DEVELOPMENT OF BATTERY SEPARATOR MATERIAL PROCESS

by

L. M. Adams
W. W. Harlowe, Jr.

FINAL REPORT

SwRI Project No. 01-2015-01
Contract No. 951718

Prepared for

Jet Propulsion Laboratory
California Institute of Technology
4800 Oak Grove Drive
Pasadena, California 91103

Attn: H. E. Patterson
Senior Contract Negotiator
Mail Station 190-212

April 1970

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April 1970

Approved:

John T. Goodwin

John T. Goodwin, Director
Department of Chemistry and
Chemical Engineering

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I. ABSTRACT, CONCLUSIONS AND RECOMMENDATIONS

This report describes the work on a process for the manufacture of a heat sterilizable battery separator.

The objectives of this work were to optimize an existing method for the manufacture of the separator material, to scale up the process and to produce 10,000 ft^2 of the material for experimental purposes. Other objectives were to improve the performance of the separator material and to study other materials for preparing battery separator materials.

The preliminary work indicated that the divinylbenzene crosslinked film was difficult to graft with acrylic acid because of materials introduced into the film during crosslinking. Reversal of this procedure, i.e., grafting with subsequent crosslinking, resulted in a product having a lower and more uniform electrical resistance.

The results of a 2^5 factorial experiment for studying the radiation-initiated grafting of polyethylene film indicate (1) higher temperatures within the range of 77-125°F favor the production of lower and more uniform electrical resistance; (2) a nitrogen atmosphere favors lower and more uniform electrical resistance; (3) dose rates studied (0.0125 to 0.0210 Mrad/hr) have little effect on the electrical resistance of the resulting grafted film; (4) total doses studied (0.671 to 1.700 Mrad) have little effect on electrical resistance;

(5) the degree of crosslinking obtained with the procedure employed has little effect on the electrical resistance; (6) within experimental error, the total dose, dose rate, and crosslinking have little effect on the tensile strength; and (7) high temperature and nitrogen atmosphere decrease tensile strength, although all values obtained were acceptable.

Of the organic solvents evaluated in the grafting solution, benzene was the best from the standpoint of uniformity of electrical resistance. Polyethylene film can be grafted with aqueous acrylic acid solutions to produce material with low electrical resistance (2-3 milliohms-inch^2), but the product cannot be recovered unless ferrous sulfate, potassium ferrocyanide, or potassium ferricyanide is used in the grafting solution to prevent gelation of the homopolymer.

Two reactors, constructed of aluminum, were used for radiation grafting of large quantities (500 feet per reactor) of polyethylene film with acrylic acid and crosslinking large quantities of grafted polyethylene film with divinylbenzene. A film processing machine for continuously neutralizing and washing the grafted film was designed, constructed, and operated for processing large quantities of battery separator material.

Battery separator material, in excess of 10,000 linear feet, was shipped to the Jet Propulsion Laboratory. The electrical resistance throughout most of the material varied from 6 to 21 milliohms-inch^2 with an average of 12.

While testing various types of polyethylene, samples of 710M, 110E, 510M, 560E and 400 polyethylene films were obtained from the Dow Chemical Company to determine a suitable replacement. The first four samples were free of additives, and the last one contained 1000 ppm of calcium carbonate. All of the samples grafted uniformly, and polyethylene film 400 was chosen because of its uniformity and availability and quality of battery separator material prepared from it. The presence of the calcium carbonate did not interfere with the acrylic acid grafting step and did not appear to be detrimental to the battery separator.

Of the materials evaluated as the interlayer material in the rolls of polyethylene film, cotton cheesecloth and nylon mesh were the best because of ease of handling and uniformity obtained in the grafted products. Cotton cheesecloth was chosen for the preparation of large quantities of acrylic acid-grafted film because of cost. At the lowest concentration (10 wt %) of acrylic acid in the grafting solution evaluated, uniformly low electrical resistances were obtained for the acrylic acid-grafted products when Dow 400 polyethylene film was used with cheesecloth interlayer. A satisfactory product could not be obtained at this low acrylic acid concentration when either the initially supplied polyethylene film or paper toweling interlayer material was used. The ability to use low concentrations of acrylic acid in the grafting solution is important because of the high exotherm encountered in the grafting procedure. At the originally used acrylic acid concentration of 25 weight percent, the exotherm becomes excessive when using the large reactors. At 15 weight percent, the exotherm remains within acceptable limits.

Throughout the experimental work, it was demonstrated that the electrical resistance of the acrylic acid-grafted film was usually lower when neutralized and washed at 97°C than when neutralized and washed at 80°C. Crosslinking of the grafted film with divinylbenzene increased the electrical resistance somewhat; the amount of the increase varied with the composition of the grafted film.

The degree of grafting of polyethylene film with acrylic acid [weight of poly(acrylic acid) in grafted film/weight of starting film] is proportional to the concentration of acrylic acid in the grafting solution. At 25 weight percent acrylic acid in the grafting solution, the degree of grafting was 0.87. The degree of grafting decreased to 0.70 at 20 weight percent, 0.52 at 15 weight percent, and 0.38 at 10 weight percent acrylic acid. In all cases, the grafting solutions contained a 5:1 acrylic acid to carbon tetrachloride ratio.

The electrical resistance throughout each 25-foot roll of the aforementioned grafted film was uniform. The values were 6-7, 9, 10, 11-13, and 14-16 milliohm-inch^2 for 25, 20, 15 and 10 weight percent acrylic acid, respectively.

In another series where the acrylic acid content in the grafting solution was maintained at 15 weight percent and the carbon tetrachloride varied from 0 to 15 weight percent, the degrees of grafting were 0.97, 0.59, 0.48, 0.50 and 0.44 at carbon tetrachloride concentrations of 0, 3, 5, 7.5, and 15 weight percent, respectively.

The electrical resistance was uniform throughout each portion which was neutralized and washed at 97°C. The range for each sample was 9-12, 11-13, 11-17 and 14-16 milliohm-inch^2 for carbon tetrachloride concentrations of 3, 5, 7.5 and 15 weight percent, respectively. On crosslinking these materials with divinylbenzene, the average electrical resistance of the samples increased to 16, 19, 36, and 89 milliohm-inch^2 for carbon tetrachloride concentrations of 3, 5, 7.5 and 15 weight percent, respectively. On sterilizing the grafted and crosslinked films, the average electrical resistances were 15, 37, 75 and 274, respectively.

Polyethylene film grafted with methacrylic acid was quite similar to the acrylic acid-grafted material in most of its properties. However, the properties were more sensitive to the carbon tetrachloride content of the grafting solution. As a consequence, it is recommended that the grafting of polyethylene film with methacrylic acid be done in the absence of carbon tetrachloride.

Polyethylene film is readily grafted with aqueous solutions of acrylic acid to yield a grafted material of low electrical resistance (2-3 milliohm-inch^2), but the homopolymer forms a tenacious rubbery gel which makes it extremely difficult to recover the grafted film. The addition of ferrous sulfate, potassium ferrocyanide or potassium ferricyanide to the aqueous grafting solution prevents this and makes it possible to recover the grafted film. The last two materials can be readily washed from the grafted film without danger of iron contamination, and the electrical resistance of the grafted films is low (5-6 milliohm-inch^2). Similar results were obtained with aqueous methacrylic acid solutions.

When polyethylene film is cografted with acrylic acid-vinyltoluene solutions free of terminator, the electrical resistances throughout the products prepared at vinyltoluene to acrylic acid ratios of 1:4, 1:1.5, 1.5:1 and 4:1 were 8-9, 13-15, 80-89, and 341-380 milliohm-inch^2, respectively. However, on crosslinking these materials with divinylbenzene, the average electrical resistance increased to 14, 56, 547 and >3000 milliohm-inch^2, respectively. When carbon tetrachloride (chain terminator) was present in the grafting solutions, the electrical resistance of the product increased more rapidly with an increase in the vinyltoluene to acrylic acid ratio for both the grafted and the grafted and crosslinked materials.

To prepare grafted polyethylene with side chains having carboxyl groups on adjacent carbons, attempts were made to graft polyethylene with aconitic acid (1, 2, 3-propenetricarboxylic acid) and with itaconic acid (methylenebutanedioic acid). The attempts were not successful, but polyethylene film could be cografted with a mixture of either acid and acrylic acid. A grafting solution containing 10 weight percent acrylic acid and 15 weight percent aconitic acid in methanol produced a grafted polyethylene film having an electrical resistance of 14-18 milliohm-inch^2. With itaconic acid and acrylic acid in the same concentrations in methanol, the electrical resistance of the grafted product was 112 milliohm-inch^2.

Silver, zincate, and hydroxyl ion migration rates were determined on a number of grafted and grafted and crosslinked polyethylene films. It was found that silver ion migration rates through acrylic acid-grafted polyethylene films increased as the degree of grafting increased. The silver ion migration rates ranged from 0.85 x 10^{-2} grams of silver/hr-inch^2-mol at a degree of grafting of 0.38 (10 weight percent acrylic acid and 2 weight percent carbon tetrachloride in benzene grafting solution) to 4.50 x 10^{-2} grams of silver/hr-inch^2-mol at a degree of grafting of 0.87 (25 weight percent acrylic acid and 5 weight percent carbon tetrachloride in benzene grafting solution).

When the carbon tetrachloride content in a 15 weight percent acrylic acid grafting solution was increased from zero (0.97 degree of grafting) to 15 weight percent (0.44 degree of grafting), the silver ion migration rate decreased from 3.02 x 10^{-2} to 0.30 x 10^{-2} grams of silver/hr-inch^2-mol.

Increasing the divinylbenzene content of the crosslinking solution from 1 volume percent to 16 volume percent decreased the silver ion migration rate from 4.10 x 10^{-2} to 1.37 x 10^{-2} grams of silver/hr-inch^2-mol.

The silver ion migration through polyethylene film cografted with vinyltoluene-acrylic acid mixtures without terminator decreased from 5.40 x 10^{-2} grams of silver/hr-inch^2-mol at a vinyltoluene to acrylic acid ratio of 1:4 to 0.16×10^{-2} grams of silver/hr-inch^2-mol at a ratio of 4:1. The material crosslinked with divinylbenzene behaved in a similar manner. When carbon tetrachloride was present in the cografting solution, the silver ion migration rate decreased from 2.98×10^{-2} grams of silver/hr-inch^2-mol at a ratio of 1:4 to 0.31×10^{-2} grams of silver/hr-inch^2-mol at a ratio of 1:1.5.

Polyethylene film cografted in a benzene solution containing 25 weight percent acrylic acid, 5 weight percent carbon tetrachloride, and 10 weight percent divinylbenzene had an electrical resistance of 90 milliohm-inch^2 and a silver ion migration rate of 0.39×10^{-2} grams of silver/hr-inch^2-mol.

Polyethylene film grafted or cografted in aqueous solutions of acrylic acid generally exhibited high silver ion migration rates.

Acrylic acid-grafted films prepared with various chain terminators in the grafting solution were evaluated. Those that appear to be of greatest interest were prepared using acetone, carbon disulfide, and sulfur. These terminators appeared to function better in 25 weight percent acrylic acid solution than in 15 weight percent acrylic acid solution. With 25 weight percent acrylic acid and acetone at 1 and 2.5 weight percent levels in the grafting solution, the grafted and crosslinked products had silver ion migration rates of 0.42×10^{-2} and 0.33×10^{-2} grams of silver/hr-inch^2-mol and electrical resistances of 25 and 26 milliohm-inch^2 respectively. The grafted and crosslinked polyethylene films prepared from 25 weight percent acrylic acid in benzene plus 2.5 weight percent carbon disulfide or saturated with sulfur had rates of 1.14×10^{-2} and 0.41×10^{-2} grams of silver/hr-inch^2-mol and electrical resistances of 25 and 22 milliohm-inch^2, respectively.

The presence of some organic compounds with fused aromatic rings or acetylenic groups or some inorganic materials in the acrylic acid grafting solution lowered the silver migration through the grafted film, but they generally increased the silver pick-up excessively.

The various 1.0 mil polyethylene films evaluated produced acrylic acid-grafted films having essentially the same silver ion migration rates.

Polyethylene films grafted with methacrylic acid had silver ion migration rates in the same range as obtained with acrylic acid-grafted polyethylene film.

On the basis of an equal wet film thickness, the hydroxyl ion migration rate through acrylic acid-grafted and crosslinked polyethylene film neutralized and washed at 97°C decreased from 10.40×10^{-3} to 5.50×10^{-3} mol/min-inch^2-mil when the acrylic acid concentration in the grafting solution decreased from 25 weight percent to 10 weight percent.

With methacrylic acid, no hydroxyl ion correlation was obtained when the acid concentration in the grafting solution varied from 25 to 10 weight percent.

Varying the carbon tetrachloride concentration in the grafting solution from 0 to 15 weight percent decreased the hydroxyl ion migration from 6.10×10^{-3} to 1.73×10^{-3} mol/min-inch^2-mil for the grafted films which were neutralized and washed at 97°C.

The hydroxyl ion migration through polyethylene films cografted with vinyltoluene-acrylic acid mixtures decreased from 7.65×10^{-3} to 4.65×10^{-3} mol/min-inch^2 as the ratio of vinyltoluene to acrylic acid in the grafting solution increased from 1:4 to 4:1.

Correlation of the effect of carbon tetrachloride concentration in the grafting solution on zincate ion migration was poor. The zincate migration rates ranged from 3.82×10^{-6} to 0.56×10^{-6} mol/inch^2-min-mil.

A good correlation was obtained between zincate ion migration and the ratio of vinyltoluene to acrylic acid in the grafting solution for the cografted polyethylene. The zincate ion migration decreased from 7.13×10^{-6} to 0.01×10^{-6} mol/inch^2-min-mil on increasing the ratio from 1:4 to 1.5:1.

The following conclusions can be made from the results of the experimental work:

1. Grafting of polyethylene film with acrylic acid followed by crosslinking with divinylbenzene yields a battery separator material superior to that produced by reversing the order of grafting and crosslinking. The product is much more uniform, and the electrical resistance is lower.

2. Results of the factorial experiment indicate that elevated temperatures within the limits studied and a nitrogen atmosphere over the grafting solution favor uniformity and low electrical resistance of the product. Dose rate and total dose within the limits studied have no effect on electrical resistance. The degree of crosslinking obtained by the procedure used has only little effect on the electrical resistance.

3. Acrylic acid-grafted polyethylene film having a low silver ion migration rate can be produced by proper choice and concentration of the chain terminator in the grafting solution. Acetone, carbon disulfide, and sulfur were the best evaluated.

4. Low silver ion migration rates can also be obtained by cografting polyethylene film with mixtures of acrylic acid and hydrocarbon-type vinyl monomers. The vinyl monomer may be mono- or difunctional, An increase in electrical resistance occurs.

5. The degree of grafting of polyethylene film with acrylic acid is proportional to the acrylic acid concentration and inversely proportional to the carbon tetrachloride concentration in the grafting solution.

6. Silver ion migration through acrylic acid-grafted polyethylene film decreases as the ratio of carbon tetrachloride to acrylic acid in the grafting solution increases, as the degree of grafting decreases, and as the concentration of divinylbenzene in the crosslinking solution (in nitrogen atmosphere) increases.

7. Polyethylene film can be cografted with acrylic acid in admixture with vinyl monomers which will not graft polyethylene when used alone.

8. An acceptable procedure for preparing large quantites of battery separator material has been developed. A device for continuously neutralizing and washing acrylic acid-grafted polyethylene film has been operated successfully.

9. Dow 400 polyethylene film is an excellent base stock for preparing battery separator material.

10. Cotton cheesecloth is an excellent material for use as an interlayer material in the grafting procedure.

11. Aqueous acrylic acid and methacrylic acid solutions can be used for grafting polyethylene film. The products generally exhibit higher silver ion migration rates and lower electrical resistances than materials prepared using organic solvents.

12. Only minor differences occur in the properties of battery separator material prepared from acrylic acid and that prepared from methacrylic acid.

It is recommended that:

1. Further work be conducted on the effect of various chain terminators on the grafting of polyethylene film with acrylic acid and with methacrylic acid.

2. Further studies be conducted on the cografting of polyethylene film with mixtures of acrylic acid or methacrylic acid and mono- and difunctional vinyl monomers to further improve the properties of battery separator material.

3. Investigation be carried out on the use of the free-acid form of acrylic acid-grafted polyethylene in the preparation of silver oxide-zinc batteries or grafted film that has been neutralized in 40 weight percent potassium hydroxide solution.

II. INTRODUCTION

A process for the preparation of a sterilizable battery separator material was developed by another organization for the Jet Propulsion Laboratory. The process involved the crosslinking of low density polyethylene film with divinylbenzene with subsequent grafting of the resultant film with acrylic acid. Both reactions were initiated by radiation from a cobalt-60 source. Selected samples of the material performed well in silver oxide-zinc batteries, but it was considered necessary to improve the reproducibility of the process and the uniformity of the separator material.

Two of the objectives of the research work were to optimize the preparative procedure so that a sterilizable battery separator material having a uniformly low electrical resistance could be produced in large quantities and to supply 10,000 feet of battery separator material to the Jet Propulsion Laboratory for their use.

Other objectives were to construct a film processing machine, develop methods of producing battery separator material having improved properties, and evaluate other monomers for preparing battery separator material. The period covered by this report is October 25, 1966 through February 20, 1970.

III. EXPERIMENTAL

A. Analytical Procedures

Methods used for analysis of the materials prepared on this program are described or referenced in Appendix A.

B. Radiation Configuration and Dosimetry

Grafting and crosslinking operations were performed in the Southwest Research Institute's radiation effects facility. The configuration which was used with the experimental samples (25- and 30-foot rolls) is shown in Figure 1. Eight glass reactors mounted on individual turntables were irradiated at the same time. The cobalt-60 holder was semi-parabolic and contained approximately 10,000 curies of cobalt-60.

Mapping of the gamma flux in the target area indicated workable isodose volumes of 20,000 rad over the eight small reactors. The volume was almost semicircular and symmetrical to the center line of the source with the center reactors offset slightly farther from the source.

Detailed dosimetry, using the Bausch and Lomb cobalt glass chip technique, was performed. The entire assembly was adjusted, in position or source strength, until the incident dose rates were those specified within the error of the dosimetry system. Dosimeters were placed within the reactors (in air) at the top, center and bottom of the volume to be occupied by the polyethylene rolls and near the inside surface of the reactors. The dosimeters were wrapped in lead foil to shield them from low-energy "knockout". The reactors were rotating in each case so that the measured dose rate is an average of the small differences in rate across the diameter of each reactor. The dose rate at various distances were determined and then redetermined at approximately six-month intervals to correct for change in activity of the cobalt-60. The uniformity of the dose rates over the eight reactors is shown in Table 1.

C. Crosslinking of Polyethylene Film with Subsequent Grafting

Eight 30-foot rolls of polyethylene film were crosslinked with divinylbenzene (inhibitor removed by washing with 9 weight percent potassium hydroxide) at 25°C by a procedure similar to that given in Appendix C. One roll was shipped to the Sponsor for evaluation, one roll was examined for changes in tensile strength, and the remaining rolls were saved for further treatment with acrylic acid. As expected, the tensile strength of the crosslinked film (Table 2) was higher than that of the untreated film.

Three rolls of the divinylbenzene-crosslinked polyethylene were grafted in a 25 weight percent acrylic acid solution using a procedure similar to that described in Appendix C. The grafting was not uniform throughout the roll and the electrical resistance (Table 3) varied from 14 to >3000 milliohm-inch^2.

Sample Number 2 was prepared by grafting with uninhibited acrylic acid (inhibitor removed by vacuum distillation of the acid), while the others were grafted with inhibited acid. There appeared to be a slight increase in the amount of grafting of Sample Number 2 as indicated by the lowering of the electrical resistance throughout most of the roll. However, a definite conclusion cannot be made as to the value of removing the inhibitor from the acrylic acid because of the nonuniformity of the grafting.

All grafting solutions were monitored for temperature change during irradiation, and no exotherm occurred.

D. Grafting with Subsequent Crosslinking

In a discussion of the above results with the technical representative of the Sponsor, it was agreed that an evaluation of polyethylene film, which was grafted first and then crosslinked, should be made before proceeding with the proposed work.

Two 30-foot rolls of untreated polyethylene film were grafted with acrylic acid. One roll was analyzed, and it was found that nonuniform grafting took place on the outermost four feet of the roll of film. This was determined by infrared studies, appearance, and electrical resistance in 40 weight percent potassium hydroxide solution.

The infrared instrument was set for 1540 cm^{-1} (6.4μ), and a strip of the film was moved through the film holder. No grafting was indicated on the first foot of film. There was intermittent grafting on the second, third, and fourth foot and continuous grafting for the remainder of the roll. When infinite absorbance was obtained, it was noted that the film swelled considerably in water and had a reticulated appearance when wetted with water. When the absorbance was nil, the film, on wetting with water; had a smooth appearance much the same as the untreated film. From four feet to the innermost part of the roll, the electrical resistance in 40 weight percent potassium hydroxide solution was well below the desired maximum value of 100 milliohm-inch^2 and ranged from 2 to 35 milliohm-inch^2 (Table 4).

