       .019       .1106       7.5       91.8         KM       .0198       .110       .0198       .110       .013       111.6       7.5.4         KM       .0198       .110       .019       .0118       .1106       7.5.4         Kmber in parentheses       represents the number of specimens used to compute the average values.       7.5.4         Highest value, L = lowest value.       .018       .11.6       7.5.4         Standard deviation       .018       .019       .018       .018         Grams of KOII absorbed per cm ³ of separator material       Samples sorbed in KOI for 162 ho	-			inch	g/cm3	No bl				0	8
KK2       (5)       .0202       .154       .003       .024       15.3       2.5       80.7       1.8         L       .0198       .149       .018       11.6       75.4       1.4         L       .0198       .149       .018       11.6       75.4       1.4         Rubber       .0198       .149       .018       11.6       75.4       1.4         I.a       .0198       .149       .018       .18.6       75.4       1.4         Rubber       .0198       .18.6       .018       .18.6       75.4       1.4         Rubber in parentheses       .0198       .018       .11.6       .018       .11.6       .25.4       1.4         Rubber in parentheses       .0198       .018       .018       .018       .11.6       .22         Rubber in parentheses       .00000000       .018       .0018       .000000000       .21.4       .22         Standard deviation       .00000000       .018       .00000000000       .21.6       .22       .22         Grans of KOII absorbed per cm ³ of separator material       .2000000000000000000000000000000000000		r,	(10) II I	.0168 .0203 .0145	.133	.006	.918 .925 .882	689.4 731.8 664.3	29.0	97.4 99.2 91.3	70.6 71.2 67.8
Number in parenthesas represents the number of specimens used to compute the average values. H = highest value, L = lowest value. Standard deviation Grams of KOH absorbed per $cm^3$ of separator material Samples extracted for two separate 24 hour periods. Sample sorted in KOH for 162 hours.	•	KK2	г = (S	.0202 .0207 .0198	.154	.003	.024	15.3 18.4 11.6	2.5	80.7 91.8 75.4	1.4
Number in parenthesis represents the number of specimens used to compute the average values. H = highest value, L = lowest value. Standard deviation Grams of KOH absorbed per cm ³ of separator material Samples extracted for two separate 24 hour periods. Sample seaked in KOH for 162 hours.											
Number in parentheses represents the number of specimens used to compute the average values. H = highest value, L = lowest value. Standard deviation Grams of KOH absorbed per cm ³ of separator material Samples extracted for two separate 24 hour periods. Sample sorked in KOH for 162 hours.											
Number in parentheses represents the number of specimens used to compute the average values. H = highest value, L = lowest value. Standard deviation Grams of KOH absorbed per cm ³ of separator material Samples extracted for two separate 24 hour periods. Sample souked in KOH for 162 hours.											
Number in parentheses represents the number of specimens used to compute the average values. H = highest value, L = lowest value. Standard deviation Grams of KOH absorbed per cm ³ of separator material Samples extracted for two separate 24 hour periods. Sample soaked in KOH for 162 hours.							*				
Standard deviation Grams of KOH absorbed per cm ³ of separator material Samples extracted for two separate 24 hour periods. Sample soaked in KOH for 162 hours.	10		n parenthes est value,	ses represents L = lowest va		er of spe	cimens used	to compute	the ave	rage values.	
Grams of KOH absorbed per cm ³ of separator material Samples extracted for two separate 24 hour periods. Sample soaked in KOH for 162 hours.	2		deviation								
Samples extracted for two separate 24 hour periods. Sample soaked in KOM for 162 hours.	51		KOII absorl	bed per cm ³ of	separato	or materia	1				•
	3 31		extracted i paked in K	for two separa 0:1 for 162 hou	ite 24 hou irs.	ır periods					

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Thickness, Apparent Density, Absorption, Retention and Porosity of Battery Separator Materials after Extracting for 24 hours in Methanoi .Table V. .

83		Thickness	Density	Density	KOH	KOH Absorption		after	KOH Retained ter centrifuge	Porosity	Fvracehlas
H 3		Inch	8/cm3	In pl	g/cm3 c/	-	SD			-	
3	(2)	.0527	.121	.003	.419	346.2	156.4		51.0	32.2	1.94
3	:	. 0496	.118		.245	205.0	•	•	30.7	18.8	
	(5)	.0105	.134	.004	.225	168.0	23.5		90.9	17.3	14.20
	= -2	.0092	.138		.169	185.9	•		93.6 87.4	19.1	
	(3)	.0148	.142	.003	.410	289.4	64.9		97.1	31.6	18.30
	=	.0163	.145.		. 270	376.2			97.9	20.8	
н	(3)	.0183	.140	.002	.103	73.8	11.5		94.4	8.0	
•	= -	.0193	.143		111.	84.2			95.5	0.6	•
		0/10.	0011			7.00			4.66	0.0	
N	(5)	.0066	.157	.006	.394	220.2	59.2		83.4	30.3	12.43
	= -1	.0064	.152		.312	219.4	i		71.5	24.0	
44	(3)	.0094	.245	.017	.348	143.6	32.4		6.06	26.8	1.25
		1600.	.218		272	105.1			89.7	20.9	
I.R	(3,	1010.	.129	.004	.290	223.2	\$5.6		89.9	22.3	17.57
		2000.	.124		.220.	172.2			87.7	1.12	