A sample of the grafted film withstood sterilization in 40 weight percent potassium hydroxide solution at 142°C for 72 hours (Table 5), and the electrical resistance decreased from 17 to 9 milliohm-inch^2 on sterilization.

Each of the aforementioned rolls of grafted film was divided into equal parts. One part of each roll was crosslinked with inhibited divinylbenzene, and the other part of each roll was crosslinked with uninhibited divinylbenzene. Properties of the products are presented in Table 6. It is difficult to draw definite conclusions as to the effect of inhibitor in the divinylbenzene because of the small number of samples. However, crosslinking appeared to increase the tensile strength of the film without having a detrimental effect on the electrical resistance.

From the aforementioned results, it appears that grafting with acrylic acid with subsequent crosslinking with divinylbenzene produces a product with lower and more uniform electrical resistance. All of the above grafting with acrylic acid was conducted in air at a dose rate of 0.21 Mrad per hour for a total dose of 1.430 Mrad.

To determine the reproducibility of the improved procedure and to prepare samples for evaluation by the Sponsor, ten 30-foot rolls of low-density polyethylene film were grafted with acrylic acid and then crosslinked with divinylbenzene. The results are presented in Table 7. As reported previously, the grafting was erratic in the outermost four feet of each roll, and the electrical resistance of the remainder of each roll was well below the maximum permissible value (100 milliohm-inch^2) and fairly uniform.

E. Factorial Experiment

A one-half replicate of a 2^5 factorial experiment (Table 8) was conducted to study the parameters which were felt to be pertinent to the improved procedure. The data obtained for the material from the runs listed in the factorial experiment are presented in Tables 9-24. A summary of the electrical resistance values is presented in Table 25.

In all cases, the composition of the grafting solution was 25 weight percent acrylic acid, 70 weight percent benzene, and 5 weight percent carbon tetrachloride. The crosslinking solution, when used, was 1.0 volume percent divinylbenzene, 1.0 volume percent benzene, and 98 volume percent methanol. Gas pockets were removed from the rolls by alternately lowering and raising the pressure in the reaction vessel. The grafted film was soaked for one hour at 80°C in five percent potassium hydroxide solution followed by a soak for one hour in distilled water at 80°C. The wet films were permitted to dry in air at ambient conditions (about 75°F and 50 percent relative humidity). Crosslinked films were rinsed with benzene after crosslinking and air dried. Samples for resistance, tensile strength, and elongation measurements were soaked in 40 percent potassium hydroxide solution for at least 18 hours before measurements were made.

Analysis of the data from the factorial experiment indicates the following:

1. The higher temperature (125°F) favors lower and more uniform electrical resistance values throughout the roll,

2. A nitrogen atmosphere favors lower and more uniform electrical resistance values throughout the roll,

3. The lower dose rate (0.0125 Mrad/hr) produces film which is grafted throughout the roll in both air and nitrogen atmospheres. With the high dose rate (0.021 Mrad/hr), grafting usually does not occur in air at ambient temperature on the outermost few feet. If the outermost few feet of the roll are not considered, dose rate in the range studied has little effect on electrical resistance.

4. Total dose, within the range studied (0.671 to 1.700 Mrad), has little effect on electrical resistance.

5. The degree of crosslinking obtained with the procedure employed has little, if any, effect on electrical resistance.

6. Within experimental error, the total dose, dose rate, and crosslinking have little effect on tensile strength. The higher temperature as well as a nitrogen atmosphere decreases the tensile strength. However, the tensile strengths obtained in all cases are acceptable.

7. A combination of the higher temperature and a nitrogen atmosphere produced modified film which was difficult to recover from the roll. It appeared to be overgrafted, and it is believed that a much

lower total dose under these conditions would produce a good battery separator material of low electrical resistance.

In no case was an exotherm produced during grafting in the presence of air at 77°F (Table 26). However, grafting in the presence of air at 125°F usually exhibited an exotherm, although the exotherm was not as great as obtained with a nitrogen atmosphere at this temperature (Table 26).

F. Effect of Sterilization

The effect of sterilizing a number of samples of modified film obtained during the statistical experiment is shown in Table 27. The samples were picked in a random fashion as only one sample was chosen for each set of conditions used in the statistical experiment. Most of the results were typical. However, the resistance of one sample (52-13) which had a relatively high resistance of 109 milliohm-inch^2 decreased to 9 milliohm-inch^2. This sample also had a relatively large increase in thickness on sterilization.

G. Miscellaneous Experiments

To check the reproducibility of the grafting of battery separator material at ambient temperature in a nitrogen atmosphere and to supply additional material for use by the Sponsor, fifteen rolls of polyethylene film were grafted and crosslinked. The reproducibility of the electrical resistance (Tables 28 and 29) was good, and the material was shipped to the Sponsor.

Good reproducibility of electrical resistance was also obtained when the grafting was carried out at 125°F in the presence of air (Tables 18, 30 and 31).

As higher temperatures and nitrogen atmosphere improved the grafting of low-density polyethylene film, attempts were made to graft divinylbenzene-crosslinked polyethylene film with acrylic acid at 90°F in nitrogen (Table 32), at 125°F in air (Table 33), and at 125°F in nitrogen (Table 34). In nitrogen at 90°F and in air at 125°F, grafting occurred only on the innermost part of the roll, while about one-half of the roll grafted in nitrogen at 125°F.

Washing of the crosslinked film in boiling five percent potassium hydroxide solution permitted grafting (90°F in nitrogen) to occur throughout the roll (Table 35); however, the resistance values were high throughout most of the roll. Lower resistance values would probably be obtained in nitrogen at 125°F. It appears that material from the divinylbenzene solution which acts as an inhibitor remains in the film after crosslinking, and satisfactory battery separator material could probably be prepared from the crosslinked film if a proper washing procedure is used. Thus, grafting with subsequent crosslinking is the preferred procedure.

Grafting of polyethylene film with acrylic acid in a nitrogen atmosphere at a low dose rate of 0.006 Mrad/hr and a total dose of 0.408 Mrad results in borderline grafting in some cases (Table 36). In all cases, the outer portion of the roll did not graft. Sample

Numbers 187 and 188 exhibited low electrical resistance on the portion of the film which grafted.

A higher total dose at this low dose rate would probably result in a satisfactory product, but irradiation time would become excessive. Two of the samples were crosslinked and analyzed (Table 37). The electrical resistance was low and fairly uniform. It decreased on sterilization, but the uniformity after sterilization was not as good as before sterilization. The tensile strength and elongation were good, and the dimensional changes on wetting with 40 percent potassium hydroxide solution and on sterilization were relatively low.

The grafting procedure was standardized at a dose rate of 0.012 Mrad/hr and a total dose of 0.815 Mrad.

H. <u>Other Solvents for Grafting Solution</u>

1. <u>Organic Solvents</u>

None of the organic solvents evaluated as substitutes for benzene in the grafting solution was as satisfactory as benzene for producing film with uniformly low electrical resistance (Table 38). Toluene and xylene were very poor and behaved as if an inhibitor was present. The replacement of one-half of the toluene with methanol was an improvement, but the mixture was not as satisfactory as benzene.

The replacement of one-half of the benzene with methanol (Tables 38 and 39) offered no obvious advantages.

Cyclohexane and VM&P naphtha appear to be good solvents for use in the grafting solution, as the electrical resistances of the grafted films were low (Table 40). These solvents cannot be compared directly with xylene and toluene mentioned above as a different polyethylene film was grafted.

2. Aqueous Systems

Polyethylene film is readily radiation grafted in aqueous acrylic acid solutions to yield a product of very low electrical resistance (2-3 milliohm-inch^2), but the homopolymer forms a tenacious rubbery gel which makes it extremely difficult to recover a grafted film. It was found that the addition of ferrous sulfate to the aqueous acrylic acid solution prevented gelation, and the grafted film could be recovered readily. The electrical resistance before sterilization is somewhat higher when the ferrous sulfate is used, but it is still fairly low and uniform (Table 41). After sterilization, some of the specimens had an electrical resistance of less than 0.5 milliohm-inch^2. The physical properties of the film were good.

It was found that potassium ferrocyanide and potassium ferricyanide also prevent gelation of the aqueous acrylic acid grafting solution. These materials have an advantage over ferrous sulfate in that they can be completely washed from the grafted film, whereas film prepared using ferrous sulfate requires special treatment to completely remove iron salts. The grafted films have a uniform and very low electrical resistance (Table 42). These samples tore into two pieces during the hot water wash. As a consequence, random samples had to be taken for electrical resistance measurements. Lower concentrations of acrylic acid in the grafting solution may prevent this.

Other salts such as nickel sulfate and cobalt sulfate were not effective in preventing gelation of the aqueous grafting solution at the standard or lower irradiation total dose. However, the small amounts of film recovered had very low electrical resistance (2-3 milliohm-inch^2).

It is reported in the literature that a partially neutralized acrylic acid solution polymerizes to a low molecular weight polymer when catalyzed by free radicals. It is also reported that the presence of mercaptosuccinic acid has the same effect in aqueous solutions. Neither prevented gelation of the aqueous acrylic acid when irradiated.

I. Effect of Various Additives in Grafting Solution

A considerable amount of homopolymer precipitates during the grafting of polyethylene film when using organic solvents, and it would be highly desirable to eliminate or minimize it. A number of metals salts and organic materials were evaluated to accomplish this. Of the metal salts evaluated (Table 43), only iron, cerium, cobalt, and nickel salts eliminated homopolymer precipitation. It is known that residual iron salts in the grafted film are detrimental to silver-zinc batteries, but the effect of the other metals is not known. A procedure could be developed to remove all of the metal salts from the film, but the extra effort and cost would probably more than nullify the advantage of eliminating homopolymer precipitation.

Of the organic materials evaluated (Table 43), acenaphthene, Surfynol 104, and ethynyl cyclohexanol show promise for preventing or minimizing homopolymer formation. The effect of these materials on the life of the silver-zinc battery is unknown.

Some of the aforementioned grafted films were crosslinked in a one volume percent divinylbenzene solution. The analytical data for these materials are presented in Table 44.

J. Chain Terminator Studies

1. Effect of Chain Terminator Concentration

When polyethylene film is grafted with acrylic acid, the concentration of chain terminator (carbon tetrachloride) in the grafting solution has little effect on the electrical resistance (Table 45) of the grafted polyethylene film over the range studied (3 to 15 weight percent). However, on crosslinking the grafted films with divinylbenzene, the films exhibited a wide range of electrical resistances (Figure 2). Analytical data for the crosslinked films, along with a crosslinked film that was prepared by grafting polyethylene film in acrylic acid free of terminator, are presented in Table 46.

When no terminator, or a low concentration of terminator, is used, the electrical resistance of the grafted and crosslinked film is lower when neutralized and washed at 97°C than when neutralized and washed at 80°C. At the higher terminator concentrations, the reverse is true.

The electrical resistance decreases on sterilization in 40 percent potassium hydroxide for films prepared with low terminator concentrations. At high terminator concentrations, the electrical resistance increases on sterilization.

Grafted and crosslinked films prepared without terminator increase in length from the dry length on sterilization. As the terminator concentration increases, there is a tendency for this increase in length to become less until a decrease in length is evident. The decrease in length is greatest at the highest terminator concentration evaluated (15 weight percent).

During the process of rolling 25-foot rolls of polyethylene film, small sections of film were cut from each roll at five-foot intervals, weighed, and replaced in their respective places in the roll. The rolls were then grafted in the usual manner. The weighed sections were neutralized and washed at 97°C and converted back to the free acid form with aqueous hydrochloric acid. They were dried in a vacuum desiccator to a constant weight. From the weight increase in the sections, the degree of grafting and the weight percent poly(acrylic acid) in the film were calculated. The effect of terminator concentration in the grafting solution on the degree of grafting is shown in Table 47 and Figure 3. As the terminator concentration increases, the degree of grafting decreases. The degree of grafting decreases rapidly with the addition of small amounts of terminator and levels off at higher concentrations.

The effect of terminator concentration on the properties of polyethylene film grafted with methacrylic acid and crosslinked with divinylbenzene (Table 48 and Figure 4) is similar to the effect obtained with acrylic acid except that the methacrylic acid-grafted film is more sensitive to terminator concentration. However, a decrease in length from the dry length is obtained in all cases on sterilization.

2. Other Chain Terminators

Other chain terminators (Table 49) that were evaluated are carbon tetrabromide, acetone, carbon disulfide, sulfur, and dodecyl mercaptan. Carbon tetrabromide is a very effective chain terminator, and a concentration as low as one weight percent in the grafting solution is excessive. Grafted films prepared with dodecyl mercaptan are not wet well by 40 percent potassium hydroxide solution.

Acetone, sulfur, and carbon disulfide appear to be good chain terminators for the acrylic acid grafting of polyethylene film. Grafted films prepared with these terminators and one prepared without terminator were crosslinked with divinylbenzene. From the limited data obtained (Table 50), carbon disulfide produced the most uniform film as determined by the electrical resistance. However, the film prepared with acetone chain terminator was almost as uniform after sterilization. All three have acceptable uniformity. The material prepared without chain terminator was also satisfactory.

It was later shown (III., Q., Ion Migration Studies) that acrylic acid-grafted polyethylene film prepared using one weight percent acetone as the terminator exhibited a low silver ion migration rate. This material was prepared using a 25 weight percent acrylic acid grafting solution and the first film supplied by the Jet Propulsion Laboratory. As a consequence, a series of acrylic acid-grafted films were prepared varying the acetone in a 15 weight percent acrylic acid-grafting solution from 1 to 10 percent and using Dow 400 polyethylene film.

The electrical resistance of the grafted films was uniformly low (Table 51) The grafted films were crosslinked with divinylbenzene, but time was not available to analyze them. Some of the films were used in the silver ion migration studies.

K. Crosslinking Studies

1. Divinylbenzene

The divinylbenzene content of crosslinked acrylic acid-grafted polyethylene film increases as the divinylbenzene content in the crosslinking solution increases. No apparent differences in the amount of divinylbenzene in the film occurred when using nitrogen or oxygen atmospheres. This was determined from ultraviolet spectra of the crosslinked films. No effort was made to determine the amount of divinylbenzene which was producing crosslinking or that which was only partially reacted.

On increasing the divinylbenzene content of the crosslinking solution from 1 to 16 volume percent (Table 52), there was no definite trend in electrical resistance or physical properties. In general, the electrical resistance was more uniform when the crosslinking was conducted in a nitrogen atmosphere.

As the concentration of divinylbenzene in the crosslinking solution increases, greater quantities of homopolymer deposit on the film. These deposits are difficult to remove.

2. Electron Beam Crosslinking Studies

Acrylic acid-grafted polyethylene film (Sample No. 120) was crosslinked in an electron beam by the Texas Nuclear Corporation, Austin, Texas. A report of their procedure appears in Appendix B. Properties of the crosslinked material are presented in Table 53 along with those of the uncrosslinked film and the film crosslinked using one volume percent divinylbenzene solution.

The electrical resistance and standard deviation of the unsterilized film did not vary excessively over the total dose range of 0 to 77 Mrad. On sterilization, the resistance changes were normal up to a total dose of 15.43 Mrad. At this total dose and higher, the electrical resistance increased on sterilization.

Increase in film thickness on wetting and on sterilization varied with total dose, and at the highest total dose (77.0 Mrad), no increase occurred. There were no correlations between total dose and dimensional changes. The elongation of the film was lowest (44%) at the maximum total dose, but it is still acceptable.

There is some question as to whether the irradiated samples should have been "annealed" at an elevated temperature prior to exposure to air. If all of the free radicals were not destroyed after 15 hours of aging at room temperature, oxidation would occur on exposure to air. It is believed that oxidation of the film tends to decrease uniformity of the film, and oxidation would probably be increased as the total dose increases.

3. <u>Other Crosslinking Studies</u>

Divinyl sulfone was used to crosslink acrylic acid-grafted polyethylene film, and the infrared spectrum of the crosslinked film indicated the presence of the sulfone group. Sterilization of this film in a stainless steel chamber produced a large amount of black material which deposited on some of the resistance specimens and could not be removed. It is believed that this deposit is partially responsible for the decrease in uniformity of the electrical resistance of the sterilized specimens (Table 54).

L. Grafting and Cografting with Other Monomers

1. Organic Solvent Systems

Several different polyethylene films have been grafted with methacrylic acid. The initial studies were conducted with the JPL polyethylene film, and the presence of carbon tetrachloride in the grafting solution resulted in grafted and crosslinked film with a higher electrical resistance than a similar film grafted in the absence of carbon tetrachloride (Table 55). On sterilization, the material prepared with carbon tetrachloride in the grafting solution increased in electrical resistance while that prepared without carbon tetrachloride decreased.

Uniformly low electrical resistance was obtained with methacrylic acid-grafted film prepared from all of the polyethylene films evaluated (Table 56).

Polyethylene film (1 mil Dow 400) can be grafted with methacrylic acid to yield materials that have uniformly low electrical resistance even with concentrations as low as 10 weight percent in the grafting solution (Table 57). Neutralizing and washing of the grafted film at 97°C yield a material of appreciably lower electrical resistance than obtained when neutralized and washed at 80°C.

Complete analytical data for the methacrylic acid-grafted films which were crosslinked with divinylbenzene are presented in Table 58. The electrical resistance values of the crosslinked material are uniformly low at all concentrations evaluated, and the tensile strength and elongation are high. A decrease in length from the dry dimension occurs on sterilization in all cases.

A large quantity of polyethylene film grafted with methacrylic acid and crosslinked with divinylbenzene was prepared, and 471 feet of this material (Table 59) was shipped to the Jet Propulsion Laboratory for evaluation.

Grafting of polyethylene film with acrylic acid-methacrylic acid mixtures does not appear to offer any advantages over grafting with the individual acids. Properties of the grafted films are presented in Table 60, and the analytical data for the grafted and crosslinked films are presented in Table 61.

It was believed that separation of the hydrophilic acrylic acid groups in acrylic acid-grafted polyethylene would improve the membrane, particularly from the standpoint of decreasing silver ion migration. As a consequence, two series of films were prepared by cografting polyethylene film with mixtures of acrylic acid and vinyltoluene. One series was prepared without chain terminator (Table 62), and the other series was prepared with chain terminator (Table 63). More vinyltoluene can be tolerated in the grafting solution when no terminator is used than when terminator is used before excessively high electrical resistance values are encountered.

Samples of the polyethylene film which were grafted with acrylic acid-vinyltoluene mixtures were crosslinked with divinylbenzene when the electrical resistance was not excessive. The analytical data for the grafted and crosslinked films are presented in Tables 64 (no terminator) and 65 (with terminator). In many cases, the hard texture of the materials prevented accurate measurement of the film thickness. The uniformity of the electrical resistance of the separator materials prepared without terminator is excellent before and after sterilization; however, crosslinking increased the resistance appreciably. The crosslinked material which was grafted in the presence of terminator exhibited poor uniformity of electrical resistance. The average electrical resistance decreased on sterilization for material prepared without terminator, while that prepared with terminator increased on sterilization.

Aconitic acid and itaconic acid were evaluated in the grafting of polyethylene film (Table 66). These acids are unsaturated polycarboxylic acids, and the effect of carboxyl groups on adjacent carbons might improve the properties of the grafted film. Because of their solubility properties, it was necessary to use methanol as the solvent in place of benzene.

With methanol solvent, little if any grafting occurred when using aconitic acid (25 wt %), itaconic acid (25 wt %), or acrylic acid (10 wt %). The lack of grafting was shown by electrical resistance measurements and infrared spectra. Grafting of the polyethylene film did occur with mixtures of acrylic acid and either aconitic acid or itaconic acid. At comparable grafting solution compositions, the aconitic acid-acrylic acid mixture produced a grafted film with an electrical resistance of 14 to 18 milliohm-inch^2 while the electrical resistance of the material grafted with the itaconic acid-acrylic acid mixture was 112 milliohm-inch^2.

When polyethylene film is grafted with N-vinyl-2-pyrrolidone, the electrical resistance of the product is greater than 3000 milliohm-inch^2. However, polyethylene film grafted with mixtures of acrylic acid and N-vinyl-2-pyrrolidone had uniformly low electrical resistance (Table 67).

Cografting of polyethylene film with mixtures of acrylic acid and styrene, divinylbenzene, or divinyl diethylene glycol diether is discussed in the section "Ion Migration Studies".

2. Aqueous Systems

Polyethylene film grafted with mixtures of acrylic acid and vinylpyridine have very low electrical resistance (Table 68). One sample was crosslinked with divinylbenzene (Table 69). These materials expand considerably more than acrylic acid-grafted films when wet with water or potassium hydroxide solution.

Attempts to graft polyethylene film with sodium vinylsulfonate or the free acid form were unsuccessful. Polyethylene film was grafted with a mixture of sodium vinylsulfonate and acrylic acid to yield a grafted film having very low and uniform electrical resistance (Table 70). The infrared spectrum of the grafted film indicates the presence of sulfonic acid groups.

Methacrylic acid behaves similarly to acrylic acid when used in aqueous systems. When ferrous sulfate is used to prevent gelation of the grafting solution (Table 71), the film remains intact in the hot water wash, while the use of potassium ferricyanide in the grafting solution (Table 72) causes the film to tear into pieces in the hot wash water. The electrical resistance values of both materials are low.

M. Potassium Content of Neutralized Grafted Films

The potassium content of acrylic acid-grafted polyethylene film which had been neutralized and washed was determined by boiling 0.2 gram of the film in 50 milliliters of 4 weight percent hydrochloric acid, rinsing the film in 50 milliliters of 4 weight percent hydrochloric acid, and then rinsing with distilled water until the combined acid and water rinsings equalled 250 milliliters. The combined washings were analyzed for potassium using a flame photometer. The moisture content of another portion of the film was determined so that the potassium content could be calculated on a dry basis.

It was found that the washing procedure used for the rolls of grafted material did not remove all of the potassium which had not reacted with the film. As a consequence, films were washed several times with distilled water and dried before analysis. This is illustrated by the data presented in Table 73.

Soaking of the grafted film in 40 percent potassium hydroxide solution followed by thorough washing with distilled water increased the potassium content of the film, and sterilization in 40 percent potassium hydroxide solution increased the potassium content of the film further.

The potassium content of the neutralized grafted film is much higher when no terminator (carbon tetrachloride) or a low concentration of terminator is used in the grafting solution than when higher concentrations are used (Table 74).

N. Polyethylene Film Studies

The optimization of the acrylic acid-grafting procedure was conducted with polyethylene film (1 mil) supplied by the Jet Propulsion Laboratory (JPL) from stock on hand. The results obtained varied throughout each 10,000-foot roll and between rolls. This was further illustrated during the scale-up studies which are discussed later in this report.

It was the general consensus that this film contained additives which interfered with the grafting reaction. On extracting some of the film with methanol, it was found that the extract contained an amide (by infrared analysis) which was probably added as a "slip" agent. No effort was made to determine the presence of other additives.

As the supply of polyethylene film was nearly depleted, films prepared from several polyethylene resins were evaluated to determine the best material for replenishing the polyethylene base stock. A series of films, free of additives, was prepared for us by the Freeport Laboratories of the Dow Chemical Company from various polyethylenes. Also, a sample of 2-mil polyethylene film (Dow 400) from a production run was obtained from Dow's Findlay, Ohio plant. This material contained 1,000 ppm of calcium carbonate which appeared to have n effect on the grafting of the film,

All of the above films were grafted with acrylic acid, and the products were much more uniform than that obtained with the JPL film (Table 75). Crosslinking of these grafted new films with divinylbenzene did not adversely affect the uniformity (Table 76). The electrical resistances, in all cases, were uniformly low before and after sterilization. All of the grafted and crosslinked films increased in length over the dry length on sterilization whether the grafted film was neutralized and washed at 80°C or at 97°C. With the grafted and crosslinked JPL film, a decrease in length usually occurs on sterilization when the grafted film is neutralized and washed at 80°C.

Because of the excellent results obtained with the 2-mil Dow 400 polyethylene film and its availability, it was recommended that 1-mil Dow 400 film be purchased to replenish the polyethylene film base stock supply. The Jet Propulsion Laboratory obtained a large quantity of this film from Dow's Fresno, California plant and supplied Southwest Research Institute with this film for subsequent work. The results of a typical grafting run with this new material are presented in Table 77.

Lowering of the acrylic acid concentration in the grafting solution to as low as 15 weight percent with most of the Dow polyethylene films evaluated resulted in satisfactory material. Dow 710M film grafted satisfactorily in 20 weight percent acrylic acid solution and in the

15 weight percent acrylic acid (Table 78). The Dow 110E film grafted satisfactorily in 20 weight percent acrylic acid but grafting was borderline at 15 weight percent in one run (Table 79).

Further studies on the effect of the concentration of acrylic acid in the grafting solution are discussed in the following sections on the study of the interlayer material and the study of scale-up procedures.

O. Study of Interlayer Material

In preparing rolls of polyethylene film for grafting in the aforementioned work, the film was backed with paper toweling and was rolled onto 1/4-inch aluminum pipe. The interlayer which is formed by the backing material is necessary to keep the polyethylene film separated and permit the grafting solution to reach all of the surface of the polyethylene film. Changes in the source of paper toweling sometimes caused a change in the results obtained in grafting of the polyethylene film.

A study made with various interlayer materials indicates that cloth is superior to paper toweling. Two of the best materials evaluated from a standpoint of effect on grafting and convenience in use are cotton cheesecloth and coarse nylon mesh. These interlayer materials permit the polyethylene film to be exposed to a greater volume of grafting solution and remove less of the acrylic acid through grafting of the interlayer material than does paper toweling. It is possible to obtain a more uniform product having lower electrical resistances using these two interlayer materials with the original JPL polyethylene film (Tables 80 and 81).

When using the original JPL polyethylene film with a cheesecloth interlayer, it is possible to lower the acrylic acid content of the grafting solution to as low as 15 weight percent and still obtain uniformly grafted film as indicated by the electrical resistance (Table 82). At 10 weight percent acrylic acid, nonuniform grafting occurred.

With the new 1-mil Dow 400 polyethylene film and a cheesecloth interlayer, satisfactory grafting is obtained at an acrylic acid concentration as low as 10 weight percent in the grafting solution. The results obtained in a series of runs varying the acrylic acid concentration from 25 to 10 weight percent are presented in Table 83. Part of the samples from this series were crosslinked with divinylbenzene by the standard procedure.

Crosslinking (Table 84) increases the electrical resistance in most cases, while neutralizing and washing at 97°C produces a material with lower electrical resistance than that neutralized and washed at 80°C. Generally, better uniformity in electrical resistance is obtained at the higher temperature. The material prepared in the lowest concentration (10 wt %) of acrylic acid increased in electrical resistance on sterilization, while at higher concentrations the electrical resistance decreased or remained unchanged on sterilization.

The film thickness and change in thickness on wetting with 40 percent potassium hydroxide solution change little, if any, on decreasing the concentration of acrylic acid in the grafting solution. However, the increase in width from the dry dimension on wetting and on sterilization tends to become smaller as the acrylic acid concentration decreases.

The same is true for changes in length, but at the lower concentrations of acrylic acid, there is a decrease in length from the dry dimension on sterilization at 135°C in 40 percent potassium hydroxide solution.

The effect of grafting solution composition on the composition of the grafted polyethylene film has been determined from a series of runs in which the acrylic acid concentrations of the grafting solution was varied from 10 to 25 weight percent. The weight ratio of acrylic acid to chain terminator (carbon tetrachloride) was maintained at 5 to 1. The procedure for determining the degree of grafting was the same as that used in the terminator study.

The degree of grafting decreases with decreasing concentration of acrylic acid in the grafting solution (Table 85 and Figure 5) and was quite uniform throughout each roll.

The electrical resistance of the grafted film was uniform throughout each roll, and, as expected, it increased with decreasing degree of grafting (Table 86 and Figure 6).

Further studies on the interlayer material are discussed in the next section on scale-up studies.

P. Scale-Up Studies and Preparation of 10,000 feet of Battery Separator

The reactor used in the scale-up work is a cylindrical chamber fabricated from aluminum sheet. The inside diameter is 12-1/2 inches, and the height is 30 inches. The wall thickness of the cylindrical portion is 1/8 inch. The reactor can be used with or without a cooling coil in the center.

The length of polyethylene film used in the initial studies was 600 feet. The procedure used for grafting was:

1. A 600-foot roll of polyethylene film backed with paper toweling was placed in the reactor, and the reactor was sealed, evacuated to about 8 mm of Hg pressure and held at this pressure for one hour to remove as much air as possible from the roll of film.

2. Nitrogen was admitted to bring the reactor pressure to atmospheric.

3. The reactor was alternately evacuated and filled with nitrogen for three additional cycles.

4. The reactor was partially evacuated to aid in the addition of the grafting solution.

5. The grafting solution (32 kilos of 25 weight percent acrylic acid solution) was added.

6. Four cycles of evacuation and repressuring with nitrogen were carried out to remove gas trapped in the roll of film.

7. The reactor was then rotated and irradiated at a dose rate of 0.022 Mrad/hr for a total dose of 1.530 Mrad for the initial runs and was then decreased to 0.012 Mrad/hr and 0.815 Mrad for the remainder of the work.

The exotherm occurring during the grafting was excessive. In the initial attempts, none of the grafted film could be recovered as the rolls were fused masses. A number of different roll configurations were tried (Table 87) at the lower dose rate and lower total dose.

All of the film from the 600-foot rolls was recovered from four runs. These were Sample Numbers 119, 152, 153, and 163. Most of Sample Number 118 was also recovered. Crosslinking of these grafted materials with divinylbenzene produced battery separator material of low electrical resistance (Tables 88-92). Other 600-foot rolls were also grafted and crosslinked, but the large roll was abandoned in favor of 100-foot rolls because of the difficulty of handling the large rolls in the laboratory. Sample Number 120, which consisted of 100-foot rolls, was much easier to process. Five of these rolls were crosslinked with divinylbenzene to produce battery separator material having electrical resistances ranging from 16 to 25 milliohm-inch^2 throughout the rolls. This material and that described in Tables 88-92 (a total of 350 feet) were shipped to the Jet Propulsion Laboratory.

As mentioned previously, the JPL polyethylene film varied throughout each roll and from roll to roll. This was further illustrated by grafting, in the same reactor, 100-foot rolls taken from two different 10,000-foot rolls of base stock (rolls Number 1 and 2). The erratic grafting as indicated by the electrical resistance is shown by the data presented in Tables 93-95. In one run (Table 93), the grafted material from roll Number 1 was more uniform than from roll Number 2. In another run (Table 94), the opposite was true.

On comparing the JPL film with Dow 110E film (Table 96), it can be seen that the Dow film is superior to the JPL film in uniformity.

All of the above samples were prepared with a paper toweling interlayer. As mentioned in the interlayer study, the use of cheesecloth in place of the paper toweling greatly improves the uniformity of the grafted film and permits lowering of the acrylic acid concentration in the grafting solution. This is further illustrated in the following temperature study in which cheesecloth interlayer was used.

The use of a cooling coil in the reactor lowered the maximum temperature obtained in the free grafting solution, but it did not lower the temperature within the rolls of polyethylene film sufficiently to form a product which could be easily recovered. Data from a run using the cooling coil with a grafting solution containing 25 weight percent acrylic acid are presented in Table 97. The electrical resistance of the grafted JPL film was uniformly low, but the film was somewhat opaque. The opaqueness frequently occurs at high temperatures.

The cooling coil was effective in lowering the temperature within the rolls when the acrylic acid concentration was lowered to 15 weight percent. At this concentration, the maximum temperature within the roll was less than $100^{\circ}F$ (Table 98). The resistance of the grafted JPL film was much higher and more erratic than that obtained at the higher temperature.

Dow 400 (1.0 mil) polyethylene film grafted under essentially the same conditions had a maximum temperature below $100^{\circ}F$ as also occurred with the JPL film. The electrical resistance of the grafted Dow 400 film was much lower and much more uniform (Table 99) than the grafted JPL film.

A comparison of the grafting of the JPL film and Dow 560E film at a higher temperature (130-150°) is shown in Table 100. Both materials had uniform electrical resistances, but the electrical resistance of grafted Dow 560E film was somewhat lower. Dow 400 film grafted under the same conditions (Table 101) behaved in a manner similar to the Dow 560E film.

The cheesecloth used in the aforementioned studies was a light weight material purchased from a local equipment supply house and was folded. To improve the ease of handling, a heavier cheesecloth (Chicopee No. 44) was obtained in roll form. Evaluation of this material showed it to be a highly satisfactory interlayer material (Table 102).

One-hundred-foot rolls of polyethylene film were grafted in two large reactors with 40 kilos of grafting solution in each reactor, and to determine the effect of neutralization and washing temperature on film properties, one roll from each reactor was cut into ten-foot lengths. Starting with the first ten feet of one roll, alternate ten-foot lengths were neutralized with five percent potassium hydroxide solution and washed with distilled water at 80°C, and the remainder were neutralized at the boiling point (97°C) of five percent potassium hydroxide solution and washed with boiling distilled water. With the other roll, the first and alternate ten-foot lengths were neutralized at the boiling point of five percent potassium hydroxide solution and washed with boiling distilled water, and the remainder processed at 80°C. The grafted and washed film was then crosslinked with divinylbenzene.

Data from the analysis of these samples are presented in Table 103. The electrical resistance of some of the specimens was higher than normal. Infrared scans across the film indicate slight differences in the spectra at the points of high film resistance. This phenomenon was encountered periodically, and it was found that these areas of high resistance are difficult to neutralize.

A comparison of the data for the two temperatures indicates:

1. The films neutralized and washed at 97°C have a lower electrical resistance before sterilization.

2. The dry film thickness and wet film thickness before sterilization are greater for the films processed at 97°C.

3. Dimensional changes (width and length) are greater for the films processed at 97°C.

Similar results were obtained with other samples discussed throughout the report.

Previous samples of grafted film were neutralized and washed in large stainless steel kettles (25 gallon) by soaking for 1 hour in each bath. A film processing machine for continuous neutralization and washing of grafted film was designed and constructed. A description of this machine and the recommended procedure for preparing battery separator material are given in Appendix C of this report. The effect of leaving the grafted film in the processing machine for prolonged periods of time on the electrical resistance of the film was slight (Table 104).

An additional 6,609 feet of battery separator material was prepared from Dow 400 (1.0 mil) polyethylene film (supplied by JPL) using a 15 weight percent acrylic acid grafting solution. The material was neutralized and washed in the film processing machine at 97°C. The conditions for preparation are given in Table 105, and the electrical resistances of the material are presented in Tables 106-112. Complete analysis of some of the material is presented in Table 113. The total battery separator material prepared in the scale-up equipment and shipped in fulfillment of the 10,000-foot request was 10,112 feet. In addition to this, a number of 30-foot rolls of acrylic acid-grafted and divinylbenzene-crosslinked material and 471 feet of methacrylic acid-grafted and divinylbenzene-crosslinked material (Table 59) were supplied.

Q. Ion Migration Studies

1. Silver Ion Migration

a. Procedure

Silver ion migration through battery separator materials was determined by a method similar to that described by T. Dirkse (Chapter 10, J. E. Cooper and Arthur Fleischer, Characteristics of Separators for Alkaline Silver Oxide-Zinc Secondary Batteries--Screening Methods). A description of the procedure is given in Appendix A. The volume of liquid on the side containing the silver oxide solution (Compartment A) was sufficiently large (200 ml) that the silver activity was essentially unchanged for the 24-hour duration of the tests. A typical change in silver activity on the downstream side of the membrane (Compartment B) with time is shown in Figure 7.

b, Effect of Acrylic Acid Concentration in Grafting Solution and the Degree of Grafting

When polyethylene film is grafted with acrylic acid, silver ion migration through the product increases as the degree of grafting increases (Table 114 and Figure 8). A similar series of acrylic acid-grafted films was prepared and then crosslinked with divinylbenzene. As the concentration of acrylic acid in the grafting solution increases, the silver ion migration through the membrane increases as expected (Table 115 and Figure 9). Neutralizing and washing of the grafted film at 97°C yields membranes with higher silver ion migration than obtained when neutralizing and washing at 80°C. Crosslinking has little if any effect on the silver ion migration when other processing conditions are the same.

c. Effect of Chain Terminator Concentration

The data obtained for the effect of the concentration of chain terminator in the grafting solution on silver ion migration are somewhat erratic (Table 116), but it appears that a high chain terminator concentration tends to lower the silver ion migration of the grafted film. For grafted films prepared with the highest terminator concentrations and neutralized and washed at 97°C, the silver ion migration is lower than for equivalent samples processed at 80°C.

d. Effect of Concentration of Divinylbenzene

It was mentioned previously that the divinylbenzene content of crosslinked acrylic acid-grafted polyethylene film increases as the divinylbenzene content of the crosslinking solution increases. The effect of the concentration of divinylbenzene content on silver ion migration was much less than anticipated (Table 117). When a nitrogen atmosphere was used in the crosslinking procedure, the silver ion migration through the product tends to decrease with increase in divinylbenzene concentration in the crosslinking solution (Figure 10). When crosslinked in air, the silver ion migration of the product was essentially the same at all concentrations with the exception of Sample No. 2-120-33.

e. Cografted Polyethylene Film

The silver ion migration through polyethylene films cografted with acrylic acid-vinyltoluene mixtures (Table 118) decreases as expected with decreasing concentration of acrylic acid in the grafting solution, and the silver pick-up is relatively low. Crosslinking of the cografted film with divinylbenzene increases the silver pick-up. There is no excessive change in silver ion migration on crosslinking with divinylbenzene except with Sample No. 392. With this material, it was difficult to obtain a good seal in the silver migration cell, and the material was softened in 5 weight percent potassium hydroxide and soaked in 40 weight percent potassium hydroxide prior to placing it in the apparatus. It is believed that this treatment is responsible for the high value obtained.

The silver ion migration through a number of cografted polyethylene films is presented in Table 119. When polyethylene film is cografted with acrylic acid and a sufficient quantity of a difunctional monomer such as divinylbenzene, the silver ion migration through the product is reduced to a low value (Sample No. S-122). However, the resistance of the cografted film is increased over the value for acrylic acid-grafted film. Cografting with acrylic acid and divinyl diethylene glycol diether mixture did not lower the silver ion migration at the concentration used (Sample No. 407G). Higher concentrations might be effective in lowering silver ion migration.

Cografting polyethylene film with acrylic acid-vinylpyridine mixtures (Sample Nos. 184GX and 240GX) in an aqueous system, resulted in products with an increased silver ion migration. When sequentially grafting polyethylene film with methanolic 2-vinylpyridine and a benzene solution of acrylic acid (Sample No. S-23), the silver ion migration rate decreases without increasing the electrical resistance.

Polyethylene films cografted with acrylic acid-methacrylic acid mixtures (Sample Nos. 343GX, 353GX, and 354GX) have silver migration rates in the range for polyethylene grafted with acrylic acid only.

The other cografting systems presented in Table 119 either show no beneficial effect or yield material with a higher silver ion migration rate than the standard acrylic acid grafted material.

f. Effect of Various Terminators

Of the various terminators, other than carbon tetrachloride, evaluated (Table 120), acetone, carbon disulfide, and sulfur show promise for the production of acrylic acid-grafted polyethylene film having reduced silver ion migration. These terminators should be studied further for grafting and cografting polyethylene film.

g. Effect of Additives in Grafting Solution

The presence of organic compounds with fused aromatic rings or acetylenic unsaturation (Table 121) in the acrylic acid grafting solution tends to lower the silver ion migration rate through the grafted product. However, they tend to increase the silver pick-up of the product. The salts evaluated (iron and zinc naphthenate) behaved similarly.

h. Effect of Polyethylene

With the exception of one polyethylene sample, all of the polyethylenes (Table 122) behave similarly in regard to silver ion migration when grafted with acrylic acid. The one sample (Dow 400 - 2 mil), because of its thickness, may have not grafted completely through the film. A low degree of grafting in the center (thickness) of the film would result in a product with a low silver ion migration rate.

Miscellaneous Studies

On another program, it was demonstrated that acrylic acid-grafted film changed in properties when treated in various ways. It was shown that heating the grafted film to about 110°C in air or nitrogen decreased its hydrophilicity and heating in water above 110°C increased its hydrophilicity. It was also found that the grafted material which was given a sequence of treatments usually behaved in a manner dependent only on the last treatment.

Because of this, some of the battery separator materials were given various treatments (Table 123). Treatment of the material at 110°C in air followed by boiling water and evaluated without drying increases the silver ion migration rate and decreases the electrical resistance while heating in air at 110°C has the opposite effect. Heating of the material in dimethylformamide, acetone or butyl alcohol at the boiling point decreases the silver migration rate with a slight or no effect on electrical resistance.

In checking the effect of the age of the neutralizing and washing baths of the film processing machine, it was found that the first and tenth (last) rolls through the machine have silver migration rates within experimental error (Sample Nos. 5-398GX and 1-399GX, Table 123).

The free acid form of acrylic acid-grafted polyethylene has a lower silver migration rate than the potassium salt prepared by neutralization in 5 percent potassium hydroxide

(Sample No. 1-389G, Table 123). As the grafted film swells more in 5 percent potassium hydroxide solution than in 40 percent potassium hydroxide solution, neutralization in the stronger solution yields a product with a lower silver ion migration rate because of the film being "tighter".

Grafting of polyethylene in aqueous acrylic acid solutions produces grafted materials having high silver ion migration (Sample Nos. 312G-313G, Table 123). This is probably due to the excessive swelling which occurs during grafting in aqueous solutions to produce a "loose" film.

Sterilization of acrylic acid-grafted polyethylene film in 40 percent potassium hydroxide produces a material which has a slightly lower silver ion migration rate than the unsterilized material (Sample No. 5-356GX, Table 123).

j. Methacrylic Acid Studies

No correlation was found for the concentration of methacrylic acid in the grafting solution and silver ion migration rate (Table 124). The silver ion migration rates are in the same range as those for acrylic acid-grafted polyethylene film.