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Table V. Continued

- 26 -

Extractables	-	0.06			2.45			
Porosity	-	21.6	29.6	15.0	15.5	29.3	10.2	
KOII Retained after centrifuge	-	89.4	. 92.6	85.4	84.6	92.1	78.6	
F	SIJ	67.0			30.2	•		
KOII Absorptio	-	212.7	302.2	139.0	81.5	158.5	53.3	
KOI	8/cm3 2/	.231	.334	, 194	.202	.381	.133	
rent sity	20 P/	900.			.016			
Appa Den	g/cm3	.133	.140	.127	.249	.281	. 233	
Thickness	inch	.0098	.0108	.0085	.0104	.0108	+600·	
Avg. a/		(2)	=	-1	(10)	=	1	
At's Sumple Code		SS			/P JJ			

Number in parentheses represents the number of specimens used to compute the average values. If - highest value, L = lowest value. 10

b/ Standard deviation.

c/ Grams of KOH absorbed per cm³ of separator material.

d/ Sample extracted for 24 hours with chloroform.

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NBS Sample	Specific Resisti	vity, ohm-cm a/	Wettability,	minutes b/
Code	As Received	Extracted	As Received	Extracted
AA	<u>e/</u>	2.10	<u>c/</u>	3.6
· BB	<u>s</u> /	<u>c/</u>	· <u>c</u> /	<u>c/</u>
CC	<u>c/</u>	<u>c/</u>	<u>s/</u>	<u>c/</u>
DD	<u>c/</u>	<u>c/</u>	<u>c/</u>	<u>c/</u>
EE	1.68 d/	1.70 <u>d</u> /	1.3	52.8
FF	<u>c/</u>	<u>c/</u>	<u>c</u> /	<u>c/</u> .
GG	<u>c/</u>	<u>c/</u>	<u>c/</u>	<u>c/</u>
HH	1.81	12.48	0.6	200 + e/
n	<u>c/</u>	<u>c/</u>	<u>c/</u>	<u>c/</u>
JJ	1.92	<u>c</u> / .	0.6	<u>c/</u>
КК	<u>c/</u>	<u>c/</u>	<u>c/</u>	<u>c/</u>
LL	2.05	<u>c/</u>	0.8	<u>c/</u>
MM	<u>c</u> /	<u>c/</u>	<u>c/</u>	<u>c/</u>
NN	2.06	2.09	0.4	200 + <u>c</u> /
PP ·	3.50	3.21	2.8	24.8
RR	2.11	<u>c/</u>	0.9	<u>c/</u>
SS	c/	<u>c/</u>	<u>c/</u>	<u>c/</u>
TT	3.54	3.33	3.4	37.0
UU	6.21	6.15	0.3	17.2
vv	1.96	<u>c/</u>	10.9	<u>c/</u>
WW	27.27 f/	<u>c/</u>	193.8	<u>c</u> ,
xx	2.28	<u>c/</u>	3.9	<u>c/</u>
22	9.52	9.56	62.8	56.4
J2	1.49 <u>s</u> /	1.49	3.0	13.0
KK2	1.86	<u>c/</u>	2.7	<u>c/</u>
		<u>c</u> /	2.7	<u>c/</u>

# Table VI. Specific Resistivity and Wettability of Battery Separator Materials

a/ Average of 10 specimens

b/ Average of 5 specimens

## Table VI. Continued

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- d/ Sample is squeezed when placed in the cell for test and does not recover before thickness measurement is made. This difference in thickness would make the resistivity answer lower than the one reported.
- e/ Test discontinued after 200 minutes.
- f/ Specimens not completely wet.

g/ Average of 20 specimens. -

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NBS Sample		As Rece	ived		Extract	ted
Code		o reach cm	cm/24 hrs.	Time to 6.0	o reach cm	cm/24 hrs
	min	sec		mii.	sec	
AA	-		<u>b/</u>	-		<u>c/</u>
BB			<u>b</u> /	-		<u>b/</u>
CC		••	<u>b</u> /	-		<u>b</u> /
DD		•	<u>b/</u>		•	<u>b</u> /
EE	-		3.0	- 1		1.9
FF		-	<u>b</u> /		-	<u>b/</u>
GG		-	<u>b</u> /		•	· <u>b/</u>
HH	6	44			•	0
11		- 6	<u>b</u> /			<u>b/</u>
JJ	4	32	-	-	-	0
KK			<u>b</u> /	-		<u>b</u> /
LL	4	. 8	-		-	0
MM ·			<u>b/</u>			<u>b/</u>
NA	. 4	15	-			0
PP	72	0		•	-	3.6
RR	8	5		-		0
SS			<u>b</u> /	-		<u>b/</u>
TT	16	.22	-	-		3.2
UU	2	39	-		-	5.1
vv	9	7	-		. 1	0
KW	-		1.0	-		0
xx	3	19	-	-	2	0
ZZ		-	<u>c</u> /	-	-	<u>c/</u>
J2	411	30		-	-	<u>c/</u>
КК2	50	3			-	0.4

# Table VII. Wicking of Battery Separator Materials a/

a/ Average of 5 specimens

b/ Sample would not wet, test could not be performed.

c/ Time for test was greater than 7 hours but less than 24 hours.