2. Hydroxyl Ion Migration

The procedure used in determining the hydroxyl ion migration rate is that described by E. L. Harris (Chapter 9, J. E. Cooper and Arthur Fleischer, Characteristics of Separators for Alkaline Silver Oxide-Zinc Secondary Batteries--Screening Methods). Figure 11 is a typical plot of hydroxyl ion migration vs. time.

When compared on the basis of an equal wet film thickness, the hydroxyl ion migration rate through acrylic acid-grafted polyethylene film decreases as the concentration of acrylic acid in the grafting solution decreases (Table 125). In most cases, the hydroxyl ion migration rate is higher through the grafted film neutralized and washed at 97°C than through material processed at 80°C.

With methacrylic acid-grafted polyethylene film, a correlation with acid concentration was not obtained (Table 126). However, the correlation between hydroxyl ion migration and neutralization and washing temperature was present.

In general, the hydroxyl ion migration decreases with increase in terminator (carbon tetrachloride) concentration in the grafting solution (Table 127). However, the correlation is not good.

When polyethylene film is cografted with acrylic acid and vinyltoluene, either with or without terminator, the hydroxyl ion migration decreases as the acrylic acid content in the grafting solution decreases (Table 128).

3. Zincate Ion Migration

The procedure used in the determination of zincate ion migration is that described by J. J. Lander (Chapter 11, J. E. Cooper and Arthur Fleischer, Characteristics of Separators for Alkaline Silver Oxide-Zinc Secondary Batteries--Screening Methods). Figure 12 is a typical plot of zincate ion migration vs. time.

The effect of terminator (carbon tetrachloride) concentration in the acrylic acid grafting solution on the zincate ion migration through the grafted product is shown in Table 129. No correlation was obtained.

Good correlation is obtained between the acrylic acid concentration in the acrylic acid-vinyltoluene grafting solution and the zincate ion migration through the cografted polyethylene film (Table 130). The zincate ion migration rate decreased rapidly with decrease in acrylic acid concentration. The presence of carbon tetrachloride terminator in the grafting solution had only a minor effect on the zincate ion migration through the cografted product.

IV. CONCLUSIONS

The following conclusions can be made from the results of the experimental work:

1. Grafting of polyethylene film with acrylic acid followed by crosslinking with divinylbenzene yields a battery separator material superior to that produced by reversing the order of grafting and crosslinking. The product is much more uniform, and the electrical resistance is lower.

2. Results of the factorial experiment indicate that elevated temperatures within the limits studied and a nitrogen atmosphere over the grafting solution favor uniformity and low electrical resistance of the product. Dose rate and total dose within the limits studied have no effect on electrical resistance. The degree of crosslinking obtained by the procedure used has only little effect on the electrical resistance.

3. Acrylic acid-grafted polyethylene film having a low silver ion migration rate can be produced by proper choice and concentration of the chain terminator in the grafting solution. Acetone, carbon disulfide, and sulfur were the best evaluated.

4. Low silver ion migration rates can also be obtained by cografting polyethylene film with mixtures of acrylic acid and hydrocarbon-type vinyl monomers, The vinyl monomer may be mono- or difunctional. An increase in electrical resistance occurs.

5. The degree of grafting of polyethylene film with acrylic acid is proportional to the acrylic acid concentration and inversely proportional to the carbon tetrachloride concentration in the grafting solution.

6. Silver ion migration through acrylic acid-grafted polyethylene film decreases as the ratio of carbon tetrachloride to acrylic acid in the grafting solution increases, as the degree of grafting decreases, and as the concentration of divinylbenzene in the crosslinking solution (in nitrogen atmosphere) increases.

7. Polyethylene film can be cografted with acrylic acid in admixture with vinyl monomers which will not graft polyethylene when used alone.

8. An acceptable procedure for preparing large quantities of battery separator material has been developed. A device for continuously neutralizing and washing acrylic acid-grafted polyethylene film has been operated successfully.

9. Dow 400 polyethylene film is an excellent base stock for preparing battery separator material.

10. Cotton cheesecloth is an excellent material for use as an interlayer material in the grafting procedure.

11. Aqueous acrylic acid and methacrylic acid solutions can be used for grafting polyethylene film. The products generally exhibit higher silver ion migration rates and lower electrical resistances than materials prepared using organic solvents.

12. Only minor differences occur in the properties of battery separator material prepared from acrylic acid and that prepared from methacrylic acid.

FIGURES

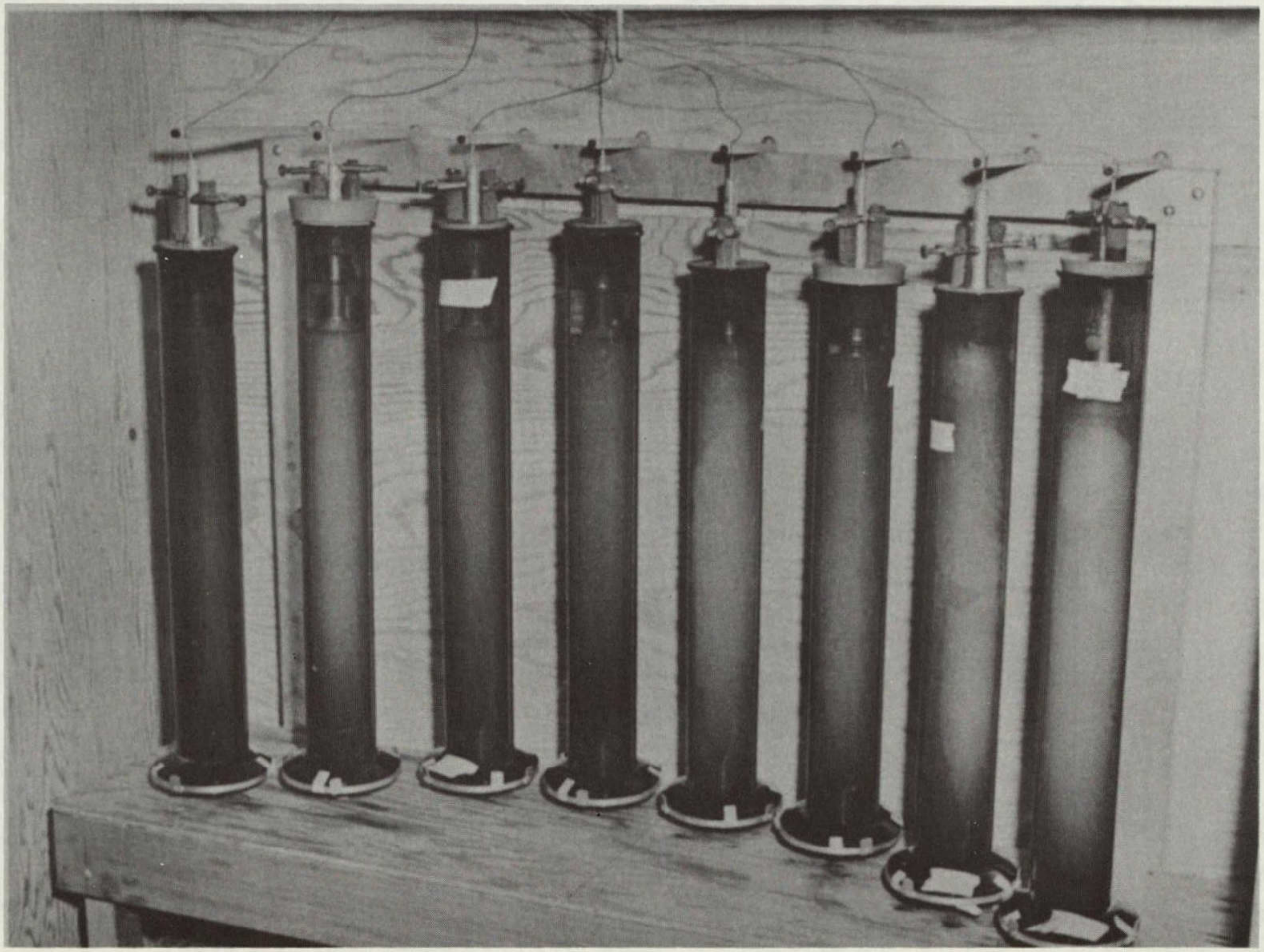

FIGURE 1. IRRADIATION TABLE

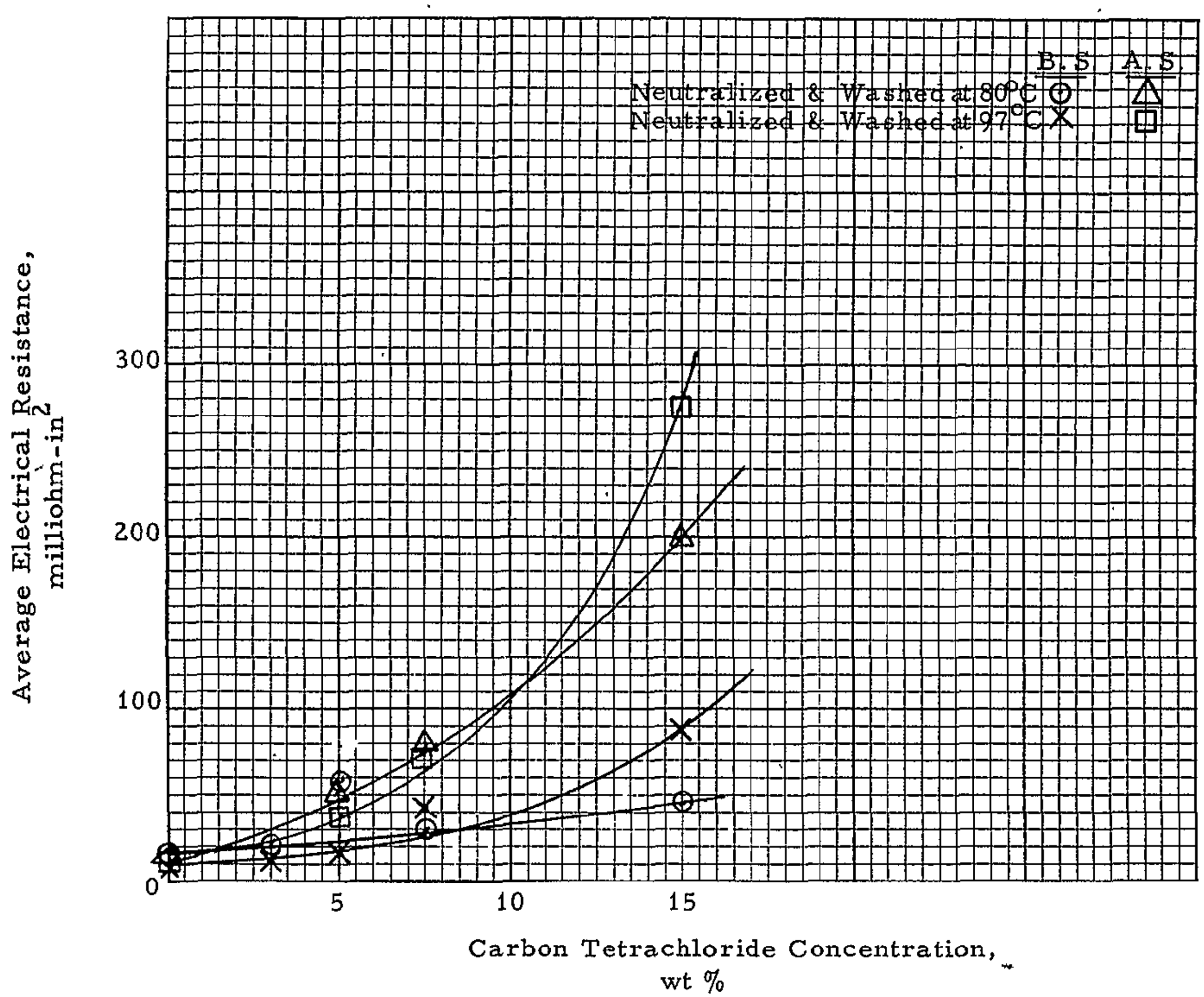

FIGURE 2. THE EFFECT OF CARBON TETRACHLORIDE IN THE ACRYLIC ACID GRAFTING SOLUTION ON ELECTRICAL RESISTANCE OF GRAFTED AND CROSSLINKED FILM

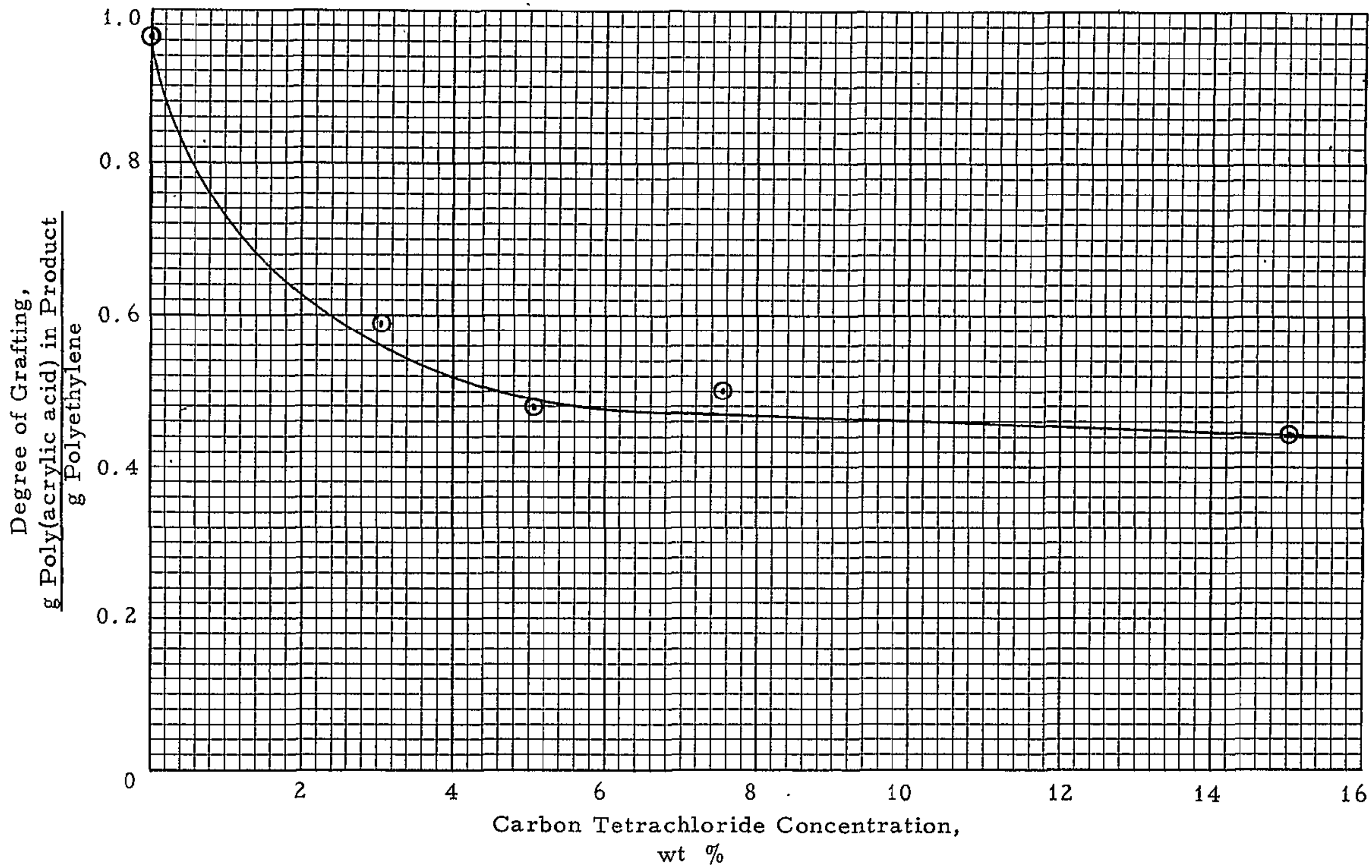

FIGURE 3. THE EFFECT OF CARBON TETRACHLORIDE CONCENTRATION IN THE ACRYLIC ACID GRAFTING SOLUTION ON THE DEGREE OF GRAFTING OF POLYETHYLENE FILM

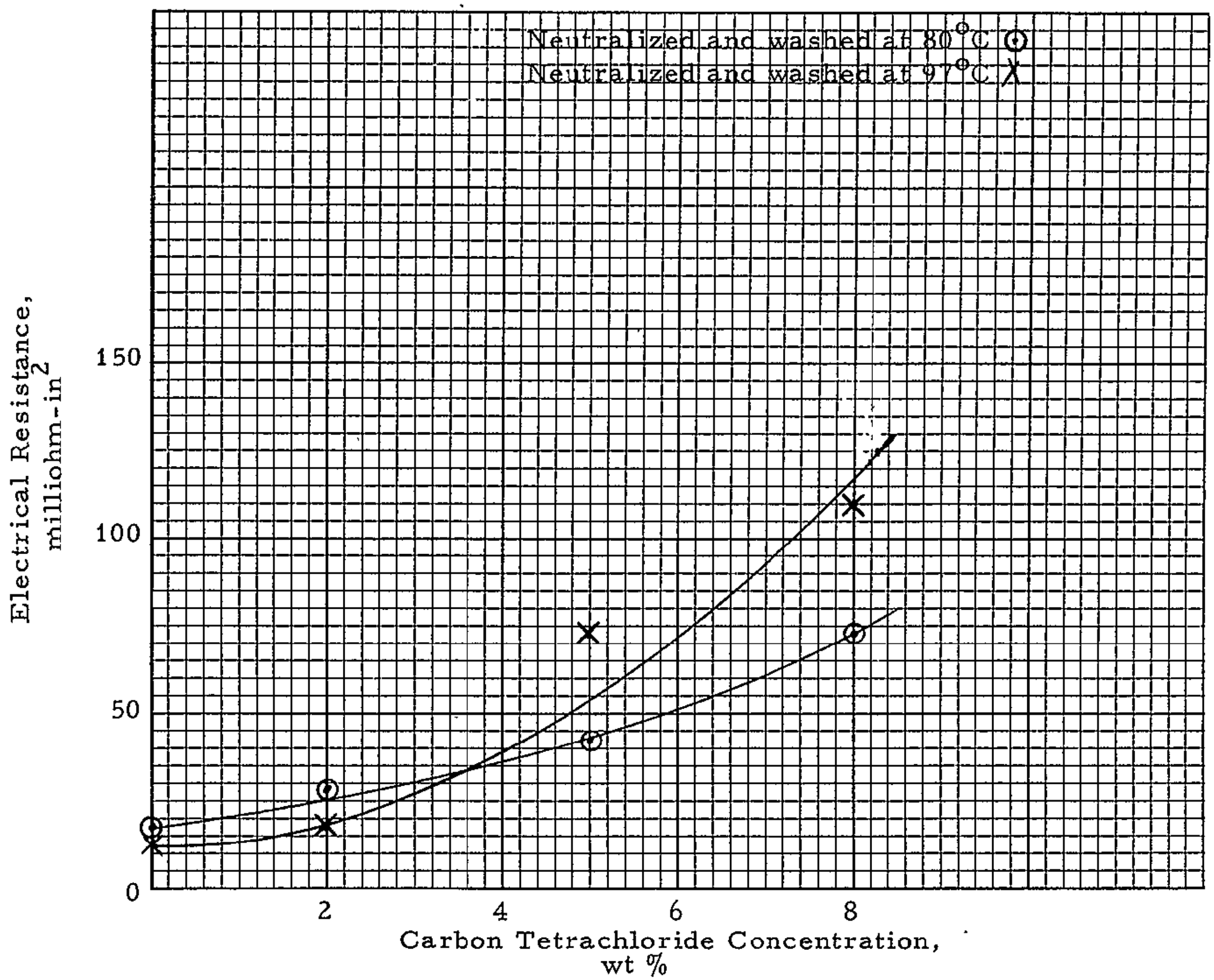

FIGURE 4. THE EFFECT OF CARBON TETRACHLORIDE IN THE METHACRYLIC ACID GRAFTING SOLUTION ON ELECTRICAL RESISTANCE OF GRAFTED AND CROSSLINKED FILM

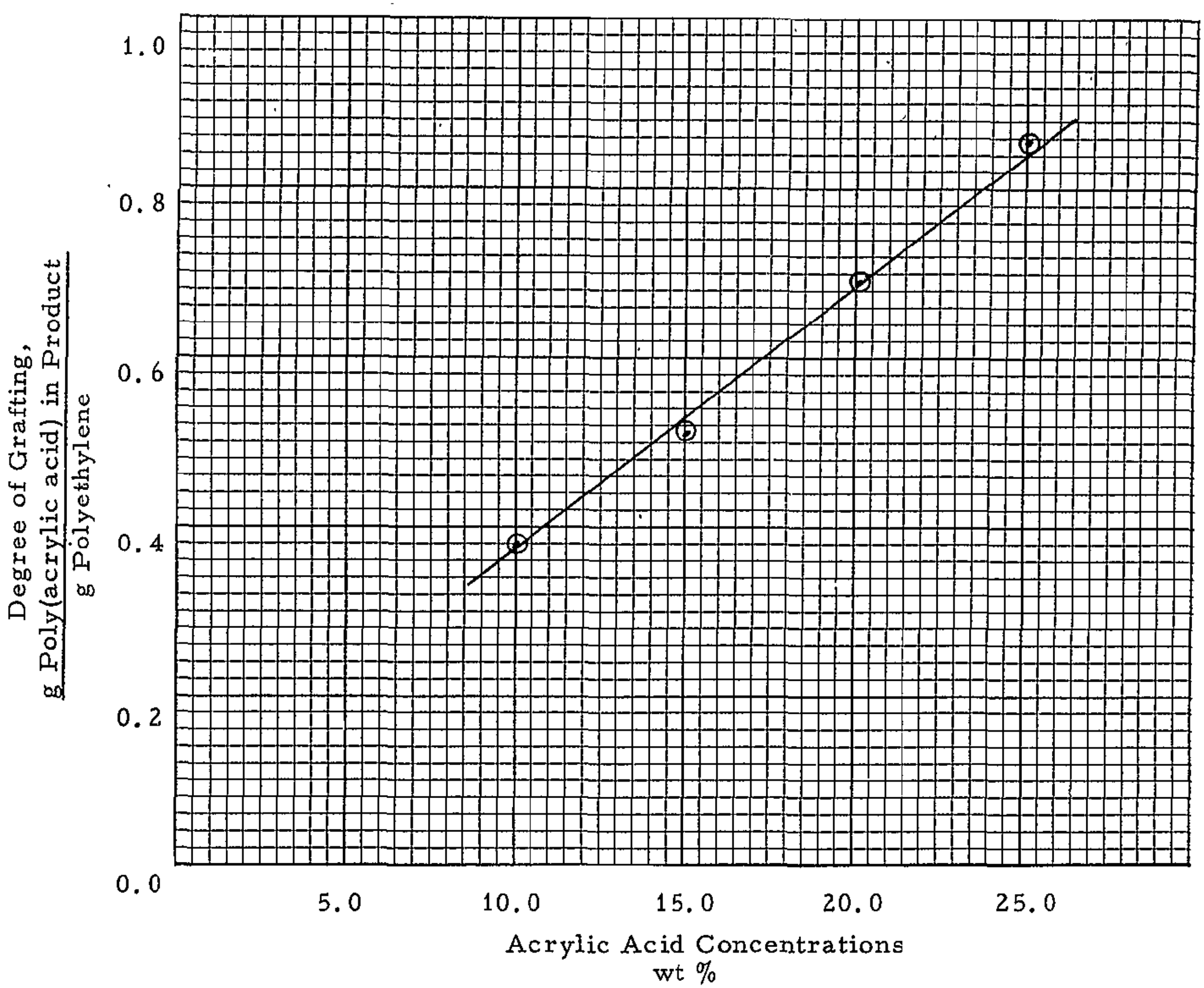

FIGURE 5. THE EFFECT OF ACRYLIC ACID CONCENTRATION IN THE GRAFTING SOLUTION ON THE DEGREE OF GRAFTING OF POLYETHYLENE FILM

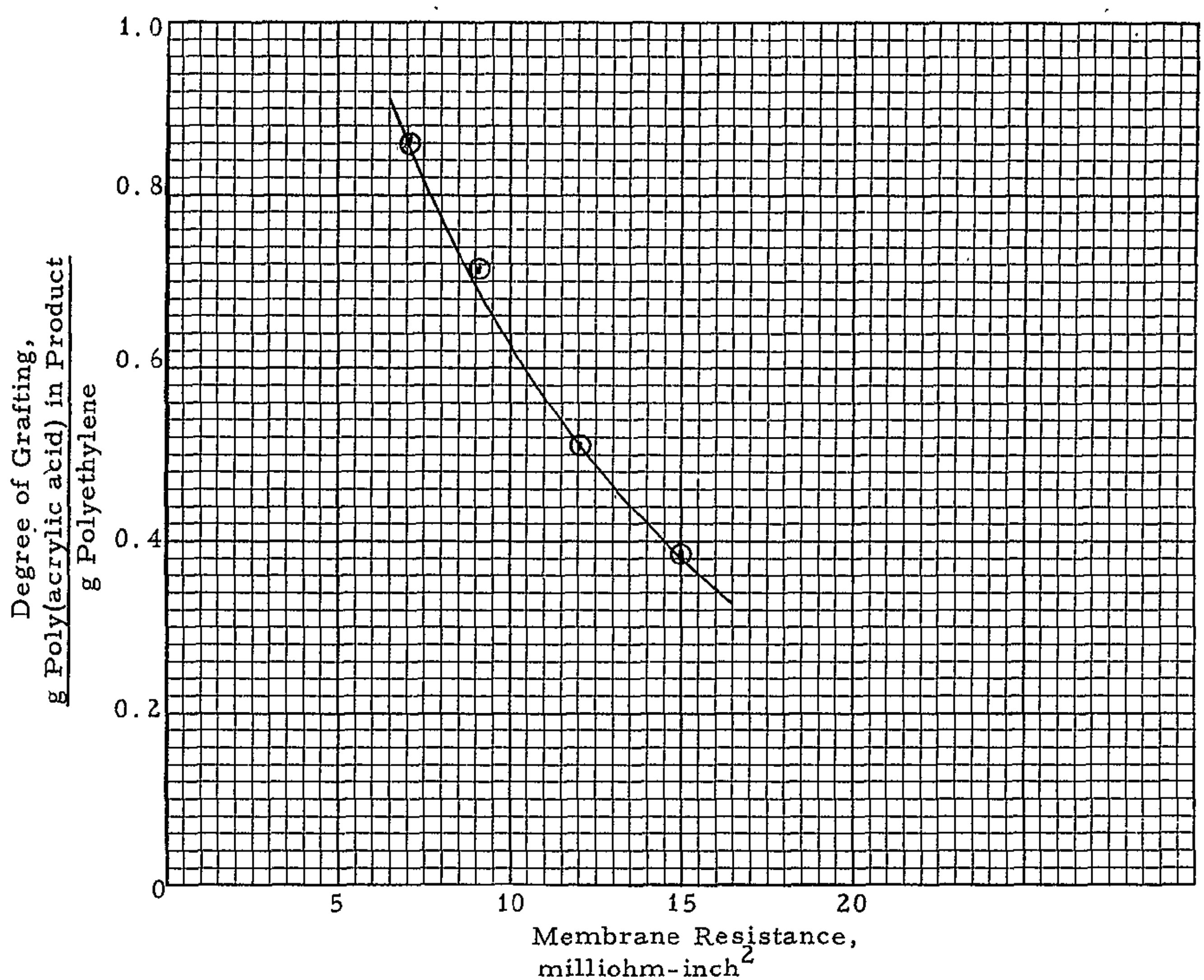

FIGURE 6. THE ELECTRICAL RESISTANCE OF ACRYLIC ACID-GRAFTED POLYETHYLENE IN 40% KOH AS A FUNCTION OF DEGREE OF GRAFTING

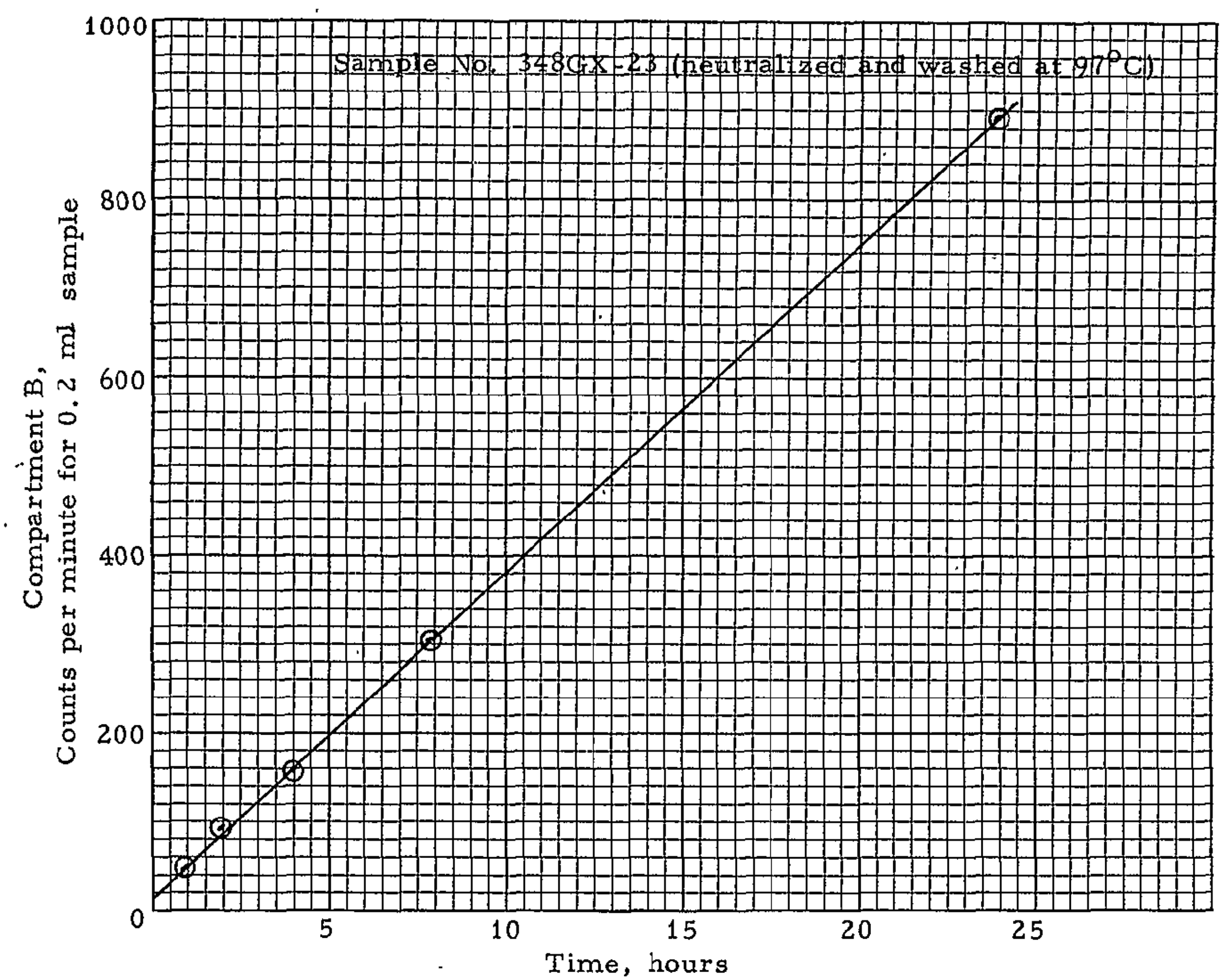

FIGURE 7. DIFFUSION OF Ag^{110m} INTO COMPARTMENT B

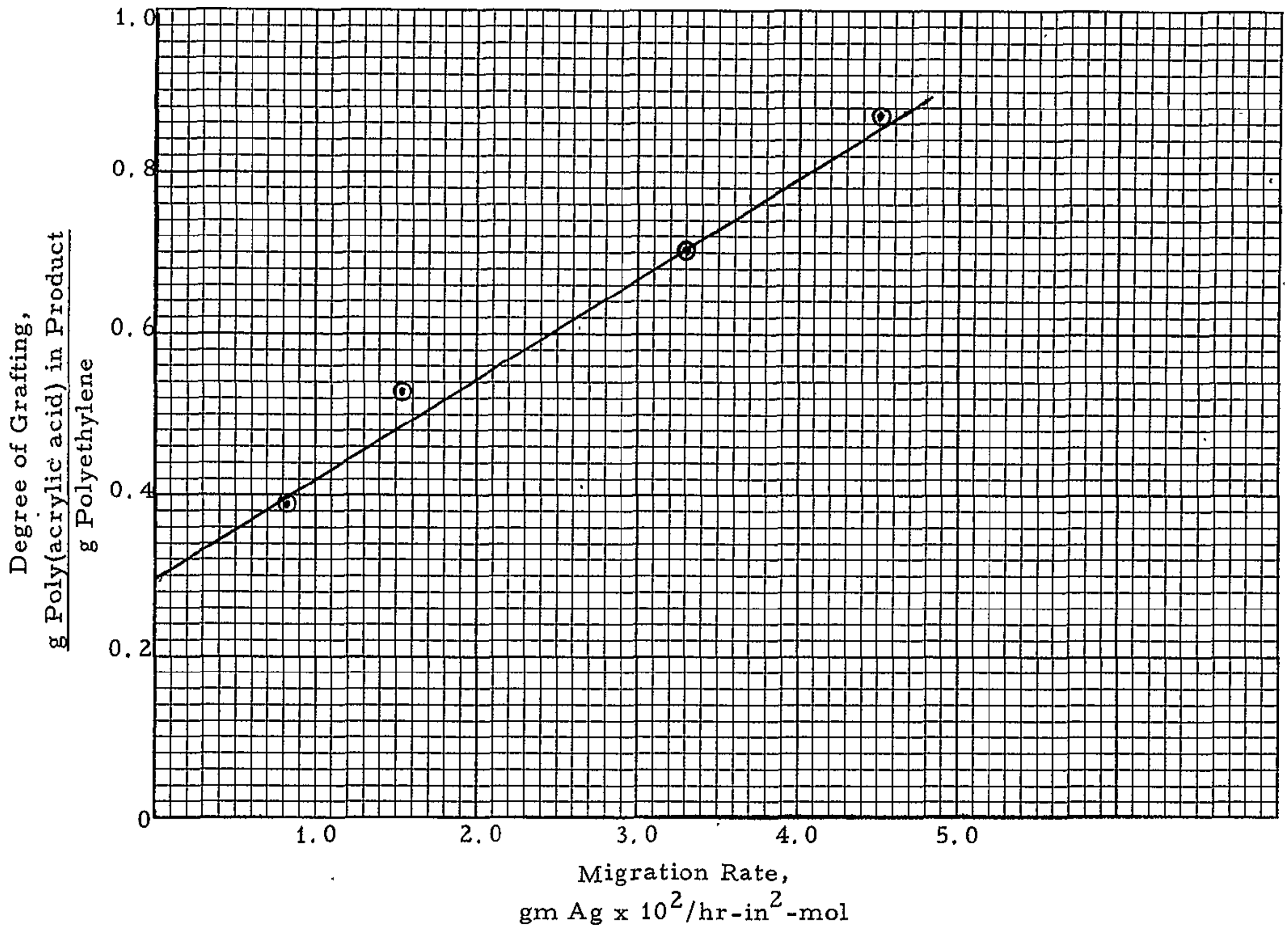

FIGURE 8. THE EFFECT OF THE DEGREE OF GRAFTING ON SILVER MIGRATION OF GRAFTED ONLY FILM

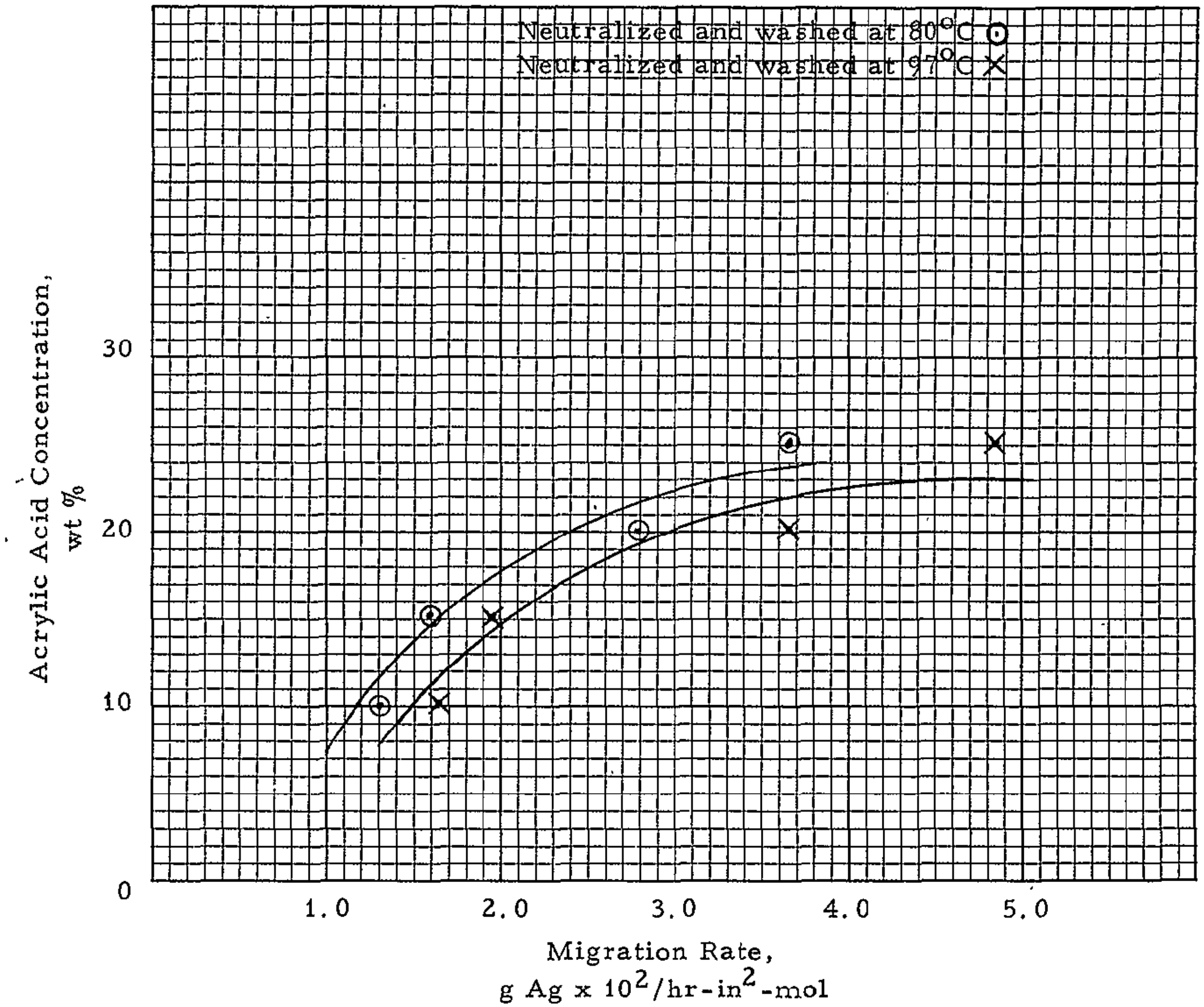

FIGURE 9. THE EFFECT OF ACRYLIC ACID CONCENTRATION IN THE GRAFTING SOLUTION ON SILVER MIGRATION OF GRAFTED AND CROSSLINKED FILM

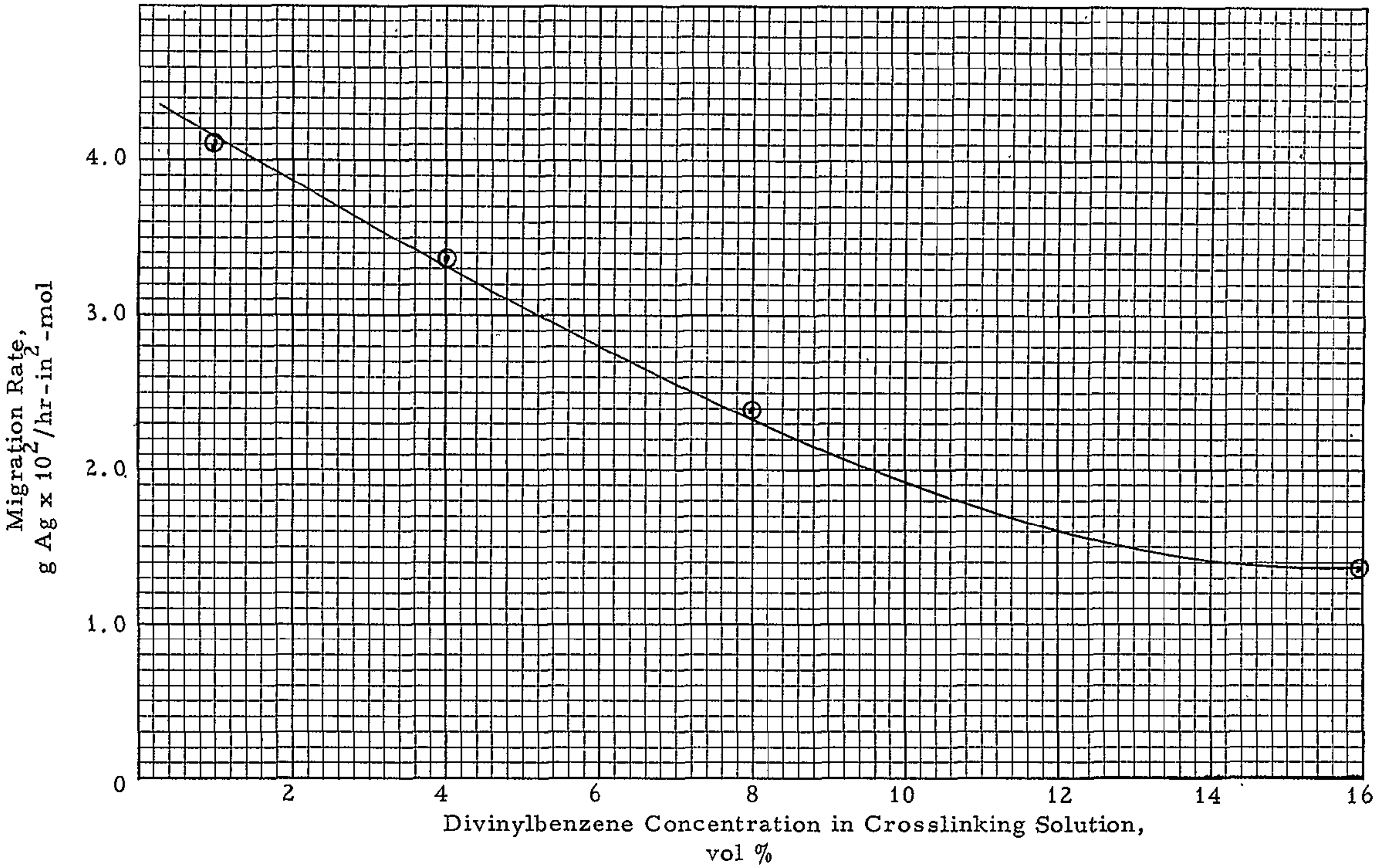

FIGURE 10. THE EFFECT OF DIVINYLBENZENE CONCENTRATION IN CROSSLINKING SOLUTION ON SILVER ION MIGRATION (NITROGEN ATMOSPHERE)