NBS Sample		As Re	eceived		Extr	racted
Code	and the second se	ne	Circle Range	Ti	ne	Circle Range
	min	sec		min	sec	
. AA		<u>b/</u>		3	26	2 1/2 - 3
BB		<u>b/</u>			<u>b/</u>	
CC		b/			b/	
DD		b/			<u>b</u> /	
EE		1.6	2 c/		<u>b</u> /	
FF		b/			b/ .	• •
GG		b/			<u>b/</u>	
HUI		19.4	3 - 4		b/	
11		b/ ·			b/	
JJ		44.2	5		<u>b</u> /	
КК		b/			<u>b</u> /	
LL		27.4	4 - 5		<u>b/</u>	
MM		<u>b/</u>			b/	
NN .		22.4	4 - 5		b/	
PP	10	11	3 1/2 - 4		b/	
RR	-	3.0	3 - 4		<u>b/</u>	
SS		<u>b</u> /			b/	
TT	-	8.4	3 - 4		b/	
UU		5.6	3 1/2 - 4		<u>b/</u>	
vv	-	9.2	3 - 3 1/2		<u>b/</u>	
WW	2	33	2 - 4 1/2		b/	
xx	-	11.2	4 1/2 - 5		b/	
ZZ	6	48 d/	4 - 5	1	49 d/	2 1/2 - 4
J2	9	9	2 - 3	8	22	2 - 2 1/2
KK2	-	3.3	2 - 2 1/2		<u>b/</u>	

Table VIII. Wet-Out Time of Battery Separator Materials a/

a/ Average of 5 specimens.

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b/ KOH in contact for one hour and specimen showed no absorption or spreading.

c/ KOII penetrates through the surface layer with very little spreading but spreads in fibers under surface to a range of 4 to 4 1/2 in an average of 25 seconds.

## Table VIII. Continued

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d/ The results given are with the KOH in contact with the asbestos side of the sample. One specimen was tested with the KOH in contact with the coated side and the results were: as received 32 min 20 sec range 3; extracted 40 min range 3.

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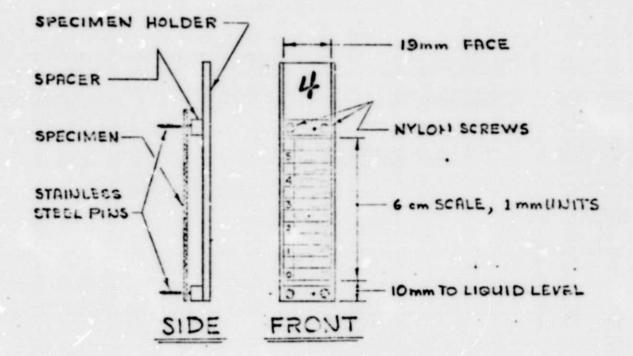
NBS Sample	No. of	Seconds to Pass	300 ml of Air
Code	Specimens	As Received	Extracted
		Avg.	Avg.
AA	. 6	13.8	11.0
EE	6	2.1	1.9
HIH	6	10.9	6.5
JJ	6	15.6	11.4
LL	6	25.6	21.0
NN	6	12.8	7.7 .
PP	10 .	5.0	4.1
RR	6	13.6	8.1
SS	6	8.7	8.6
TT	6	1.9	1.4
UU	6	4.)	4.0
vv	6	3.4	1.1
WW	6	3.0	3.4
xx	6	2.6	2.4
22	-	b/	
J2	20	2.4	2.4
KK2	10	19.0	16.6

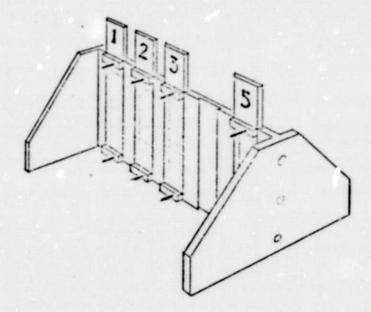
## Table IX. Air Permeability of Battery Separator Materials a/

a/ Some samples were not subjected to this test at the request of the sponsor.

b/ One specimen tested, the result was 88 hours to pass approximately 175 ml of air.

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# PERSPECTIVE VIEW

RACK AND SPECIMEN HOLDER

Figure 1. Rack and Specimen holders for wicking test. All materials of acrylic plastic sheet unless otherwise noted. Pins on individual holders are offset to the right to facilitate realing of scale.

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## References

- Adapted from: "Characteristics of Separators for Alkaline Silver Oxide Zinc Secondary Batteries," Ed., Cooper, J.E. and Fleisher, A., report from the Air Force Aero Propulsion Laboratory, Wright-Patterson Air Force Base, Ohio.
- Ibid., "Electrical Resistance: Alternating Current Method," Chapter 4, Section b.