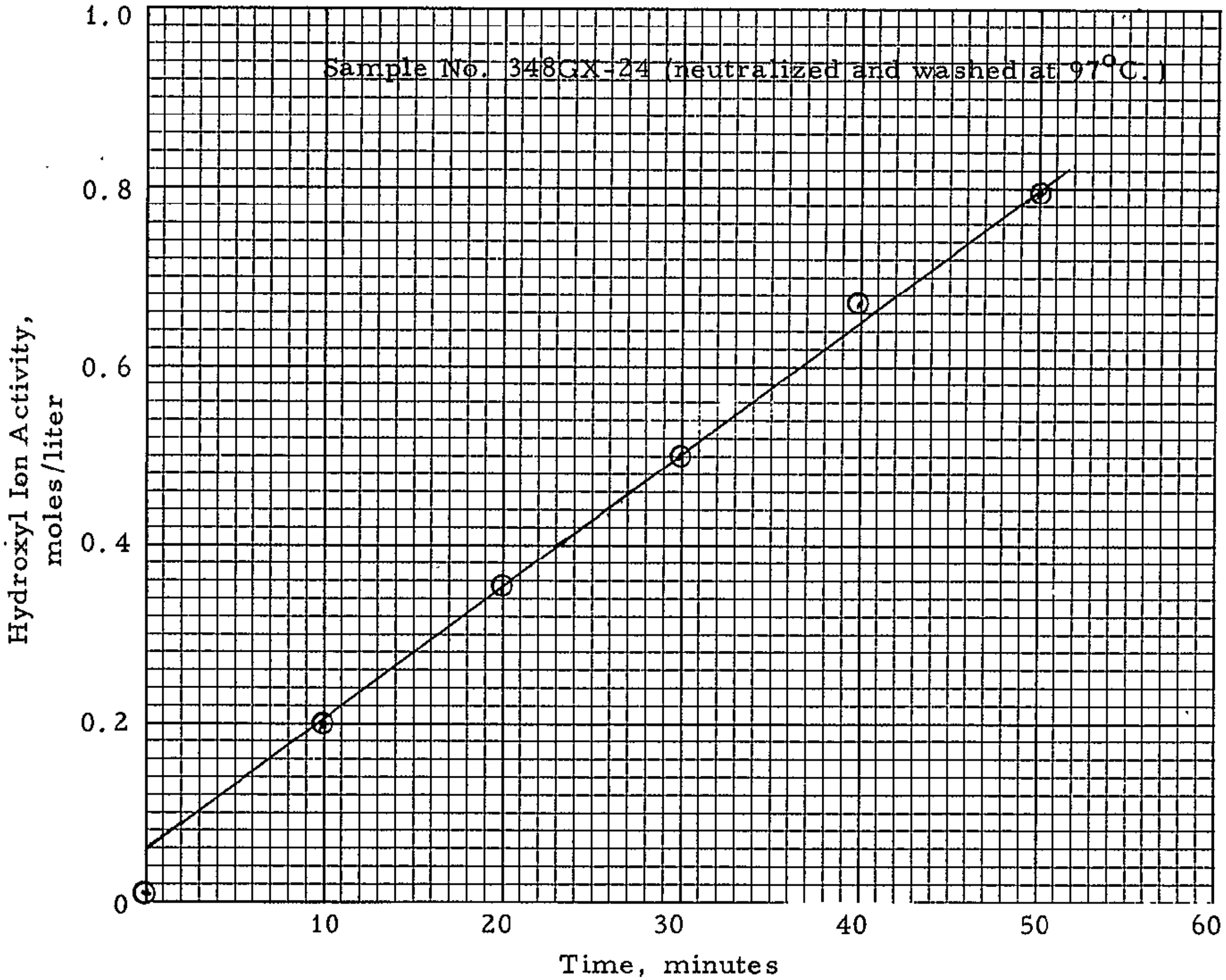

FIGURE 11. HYDROXYL ION MIGRATION THROUGH ACRYLIC ACID GRAFTED AND CROSSLINKED FILM

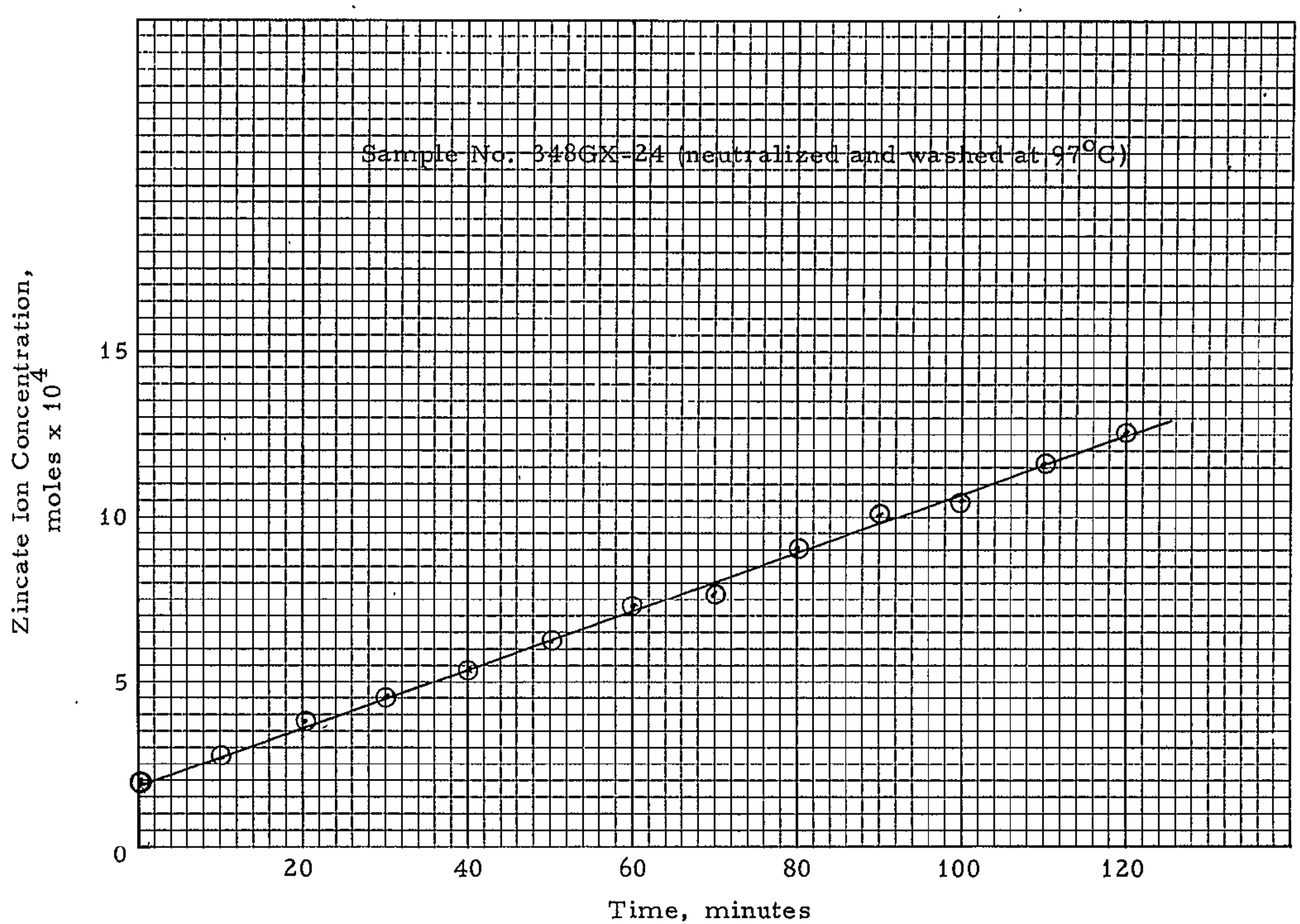

FIGURE 12. ZINCATE ION MIGRATION THROUGH ACRYLIC ACID GRAFTED AND CROSSLINKED FILM

TABLES

TABLE 1. UNIFORMITY OF DOSE RATE

(41.0 Inches from Source to Target)

Position in Reactor		Dose Rate (Mrad/hr)
1	Top	0.020
	Center	0.020
	Bottom	0.020
2	Top	0.020
	Center	0.023
	Bottom	0.024
3	Top	0.021
	Center	0.020
	Bottom	0.020
4	Top	0.020
	Center	0.020
	Bottom	0.021
5	Top	0.020
	Center	0.020
	Bottom	0.020
6	Top	0.020
	Center	0.020
	Bottom	0.021
7	Top	0.021
	Center	0.021
	Bottom	0.021
8	Top	0.020
	Center	0.021
	Bottom	0.020

TABLE 2. TENSILE STRENGTH AND ELONGATION OF CROSSLINKED LOW DENSITY POLYETHYLENE FILM

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)
Control (untreated film)	1850	>100
	1735	>100
5-1	2410	>100
	2190	>100
5-12	2320	>100
	2370	>100
5-23	2320	>100
	1945	>100
5-30	2200	>100
	2320	>100

Note: Prepared from JPL (1.0 mil) polyethylene film with paper interlayer.

(1) Determined with a Gardner Film Tester.

TABLE 3. PROPERTIES OF POLYETHYLENE FILM WHICH WAS CROSSLINKED AND THEN GRAFTED .

Sample No, Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance milliohm-inch^2
2(2)-1	2370	>100	1.0	>3000
	2370	>100	1.0	>3000
2(2)-12	1945	>100	1.3	62
	1820	>100	1.3	139
2(2)-23	1530	>100	1.4	245
	1350	99	1.4	57
2(2)-32	1028	67	1.5	841
	-	-	1.5	>3000
3-1	2730	>100	1.1	>3000
	-	-	1.1	>3000
3-6	2120	>100	1.2	2500
	-	-	1.2	>3000
3-17	2120	>100	1.1	>3000
	-	-	1.1	>3000
3-30	2020	>100	1.2	14
	-	-	1.2	214
4-1	2210	>100	1.1	>3000
4-4	-	-	1.1	1380
4-30	-	-	1.1	>3000

Note: Prepared from JPL (1.0 mil) polyethylene film with paper interlayer.

---

(1) Wet with 40% potassium hydroxide solution

(2) Inhibitor removed from acrylic acid prior to grafting.

TABLE 4. PROPERTIES OF ACRYLIC ACID-GRAFTED POLYETHYLENE FILM - GRAFTED ONLY

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance milliohm-inch^2
10-1	-	-	-	>3000
10-7	1335	95	1.4	18
	840	85	1.5	15
10-13	1690	72	1.3	28
	1395	85	1.3	12
10-24	1170	73	1.5	13
	1400	>100	1.5	2
10-35	975	>100	1.4	20
	1060	>100	1.4	16
10-35	-	-	1.4	21
			1.4	11
10-35	-	-	1.4	35
			1.4	16

Note: Prepared from JPL (1.0 mil) polyethylene film with paper interlayer.

(1) Wet with 40% potassium hydroxide solution.

TABLE 5. STERILIZATION OF GRAFTED POLYETHYLENE AT 142°C FOR 72 HOURS

Sample No. 10

Thickness, mils	
Before sterilization - dry	1.2
Before sterilization - wet	1.5
After sterilization - wet	1.6
Width, inches	
Before sterilization - dry	0.40
Before sterilization - wet	0.44
After sterilization - wet	0.46
Length, inches	
Before sterilization - dry	4.78
Before sterilization - wet	5.52
After sterilization - wet	5.44
Resistance at 25.4°C, milliohm-inch^2	
Before sterilization	17
After sterilization	9
Tensile strength, psi	
Before sterilization - wet	1040
After sterilization - wet	750
Elongation, %	
Before sterilization - wet	90
After sterilization - wet	53

TABLE 6. ACRYLIC ACID-GRAFTED POLYETHYLENE FILM GRAFTED AND CROSSLINKED

Sample No. Footage	Inhibitor in DVB	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch^2
9-1	no	2210	>100	1.1	>3000
		2120	>100	1.2	>3000
9-7	no	-	-	1.1	3
9-17	yes	1490	>100	1.4	16
		1430	>100	1.4	13
10-8	yes	1285	>100	1.5	24
		1650	>100	1.5	18
10-14	no	1610	>100	1.4	22
		1575	>100	1.5	24
10-23	no	1620	>100	1.7	11
		1750	>100	1.6	11

Note: Prepared from JPL (1.0 mil) polyethylene film with paper interlayer.

(1) Wet with 40% potassium hydroxide solution.

TABLE 7. PROPERTIES OF ACRYLIC ACID-GRAFTED POLYETHYLENE FILM - GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.021 Mrad/hr
70 Benzene	Total Dose:	1.430 Mrad
5 Carbon tetrachloride	Temperature:	77°F
	Atmosphere:	Air

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch²
20-6	1187	>100	1.5	16
	1575	>100	1.5	15
20-12	1320	>100	1.5	17
	1480	>100	1.5	22
20-21	1465	>100	1.2	26
	1465	>100	1.2	23
20-32	1360	>100	1.5	19
	1540	>100	1.5	-
23-6	1175	63	1.5	22
	1500	90	1.5	22
23-15	1305	94	1.6	31
	1390	>100	1.5	24
23-24	1375	>100	1.6	20
	1465	>100	1.5	20
23-34	1284	>100	1.5	27
	1560	>100	1.2	19
28-5	-	-	-	20
28-35	1270	>100	1.3	46
	-	-	-	49
29-6	1495	>100	1.4	15
29-24	1650	>100	1.4	46
30-11	1430	>100	1.5	15

TABLE 7. (Continued)

Sample No. Footage	Tensile Strength[1] (psi)	Elongation[1] (%)	Thickness[1] (mils)	Resistance, milliohm-inch^2
30-36	1415	>100	1.4	17
31-7	1570	80	1.4	16
31-30	1540	80	1.5	72
32-6	1260	>100	1.5	20
32-33	1440	90	1.3	23
33-7	1390	>100	1.5	18
33-35	1450	98	1.4	21
34-6	1260	90	1.4	12
34-33	1480	>100	1.3	16
35-6	845	80	1.3	27
35-35	1455	>100	1.4	17

Note
Prepared from JPL (1.0 mil) polyethylene film, Dose rate of 0.025 Mrad per hour for a total dose of 0.550 Mrad for crosslinking.

(1) Wet with 40% KOH.

TABLE 8. FACTORIAL EXPERIMENT

		Standard Dose Rate				1/2 Standard Dose Rate			
		Std. Dose		1/2 Std. Dose		Std. Dose		1/2 Std. Dose	
		O_2	N_2	O_2	N_2	O_2	N_2	O_2	N_2
Grafted Only	Temp 1	X			X		X	X	
	Temp 2		X	X		X			X
Grafted then Crosslinked	Temp 1		X	X		X			X
	Temp 2	X			X		X	X	

Standard Dose Rate = 0.021 Mrad/hr

Standard Dose = 1.430 Mrad

Temp 1 = Ambient (about 75°F)

Temp 2 = 125°F

Standard washing procedure = 1 hr. in KOH at 80°C
1 hr. in H_2O at 80°C

Properties to be determined: (1) Electrical resistance at 25°C, ± 1°C
(2) Tensile strength (wet)
(3) Elongation

Crosslinking: Dose Rate: 0.025 Mrad/hr
Total Dose: 0.550 Mrad
Temperature: Ambient (about 75°F)

TABLE 9. SAMPLE NUMBERS 36 AND 37

GRAFTED ONLY

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 77°F
Atmosphere: Air

RESULTS:

Sample No. Footage	Tensile Strength[1] (psi)	Elongation[1] (%)	Thickness[1] (mils)	Resistance, milliohm-inch^2
36-1	900	>100	1.1	>3000
	1330	>100	1.2	>3000
36-32	1690	>100	1.3	17
	1440	>100	1.3	17
37-1	1440	>100	1.3	16
	1605	>100	1.3	12
37-11	1970	>100	1.0	28
	2030	>100	1.0	40
37-20	2070	>100	1.2	48
	1695	>100	1.2	58
37-32	1130	>100	1.3	16
	1160	>100	1.4	16
Average[2]	1623	-	1.2	27
Range[2]	1130-2070	-	1.0-1.4	12-58

(1) Wet with 40% KOH.
(2) Does not include high resistance specimens.

TABLE 10. SAMPLE NUMBERS 38 AND 39

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting		Experimental Conditions for Crosslinking	
Dose Rate:	0.021 Mrad/hr	Dose Rate:	0.025 Mrad/hr
Total Dose:	1.430 Mrad	Total Dose:	0.550 Mrad
Temperature:	77°F	Temperature:	77°F
Atmosphere:	Nitrogen	Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch²
38-1	1175	87	1.5	30
	1140	75	1.4	9
38-32	1320	99	1.5	27
	1305	97	1.6	19
39-1	1350	98	1.3	11
	1255	80	1.4	11
39-9	1605	>100	1.3	38
	1510	99	1.2	29
39-17	1375	92	1.6	27
	1250	98	1.8	25
39-25	1255	>100	1.4	19
	1140	90	1.4	20
39-33	1395	93	1.5	20
	1395	98	1.5	27
Average	1319	-	1.5	22
Range	1140-1605	-	1.2-1.8	9-38

(1) Wet with 40% KOH.

TABLE 11. SAMPLE NUMBERS 44 AND 45

GRAFTED ONLY

Experimental Conditions for Grafting:

Dose Rate:	0.021 Mrad/hr
Total Dose:	0.671 Mrad
Temperature:	77°F
Atmosphere:	Nitrogen

RESULTS:

Sample No., Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch^2
44-1	755	64	1.6	12
	870	82	1.5	12
44-10	1250	>100	1.6	12
	1395	>100	1.5	21
44-19	840	60	1.5	16
	935	72	1.6	11
44-27	970	70	1.7	19
	865	40	1.6	18
44-33	1170	85	1.6	14
	1030	73	1.5	14
45-1	1260	>100	1.7	10
	1255	>100	1.5	9
45-34	1170	80	1.5	13
	1535	>100	1.5	7
Average	1093	-	1.6	13
Range	755-1535	-	1.5-1.7	9-21

(1) Wet with 40% KOH.

TABLE 12. SAMPLE NUMBERS 46 AND 47

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting:		Experimental Conditions for Crosslinking.	
Dose Rate:	0.021 Mrad/hr	Dose Rate:	0.025 Mrad/hr
Total Dose:	0.671 Mrad	Total Dose:	0.550 Mrad
Temperature:	77°F	Temperature:	77°F
Atmosphere:	Air	Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch^2
46-1	1265	>100	1.0	>3000
	1265	>100	1.0	>3000
46-34	1160	80	1.7	15
	1305	>100	1.6	18
47-1	2160	>100	1.1	>3000
	2160	>100	1.1	>3000
47-9	1430	84	1.5	36
	1700	>100	1.5	37
47-17	1035	75	1.7	13
	1340	90	1.6	19
47-25	1165	75	1.7	21
	1340	90	1.6	19
47-34	1280	88	1.5	16
	1250	80	1.5	18
Average(2)	1277	-	1.6	20
Range(2)	1035-1700	-	1.5-1.7	10-37

(1) Wet with 40% KOH.

(2) Does not include high resistance specimens.

TABLE 13. SAMPLE NUMBERS 48 AND 49

GRAFTED ONLY

Experimental Conditions for Grafting

Dose Rate: 0.0125 Mrad/hr
Total Dose: 1.700 Mrad
Temperature: 77°F
Atmosphere: Nitrogen

RESULTS:

Sample No. Footage	Tensile Strength[(1)] (psi)	Elongation[(1)] (%)	Thickness[(1)] (mils)	Resistance, milliohm-inch^2
48-1	920	>100	1.5	16
	735	83	1.5	20
48-34	1160	98	1.8	20
	1165	97	1.7	18
49-1	1465	>100	1.5	17
	1535	>100	1.5	17
49-10	1260	96	1.7	28
	1530	>100	1.4	26
49-19	1575	>100	1.5	25
	1100	95	1.8	34
49-26	1390	>100	1.5	26
	1390	>100	1.5	29
49-34	1360	97	1.5	30
	1210	75	1.5	24
Average	1271	-	1.6	24
Range	735-1575	-	1.4-1.8	16-34

(1) Wet with 40% KOH.

TABLE 14. SAMPLE NUMBERS 50 AND 51

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting		Experimental Conditions for Crosslinking	
Dose Rate:	0.0125 Mrad/hr	Dose Rate:	0.025 Mrad/hr
Total Dose:	1.700 Mrad	Total Dose:	0.550 Mrad
Temperature:	77°F	Temperature:	77°F
Atmosphere:	Air	Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength[1] (psi)	Elongation[1] (%)	Thickness[1] (mils)	Resistance milliohm-inch^2
50-1	1215	70	1.4	20
	1375	75	1.5	21
50-34	1465	87	1.5	36
	1430	92	1.5	71
51-1	1430	>100	1.5	21
	1595	>100	1.4	20
51-9	1855	>100	1.4	66
	1850	>100	1.5	65
51-17	1450	>100	1.6	29
	1580	>100	1.5	35
51-25	1535	>100	1.7	62
	1465	>100	1.5	64
51-34	1650	>100	1.4	35
	1695	>100	1.4	55
Average	1542	-	1.5	43
Range	1215-1695	-	1.4-1.7	20-71

(1) Wet with 40% KOH.

TABLE 15. SAMPLE NUMBERS 52 AND 53

GRAFTED ONLY

Experimental Conditions for Grafting

Dose Rate:	0.0125 Mrad/hr
Total Dose:	0.800 Mrad
Temperature:	77°F
Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength[1] (psi)	Elongation[1] (%)	Thickness[1] (mils)	Resistance, milliohm-inch²
52-1	1255	80	1.5	11
	855	76	1.6	13
52-13	2220	>100	1.2	133
	2160	>100	1.2	109
	-	-	-	71
	-	-	-	36
52-23	2500	>100	1.3	15
	1440	>100	1.3	28
52-34	1845	>100	1.4	29
	1570	>100	1.4	23
53-1	920	>100	1.5	13
	845	82	1.5	10
53-34	1480	>100	1.6	12
	1685	>100	1.5	12
Average	1565	-	1.4	34
Range	885-2500	-	1.2-1.6	10-133

(1) Wet with 40% KOH.

TABLE 16. SAMPLE NUMBERS 54 AND 55

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting		Experimental Conditions for Crosslinking	
Dose Rate:	0.0125 Mrad/hr	Dose Rate:	0.025 Mrad/hr
Total Dose:	0.800 Mrad	Total Dose:	0.550 Mrad
Temperature:	77°F	Temperature:	77°F
Atmosphere:	Nitrogen	Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength[(1)] (psi)	Elongation[(1)] (%)	Thickness[(1)] (mils)	Resistance, milliohm-inch^2
54-1	1540	>100	1.5	15
	1390	>100	1.5	18
54-34	1200	76	1.6	14
	1130	70	1.7	15
55-1	1295	75	1.4	15
	1395	>100	1.5	13
55-9	1470	>100	1.5	28
	1640	>100	1.3	27
55-17	1475	>100	1.3	26
	1475	>100	1.3	24
55-25	1580	>100	1.5	40
	1740	>100	1.5	38
55-34	1085	84	1.5	21
	1085	45	1.5	18
Average	1393	-	1.5	22
Range	1085-1740	-	1.3-1.7	13-40

(1) Wet with 40% KOH.

TABLE 17. SAMPLE NUMBERS 56 AND 57

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting		
Dose Rate:	0.021 Mrad/hr	Sample unavailable for crosslinking
Total Dose:	0.671 Mrad	
Temperature:	125°F	
Atmosphere:	Nitrogen	

RESULTS:

Specimens were solid rods, no samples taken.

TABLE 18. SAMPLE NUMBERS 58 AND 59

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting		Experimental Conditions for Crosslinking	
Dose Rate:	0.021 Mrad/hr	Dose Rate:	0.025 Mrad/hr
Total Dose:	1.430 Mrad	Total Dose:	0.550 Mrad
Temperature:	125°F	Temperature:	80°F
Atmosphere:	Air	Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch^2
58-1	1050	88	1.5	8
	1250	>100	1.5	8
58-9	1075	>100	2.0	10
	1310	>100	2.0	12
58-17	810	>100	2.1	4
	835	>100	2.2	5
58-25	1075	>100	2.3	9
	1050	>100	2.2	8
58-34	1150	>100	2.0	5
	1070	>100	2.0	6
59-1	1535	>100	1.4	11
	1200	>100	1.5	9
59-9	1340	>100	1.8	12
	1340	>100	1.8	8
59-17	945	>100	1.8	4
	890	>100	1.8	4
59-25	1020	>100	2.3	8
	1070	>100	2.4	7
59-34	840	>100	2.0	3
	680	>100	2.0	6
Average	1077	-	1.9	7
Range	680-1535	-	1.4-2.4	3-12

(1) Wet with 40% KOH.

TABLE 19. SAMPLE NUMBERS 60 AND 61

GRAFTED ONLY

Experimental Conditions for Grafting

Dose Rate:	0.021 Mrad/hr
Total Dose:	0.671 Mrad
Temperature:	125°F
Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch^2
60-1	1100	-	1.4	20
	940	70	1.5	19
60-9	1415	>100	1.4	34
	1240	90	1.6	34
60-17	860	94	1.7	7
	755	85	1.8	10
60-25	905	70	1.7	18
	845	70	1.8	19
60-34	700	84	2.3	10
	750	90	2.1	9
61-1	920	-	1.2	15
	905	70	1.5	18
61-9	710	65	1.7	22
	775	70	1.7	21
61-17	730	87	1.8	9
	555	70	1.7	8
61-25	770	82	2.0	7
	1050	84	2.0	7
61-35	580	85	1.8	5
	780	>100	1.8	7
Average	865	-	1.7	15
Range	555-1415	-	1.2-2.3	5-34

(1) Wet with 40% KOH.

TABLE 20. SAMPLE NUMBERS 62 AND 63

GRAFTED ONLY

Experimental Conditions for Grafting

Dose Rate: 0.021 Mrad/hr
Total Dose: 1,430 Mrad
Temperature: 125°F
Atmosphere: Nitrogen

RESULTS:

Sample No. Footage	Tensile Strength[1] (psi)	Elongation[1] (%)	Thickness[1] (mils)	Resistance, milliohm-inch^2
62[2]	1220	-	3.0	14
	400	85	3.0	13
	500	>100	3.5	11
	450	90	3.2	12
	530	>100	3.1	9
	600	70	2.8	12
	440	80	3.5	14
	-	-	-	14
	-	-	-	11
	-	-	-	9
62-34	-	-	3.6	12
	-	-	3.6	11
63	No Samples Taken			

(1) Wet with 40% KOH.
(2) Random sampling when possible.

TABLE 21. SAMPLE NUMBERS 64 AND 65

GRAFTED ONLY

Experimental Conditions for Grafting

Dose Rate:	0.0125 Mrad/hr
Total Dose:	1.700 Mrad
Temperature:	125^oF
Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch^2
64-1	865	90	1.4	7
	765	80	1.4	6
64-9	1045	>100	1.5	8
	800	90	1.7	7
64-17	880	70	1.5	15
	775	>100	1.7	15
64-25	1065	>100	1.7	9
	880	>100	1.5	8
64-34	795	80	1.8	7
	855	90	1.8	9
65-1	1100	95	1.5	11
	1050	>100	1.5	11
65-9	745	>100	1.4	16
	785	97	1.4	19
65-17	865	>100	1.4	15
	865	>100	1.4	19
65-25	880	90	1.5	11
	840	80	1.5	10
65-34	740	50	1.7	6
	630	70	1.5	5
Average	861	-	1.5	10
Range	630-1100	-	1.4-1.8	5-19

(1) Wet with 40% KOH.

TABLE 22. SAMPLE NUMBERS 66 AND 67

GRAFTED ONLY

Experimental Conditions for Grafting

Dose Rate:	0.0125 Mrad/hr
Total Dose:	0.800 Mrad
Temperature:	125°F
Atmosphere:	Nitrogen

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness (1) (mils)	Resistance, milliohm-inch^2
66-1	680	90	1.7	4
	650	90	1.7	3
66-8	470	>100	1.9	6
	300	65	2.0	5
67-1	1100	>100	1.8	3
	795	>100	1.8	2

Remarks: Specimens were very tacky, unable to obtain samples further in roll.

(1) Wet with 40% KOH.

TABLE 23. SAMPLE NUMBERS 68 AND 69

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting		Experimental Conditions for Crosslinking	
Dose Rate:	0.0125 Mrad/hr	Dose Rate:	0.025 Mrad/hr
Total Dose:	1.700 Mrad	Total Dose:	0.550 Mrad
Temperature:	125°F	Temperature:	80°F
Atmosphere:	Nitrogen	Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength[1] (psi)	Elongation[1] (%)	Thickness[1] (mils) Before crosslinking	Thickness[1] (mils) After crosslinking	Resistance, milliohm-inch² Before crosslinking	Resistance, milliohm-inch² After crosslinking
68[2]	-	-	2.4	2.1	10	4
	-	-	2.5	2.3	8	4
	-	-	2.5	2.2	9	4
	-	-	2.4	2.2	7	4
	-	-	2.1	2.2	7	5
	-	-	2.0	2.2	6	5
	-	-	2.3	2.2	6	5
	-	-	2.6	2.2	7	4
	-	-	2.6	2.0	7	5
	-	-	2.5	2.0	6	5
69	Discarded -- unable to obtain samples.					
Average	-	-	2.4	2.2	7	5
Range	-	-	2.0-2.6	2.0-2.3	6-10	4-5

(1) Wet with 40% KOH.
(2) Random sampling when possible.

TABLE 24. SAMPLE NUMBERS 70 AND 71

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting		Experimental Conditions for Crosslinking	
Dose Rate:	0.0125 Mrad/hr	Dose Rate:	0.025 Mrad/hr
Total Dose:	0.800 Mrad	Total Dose:	0.550 Mrad
Temperature:	125°F	Temperature:	80°F
Atmosphere:	Air	Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch²
70-1	1065	>100	1.2	12
	1100	>100	1.3	11
70-9	1000	>100	2.0	13
	925	>100	1.9	15
70-17	910	>100	2.0	15
	715	80	2.0	16
70-25	1045	>100	2.0	13
	1040	>100	1.9	10
70-34	980	>100	1.8	6
	980	95	1.8	8
71-1	785	65	1.4	12
	1180	84	1.4	14
71-9	990	90	2.0	22
	990	84	2.0	17
71-17	1070	>100	2.0	12
	935	90	2.0	13
71-25	915	89	1.8	9
	980	90	1.8	9
71-34	2260	>100	1.3	600
	1235	>100	1.5	21
Average(2)	987	-	1.8	13
Range(2)	715-1235	-	1.2-2.0	6-22

(1) Wet with 40% KOH.
(2) Does not include high resistance specimen.

TABLE 25. SUMMARY OF RESISTANCE VALUES FROM FACTORIAL EXPERIMENT[1]

		Grafting Dose Rate; 0.021 Mrad				Grafting Dose Rate: 0.0125 Mrad			
		Total Dose: 1.430 Mrad		Total Dose: 0.671 Mrad		Total Dose: 1.700 Mrad		Total Dose: 0.800 Mrad	
		O_2	N_2	O_2	N_2	O_2	N_2	O_2	N_2
Grafted Only	77°F	12-58 (27)			9-21 (13)		16-34 (24)	10-133 (34)	
	125°F		9-14 (12)	5-34 (15)		5-19 (10)			2-6 (4)
Grafted then Crosslinked	77°F		9-38 (22)	10-37 (20)		20-71 (43)			13-40 (22)
	125°F	3-12 (7)			No samples		4-5 (5)	6-22 (13)	

(1) Values are in milliohm-inch^2. Values in parentheses are average values, and the other values are the ranges obtained. When high resistance values occurred in the outermost few feet of the roll, these values were disregarded.

TABLE 26. GRAFTING CONDITIONS AND EXOTHERM

Sample No.	Atmosphere	Dose Rate (Mrad/hr)	Total Dose (Mrad)	Exotherm Conditions: Time to (hrs)	Exotherm Conditions: Duration of (hrs)	Exotherm Conditions: Max. Temp. (°F)
$T_1 = 77^oF$						
36	O_2	0.021	1.430	N/A		
37	O_2	0.021	1.430	N/A		
38	N_2	0.021	1.430	8.0	8	95
39	N_2	0.021	1.430	8.0	8	95
44	N_2	0.021	0.671	N/A		
45	N_2	0.021	0.671	N/A		
46	O_2	0.021	0.671	N/A		
47	O_2	0.021	0.671	N/A		
48	N_2	0.0125	1.700	N/A		
49	N_2	0.0125	1.700	N/A		
50	O_2	0.0125	1.700	N/A		
51	O_2	0.0125	1.700	N/A		
52	O_2	0.0125	0.800	N/A		
53	O_2	0.0125	0.800	N/A		
54	N_2	0.0125	0.800	N/A		
55	N_2	0.0125	0.800	N/A		
$T_2 = 125^oF$						
56	N_2	0.021	0.671	0.3	5	200
57	N_2	0.021	0.671	0.6	6	190
58	O_2	0.021	1.430	10.0	9	130
59	O_2	0.021	1.430	10.0	9	146
60	O_2	0.021	0.671	10.0	9	154
61	O_2	0.021	0.671	10.0	9	136
62	N_2	0.021	1.430	1.0	8	178
63	N_2	0.021	1.430	0.6	9	180
64	O_2	0.0125	1.700	8.0	2	135
65	O_2	0.0125	1.700	8.0	2	135
66	N_2	0.0125	0.800	0.6	4	135
67	N_2	0.0125	0.800	0.6	5	180
68	N_2	0.0125	1.700	0.6	6	175
69	N_2	0.0125	1.700	0.6	4	185
70	O_2	0.0125	0.800	N/A		
71	O_2	0.0125	0.800	N/A		

TABLE 27. EFFECT OF STERILIZATION ON RESISTANCE OF TEST PLAN SAMPLES

Sample No.	Thickness, mils		Resistance, milliohm-inch^2	
Footage	Before	After	Before	After
37-1	1.3	1.6	16	9
38-42	1.5	1.6	27	11
44-10	1.6	2.0	12	15
46-34	1.7	1.8	15	5
48-1	1.5	2.0	16	7
50-34	1.5	1.6	36	16
52-13	1.2	2.0	109	9
54-34	1.6	1.6	14	9
58-17	2.1	2.2	5	6
60-17	1.8	2.0	10	8
62(1)	3.0	3.0	12	4
64-17	1.5	1.7	15	10
66-1	1.7	1.7	4	13
68(1)	2.2	2.2	5	4
70-25	1.9	1.7	10	5

Remarks: Samples were sterilized for 64 hours at 135°C in 40% KOH.

(1) Random samples.

TABLE 28. SAMPLE NUMBERS 81 THROUGH 88

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.021 Mrad/hr
70 Benzene	Total Dose:	1.430 Mrad
5 Carbon tetrachloride	Temperature:	89°F
	Atmosphere:	Nitrogen

RESULTS:

Sample No. -Footage	Resistance, milliohm-inch^2
81-1	9
	9
81-34	12
	12
82-1	9
	9
82-34	30
	18
83-1	12
	7
83-34	9
	10
84-1	12
	7
84-34	9
	8
85-1	13
	12
85-34	8
	9
86-1	11
	10
86-34	10
	10
87-1	14
	11
87-34	5
	13
88-1	17
	16
88-34	14
	17
Average	12
Range	5-30

Exotherm Data for Grafting Samples:	82	86	88
Time to exotherm, hrs:	2	4	4
Duration of Exotherm, hrs:	18	16	16
Maximum Temperature, °F:	112	120	100

TABLE 29. SAMPLE NUMBERS 105 THROUGH 112

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.0210 Mrad/hr
70 Benzene	Total Dose:	1.430 Mrad
5 Carbon tetrachloride	Temperature:	86°F
	Atmosphere:	Nitrogen

RESULTS:

Sample No. -Footage	Resistance, milliohm-inch^2
105-1	8
	8
105-34	7
	11
106-1	8
	7
106-34	6
	8
108-1	10
	11
108-34	8
	8
109-1	10
	10
109-34	14
	14
110-1	10
	12
110-34	11
	9
111-1	7
	7
111-34	15
	11
112-1	9
	8
112-34	11
	8
Average	10
Range	6-15

Exotherm Data for Grafting	Samples: 105	108	112
Time to exotherm, hrs:	2	2	2
Duration of exotherm, hrs:	20	18	19
Maximum temperature, °F:	103	100	98

TABLE 30. SAMPLE NUMBER 73

GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.021 Mrad/hr
70 Benzene	Total Dose:	1.430 Mrad
5 Carbon tetrachloride	Temperature:	125°F
	Atmosphere:	Air

RESULTS:

Sample No. Footage	Thickness[1] (mils)	Resistance, milliohm-inch^2
73-1	1.3	18
	1.5	18
73-9	1.6	7
	1.7	6
73-17	1.7	7
	1.8	7
73-25	1.9	6
	1.8	5
73-34	1.9	4
	1.9	5
Average	1.7	8
Range	1.3-1.9	4-18

Exotherm Data for Grafting	
Time to exotherm, hrs:	5
Duration of exotherm, hrs:	19
Maximum temperature, °F:	140

(1) Wet with 40% KOH.

TABLE 31. SAMPLE NUMBER 74

GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.021 Mrad/hr
70 Benzene	Total Dose:	1.430 Mrad
5 Carbon tetrachloride	Temperature:	125°F
	Atmosphere:	Air

RESULTS:

Sample No. Footage	Thickness[1] (mils)	Resistance milliohm-inch^2
74-1	1.5	11
	1.5	12
74-9	1.5	16
	1.5	17
74-17	1.9	12
	2.0	8
74-25	1.9	8
	1.9	8
74-34	2.2	6
	2.2	5
Average	1.8	10
Range	1.5-2.2	5-17

Exotherm Data for Grafting

Time to exotherm, hrs:	5
Duration of exotherm, hrs:	19
Maximum temperature, °F:	146

(1) Wet with 40% KOH.

TABLE 32. SAMPLE NUMBER 15

CROSSLINKED AND GRAFTED

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.021 Mrad/hr
70 Benzene	Total Dose:	1.430 Mrad
5 Carbon tetrachloride	Temperature:	90°F
	Atmosphere:	Nitrogen

RESULTS:

Sample No. Footage	Tensile Strength[1] (psi)	Elongation[1] (%)	Thickness[1] (mils)	Resistance, milliohm-inch^2
15-1	1690	70	1.2	1175
	1860	92	1.1	>3000
15-11	2060	>100	-	>3000
	1915	85	-	>3000
15-23	2130	>100	-	>3000
	1955	>100	-	>3000
15-34	1600	85	1.5	150
	1745	90	1.7	10
Average	1870	-	-	-
Range	1600-2130			

Exotherm Data for Grafting

Time to exotherm, hrs:	3
Duration of exotherm, hrs:	17
Maximum temperature, °F:	111

(1) Wet with 40% KOH.

TABLE 33. SAMPLE NUMBER 12

CROSSLINKED AND GRAFTED

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0,021 Mrad/hr
70 Benzene	Total Dose:	0,671 Mrad
5 Carbon tetrachloride	Temperature:	125°F
	Atmosphere:	Air

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch^2
12-1	3100	>100	1.0	>3000
	3480	>100	1.0	>3000
12-9	3360	>100	1.0	>3000
	3240	>100	1.0	>3000
12-17	2670	>100	1.0	>3000
	2640	>100	1.1	>3000
12-25	1960	>100	1.5	>3000
	1860	>100	1.6	>3000
12-34	2050	>100	1.0	400
	2080	>100	1.0	77

Exotherm Data for Grafting

Time to exotherm, hrs:	7
Duration of exotherm, hrs:	5
Maximum temperature, °F:	130

(1) Wet with 40% KOH.

TABLE 34. SAMPLE NUMBER 13

CROSSLINKED AND GRAFTED

Grafting Solution Composition, Wt%

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.021 Mrad/hr
Total Dose:	0.671 Mrad
Temperature:	125°F
Atmosphere:	Nitrogen

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch^2
13-1	1510	>100	1.3	>3000
	1380	83	1.2	>3000
13-9	890	88	2.2	>3000
	970	85	2.2	>3000
13-17	775	>100	1.7	6
	855	>100	1.8	8
13-25	945	>100	2.1	8
	880	>100	2.0	13
13-34	1040	>100	2.0	9
	1070	>100	2.0	4

Exotherm Data for Grafting

Time to exotherm, hrs:	2
Duration of exotherm, hrs:	10
Maximum temperature, °F:	141

(1) Wet with 40% KOH.

TABLE 35. SAMPLE NUMBER 16

CROSSLINKED AND GRAFTED
(5% KOH wash before grafting)

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.021 Mrad/hr
70 Benzene	Total Dose:	1.430 Mrad
5 Carbon tetrachloride	Temperature:	$90^{o}F$
	Atmosphere:	Nitrogen

RESULTS:

Sample No. Footage	Tensile Strength(1) (psi)	Elongation(1) (%)	Thickness(1) (mils)	Resistance, milliohm-inch^2
16-1	1125	95	1.7	8
	885	50	1.8	10
16-11	1375	>100	1.6	825
	1530	>100	1.7	391
16-23	1550	90	1.4	860
	1500	>100	1.5	165
16-34	1715	>100	1.2	169
	1695	>100	1.3	117
Average	1430	-	1.5	318
Range	885-1715	-	1.2-1.8	8-860

Exotherm Data for Grafting

Time to exotherm, hrs:	3
Duration of exotherm, hrs:	17
Maximum temperature, ^{o}F:	120

(1) Wet with 40% KOH.

TABLE 36. EFFECT OF LOW DOSE RATE AND LOW TOTAL DOSE
GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.006 Mrad/hr
70 Benzene	Total Dose:	0.408 Mrad
5 Carbon tetrachloride	Temperature:	79°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Sample No.	Resistance, milliohm-inch^2 Average	Range
185	33[(1)]	14-62
186	45[(2)]	11-117
187	13[(3)]	10-18
188	20[(4)]	10-29

Note: Prepared from JPL polyethylene film with paper toweling interlayer. Outer portion of each roll did not graft. Neutralized and washed at 80°C.

(1) Average of 7 samples throughout roll.
(2) Average of 6 samples throughout roll.
(3) Average of 6 samples throughout roll.
(4) Average of 6 samples throughout roll.

TABLE 37. EFFECT OF LOW DOSE RATE AND LOW TOTAL DOSE ON GRAFTING GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.006 Mrad/hr
70 Benzene	Total Dose:	0.408 Mrad
5 Carbon tetrachloride	Temperature:	79°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

A. Electrical Properties

Sample No.	milliohm-inch²		Standard Deviation		Standard Deviation % of Average	
	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)
187	25	19	9.9	9.5	39.5	50.0
188	22	18	9.2	13.3	41.7	73.9

B. Physical Properties

Sample No.	Thickness, mils			Dimensional Change(3), % Width		Dimensional Change(3), % Length		Tensile Strength(1) (psi)	Elongation(1) (%)
	Dry	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)		
187	1.4	1.5	1.9	6.1	5.1	7.0	-1.0	1561	95
188	1.2	1.3	1.7	7.1	6.1	8.0	-0.5	1737	>100

Note

Crosslinking Solution Composition, vol %	Conditions for Crosslinking	
1 Divinylbenzene	Dose Rate:	0.025 Mrad/hr
1 Benzene	Total Dose:	0.55 Mrad
98 Methanol	Atmosphere:	Nitrogen
	Temperature:	Ambient

Resistance is average of 18 samples. Thickness is average of 24 samples. Dimensional change is average of 6 samples. Tensile strength and elongation are average of 6 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH
(3) Change from dry dimensions.

TABLE 38. EFFECT OF SOLVENTS
GRAFTED ONLY

Grafting Solution Composition, Wt %

25 Acrylic acid
75 As shown in table

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	86^oF
Atmosphere:	Nitrogen
Roll Length:	30 feet

Sample No.	Solvent (wt %)	Carbon Tetrachloride, (wt %)	Average Resistance, milliohm-inch^2	Standard Deviation	Standard Deviation % of Average
122	Xylene-70	5	85	77.4	90.6
129	Toluene-70	5	190	283	148.9
130	Toluene-75	0	most of sample not grafted		
133	Toluene-35 Methanol-35	5	21	13.3	63.5
134	Benzene-35 Methanol-35	5	7	3.0	42.8
135	Benzene-70	5	11	2.2	21.1

Note
Prepared from JPL film with paper toweling interlayer. Resistance is average of 18 samples.

TABLE 39. EFFECT OF SOLVENTS - SAMPLE NO. 134

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.012 Mrad/hr
35 Benzene	Total Dose:	0.815 Mrad
35 Methanol	Temperature:	86°F
5 Carbon tetrachloride	Atmosphere:	Nitrogen
	Roll Length:	30 feet

A. Electrical Properties

Sample No.	Avg. Resistance, milliohm-inch² B.S.[1]	Avg. Resistance, milliohm-inch² A.S.[2]	Standard Deviation B.S.[1]	Standard Deviation A.S.[2]	Standard Deviation % of Average B.S.[1]	Standard Deviation % of Average A.S.[2]
134	10	5	2.3	1.8	22.3	39.3

B. Physical Properties

Sample No.	Thickness, mils Dry	Thickness, mils B.S.[1]	Thickness, mils A.S.[2]	Dimensional Changes[3], % Width B.S.[1]	Dimensional Changes[3], % Width A.S.[2]	Dimensional Changes[3], % Length B.S.[1]	Dimensional Changes[3], % Length A.S.[2]	Tensile Strength[1] (psi)	Elongation[1] (%)
134	1.2	1.5	1.9	13.4	18.6	9.6	5.6	1390	>100

Note

Resistance is average of 18 samples. Thickness is average of 24 samples. Dimensional change is average of 6 samples. Tensile strength and elongation are average of 6 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Change from dry dimensions.

TABLE 40. EFFECT OF SOLVENTS - SAMPLE NOS. 325 AND 326

GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.012 Mrad/hr
75 Solvent	Total Dose:	0.815 Mrad
	Temperature:	75°F
	Atmosphere:	Nitrogen
	Roll Length:	5 feet

Electrical Properties

Sample No.	Solvent	Resistance, milliohm-inch^2
325 (Random sampling)	VM&P naphtha	12, 11
		11, 11
326 (Random sampling)	Cyclohexane	12, 11
		11, 11

Note

Prepared from Dow 560E (1-mil) polyethylene film with St. Regis paper interlayer. Neutralized and washed at 97°C.

TABLE 41. GRAFTING WITH AQUEOUS ACRYLIC ACID SAMPLE NUMBER 125

GRAFTED ONLY

Grafting Solution Composition, Wt %

25 Acrylic acid
75 Distilled water plus 5.2 g $FeSO_4 \cdot 7H_2O$ per 2 kilo of solution.

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	82 °F
Atmosphere:	Nitrogen
Roll Length:	30 feet

A. Electrical Properties

Sample No.	Average Resistance, milliohm-inch^2 B.S.(1)	A.S.(2)	Standard Deviation B.S.(1)	A.S.(2)	Standard Deviation % of Average B.S.(1)	A.S.(2)
125	16	3	1.6	1.6	10	53.3

B. Physical Properties

Sample No.	Thickness, mils Dry	B.S.(1)	A.S.(2)	Dimensional Changes(3), % Width B.S.(1)	Width A.S.(2)	Length B.S.(1)	Length A.S.(2)	Tensile Strength(1) (psi)	Elongation(1) (%)
125	1.2	1.4	1.6	11.0	25.0	10.0	3.5	1361	>100

Note
Resistance is average of 18 samples. Thickness is average of 18 samples. Dimensional change is average of six samples. Tensile strength and elongation are average of 6 samples.

(1) Before sterilization - wet with 40% KOH
(2) After sterilization - wet with 40% KOH.
(3) Change from dry dimensions.

TABLE 42. GRAFTING WITH AQUEOUS ACRYLIC ACID SAMPLE NOS. 312, 313, AND 346

GRAFTED ONLY

Grafting Solution Composition

As listed.

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	72°F
Atmosphere:	Nitrogen
Roll Length:	25-30 feet

Sample No.	Acrylic Acid Concentration (wt %)	Additive[1] (g)	Resistance, milliohm-inch^2
312[2]	25	$K_4Fe(CN)_6 \cdot 3H_2O$ (9.1)	4, 3, 4, 5, 5, 5
313[2]	25	$K_3Fe(CN)_6$ (7.1)	6, 5, 5, 5, 5, 6
346[3]	15	$K_3Fe(CN)_6$ (4.3)	5, 5, 6, 6, 5, 5

Note

Grafted film was neutralized and washed at 97°C.

Exotherm Data for Grafting — Samples:	312	313	346
Time to exotherm, hrs:	-	2.5	2.0
Time to maximum exotherm, hrs:	-	3.5	8.0
Maximum temperature, °F:	-	78	83

(1) Grams of additive per 2 kilograms of solution.
(2) Prepared from Dow 560E (1 mil) polyethylene film with St. Regis paper interlayer.
(3) Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

TABLE 43. EFFECT OF ADDITIVES IN GRAFTING SOLUTION
GRAFTED ONLY

Grafting Solution Composition, Wt %

25 Acrylic acid
75 Benzene
0 Carbon tetrachloride unless otherwise indicated

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	As indicated
Atmosphere:	Nitrogen unless otherwise indicated
Roll Length:	30 feet with St. Regis paper interlayer

Sample No.	Additive[1] (g)	Homopolymer Formation	Resistance Range, milliohm-inch^2	Grafting Temperature, °F	Time to Exotherm, hr	Time to Maximum Temperature, hr	Maximum Temperature, °F
140	Cerium 2-ethylhexanoate (20)	No	>3000	78	4.0	7.5	88
156	Cerium 2-ethylhexanoate (4)	No	11-13	74	6.5	11.0	79
171	Cerium 2-ethylhexanoate (0.4)	Yes	10-14	77	3.5	5.0	80
128	6% Iron naphthenate (20)	No	7-17	82	7.0	9.0	95
144	6% Iron naphthenate (20)	No	12-12	77	No exotherm		
147	6% Cobalt naphthenate (20)	No	10-13	77	No exotherm		
167	6% Cobalt naphthenate (4)	No	9-10	77	3.5	5.0	79
179[2]	6% Cobalt naphthenate (4)	No	7-16	74	No exotherm		
157	6% Nickel naphthenate (20)	No	10-16	74	6.5	11.0	79
166	6% Nickel naphthenate (4)	Slight	6-14	77	3.5	5.0	82
168	Tin 2-ethylhexanoate (4)	Yes	9-16	77	No exotherm		
268[3][4]	6% Zirconium octoate (20)	Yes	7-9	82	1.5	2.5	106

TABLE 43. (Continued)

Sample No.	Additive[1] (g)	Homopolymer Formation	Resistance Range, milliohm-inch[2]	Grafting Temperature, °F	Time to Exotherm, hr	Time to Maximum Temperature, hr	Maximum Temperature, °F
269[4]	6% Zirconium octoate (20)	Yes	9-12	82	2.5	4.0	99
274	6% Calcium naphthenate (20)	Yes	8-11	82	1.5	4.0	95
198	8% Zinc naphthenate (20)	Yes	32-66	80	7.0	8.5	98
210[3]	Acenaphthene (20)	No	13-18	87	4.0	5.0	97
220[4]	Acenaphthene (20)	Yes	7-11	81	2.0	4.0	106
211[3]	Anthraquinone (20)	Yes	15-25	87	2.0	3.5	132
280[4]	Thiokol LP-8 (20)	Yes	8-11	75	-	-	-
315[4][5]	Ethynyl cyclohexanol (4.0)	Slight	8-13	72	2.5	3.5	78
316[4][5]	Surfynol 104 (4.0)	Slight	11-13	72	0.5	1.5	121
317[3][4][5]	Surfynol 104 (4.0)	Yes	8-11	72	2.5	3.5	78

(1) Weight of additive per 2 kilograms of grafting solution.
(2) Air atmosphere during grafting.
(3) Grafting solution contained 5 wt % carbon tetrachloride.
(4) Neutralized and washed at 97°C; all others at 80°C.
(5) Prepared from Dow 560E polyethylene film. All others prepared from original JPL polyethylene film.

TABLE 44. EFFECT OF ADDITIVES IN GRAFTING SOLUTION

GRAFTED AND CROSSLINKED

A. Electrical Properties

Sample No.	Additive	Avg. Resistance, milliohm-inch^2 B.S.(1)	A.S.(2)	Standard Deviation B.S.(1)	A.S.(2)	Standard Deviation % of Average B.S.(1)	A.S.(2)
144	Iron	17	10	6.5	2.5	38.9	23.9
147	Cobalt	15	11	2.3	2.9	15.4	26.4
156	Cerium	14	13	4.9	2.7	35.1	21.4
157	Nickel	18	11	3.2	3.1	17.7	27.2
166	Nickel	14	11	2.8	2.0	20.2	17.4
167	Cobalt	14	11	1.3	1.3	9.5	11.7
168	Tin	17	12	2.6	2.3	16.0	18.7
171	Cerium	20	15	3.1	1.6	15.7	10.7
179	Cobalt	17	10	3.2	1.7	18.8	17.3

B. Physical Properties

Sample No.	Thickness, mils Dry	B.S.(1)	A.S.(2)	Dimensional Changes(3), % Width B.S.(1)	A.S.(2)	Length B.S.(1)	A.S.(2)	Tensile Strength(1) (psi)	Elongation(1) (%)
144	1.6	1.7	1.7	11.0	16.3	10.0	10.5	1044	80
147	1.4	1.5	1.6	12.2	14.2	10.5	11.5	1047	54
156	1.6	1.7	1.8	9.0	12.0	9.5	10.9	1117	85
157	1.4	1.5	1.6	6.9	11.9	7.4	7.4	1262	68
166	1.4	1.5	1.6	10.3	13.4	10.7	10.7	1145	68
167	1.4	1.6	1.6	12.4	13.4	11.1	12.1	1093	68
168	1.6	1.7	1.7	8.2	11.2	8.1	7.6	829	50
171	1.6	1.7	1.8	9.2	10.2	7.5	5.5	1287	76
179	1.5	1.6	1.7	9.0	13.0	8.4	9.9	864	47

Note For grafting conditions, see Table 43. Resistance is average of 18 samples. Thickness is average of 18 samples. Dimensional change is average of 6 samples. Tensile strength and elongation are average of 6 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Change from dry dimensions.

TABLE 45. EFFECT OF CHAIN TERMINATOR CONCENTRATION IN GRAFTING SOLUTION - SAMPLE NOS. 348-352 GRAFTED ONLY

Grafting Solution Composition, Wt %

348	349	350	351	352	
15	15	15	15	15	Acrylic acid
82	81.25	80	77.5	70	Benzene
3	3.75	5	7.5	15	Carbon tetrachloride (CCl_4)

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	75°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

Electrical Properties

	Resistance, milliohm-inch^2				
	3 wt % CCl_4	3.75 wt % CCl_4	5 wt % CCl_4	7.5 wt % CCl_4	15 wt % CCl_4
Footage	348	349	350	351	352
5(1)	18, 15	18, 16	34, 19	20, 23	22, 27
15(1)	14, 18	19, 22	19, 22	18, 27	22, 22
20(2)	9, 10	13, 12	11, 13	12, 17	15, 15
25(2)	12, 11	10, 10	11, 12	14, 11	14, 16

Exotherm Data

Time to exotherm, hr:	4.0	4.0	4.0	6.5	4.0
Time to maximum exotherm, hr:	14.0	14.0	14.0	10.0	10.0
Maximum temperature, °F:	78	79	80	80	93

Note

Prepared from Dow 410 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Neutralized and washed at 80°C.
(2) Neutralized and washed at 97°C.

TABLE 46. EFFECT OF CHAIN TERMINATOR CONCENTRATION IN GRAFTING SOLUTION(1)

Grafting Solution Composition, Wt %

368	348	350	351	352	
15	15	15	15	15	Acrylic acid
85	82	80	77.5	70	Benzene
--	3	5	7.5	15	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	75°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

A. Electrical Properties

Sample No.	Carbon Tetrachloride, wt %	Average Resistance, milliohm-inch² B.S.(2)	A.S.(3)	Standard Deviation B.S.(2)	A.S.(3)	Standard Deviation % of Average B.S.(2)	A.S.(3)
368(4)	0	15	13	1.2	1.1	8.0	8.6
368(5)	0	11	13	0.8	1.8	6.9	13.3
348(4)	3	20	16	0.6	3.1	8.0	18.0
348(5)	3	16	15	1.7	2.6	10.5	17.9
350(4)	5	38	49	6.2	15.2	16.4	30.9
350(5)	5	19	37	1.9	12.6	9.9	33.6
351(4)	7.5	30	79	5.0	40.6	16.3	51.5
351(5)	7.5	36	75	18.3	44.7	50.5	59.5
352(4)	15	45	198	6.3	71.0	14.2	35.9
352(5)	15	89	274	85.0	255.8	95.6	93.2

B. Physical Properties

Sample No.	Carbon Tetrachloride, wt %	Average Thickness, mil Dry	B.S.(2)	A.S.(3)	Average Dimensional Changes(6), % Width B.S.(2)	Width A.S.(3)	Length B.S.(2)	Length A.S.(3)	Tensile Strength(2), psi	Elongation(2), %
368(4)	0	1.4	1.6	1.6	13.3	14.3	12.5	14.5	1547	>100
368(5)	0	1.3	1.5	1.6	12.2	10.2	14.5	15.0	1428	>100
348(4)	3	1.1	1.3	1.4	7.1	8.2	5.5	0	1578	>100
348(5)	3	1.0	1.2	1.5	7.2	4.1	7.5	0.5	1500	>100
350(4)	5	1.1	1.1	1.5	6.1	6.1	4.5	-3.5	1866	>100
350(5)	5	1.0	1.1	1.4	7.1	4.1	5.5	-3.0	1650	>100
351(4)	7.5	1.1	1.3	1.5	7.1	7.1	6.0	-2.0	2100	>100
351(5)	7.5	1.0	1.2	1.5	6.1	4.0	5.0	-4.0	2296	>100
352(4)	15	1.1	1.3	1.8	5.0	6.0	4.5	-3.0	1611	>100
352(5)	15	1.0	1.2	1.6	6.1	5.1	3.5	-8.0	1990	>100

Note
Resistance is average of 12 samples. Thickness is average of 16 samples. Dimensional change is average of 4 samples. Tensile strength and elongation are average of 4 samples.

(1) Grafted with acrylic acid and crosslinked with divinylbenzene.
(2) Before sterilization (B.S.) - wet with 40% KOH.
(3) After sterilization (A.S.) - wet with 40% KOH.
(4) Neutralized and washed at 80°C.
(5) Neutralized and washed at 97°C.
(6) Change from dry dimension.

TABLE 47. EFFECT OF TERMINATOR CONCENTRATION ON DEGREE OF GRAFTING

Sample No.	Grafting Solution Composition, Wt %: Acrylic Acid	CCl_4	C_6H_6	Footage	Degree of Grafting(1)	Avg.	Poly(acrylic acid) in Product, wt %	Avg.
380	15	0	85	10	0.91		47.6	
				15	1.01	0.97	50.0	49.1
				20	0.99		49.8	
381	15	3	82	5	0.65		39.1	
				10	0.57	0.59	36.5	36.8
				15	0.53		34.5	
				20	0.60		37.2	
382	15	5	80	5	0.50		33.1	
				10	0.46	0.48	31.4	32.4
				15	0.45		31.2	
				20	0.52		34.0	
383	15	7.5	77.5	5	0.47		32.0	
				10	0.49	0.50	33.0	33.2
				15	0.52		34.1	
				20	0.51		33.6	
384	15	15	70	5	0.38		27.6	
				10	0.46	0.44	31.7	30.7
				15	0.45		31.0	
				20	0.48		32.4	

Note Prepared from Dow 400 (1.0 mil) polyethylene film with cheesecloth interlayer. Neutralized and washed at 97°C.

(1) Weight of poly(acrylic acid) in grafted polyethylene film/weight of starting polyethylene film

TABLE 48. EFFECT OF CHAIN TERMINATOR-METHACRYLIC ACID GRAFTED POLYETHYLENE FILM - SAMPLE NOS. 369-372

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

369	370	371	372	
15	15	15	15	Methacrylic acid
85	83	80	77	Benzene
--	2	5	8	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	75°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

A. Electrical Properties

Sample No.	Carbon Tetrachloride, %	Average Resistance, milliohm-inch² B.S.(1)	A.S.(2)	Standard Deviation B.S.(1)	A.S.(2)	Standard Deviation % of Average B.S.(1)	A.S.(2)
369(3)	0	18	7	1.1	1.2	5.9	16.4
369(4)	0	10	8	1.1	1.9	10.7	25.1
370(3)	2	27	22	7.0	11.6	26.1	52.6
370(4)	2	18	31	2.9	10.1	16.4	32.7
371(3)	5	42	113	24.2	43.5	57.2	38.4
371(4)	5	72	high	34.1	--	47.1	--
372(3)	8	74	high	10.6	--	14.3	--
372(4)	8	110	high	27.9	--	25.3	--

B. Physical Properties

Sample No.	Carbon Tetrachloride %	Average Thickness, mil Dry	B.S.(1)	A.S.(2)	Average Dimensional Changes(5), % Width B.S.(1)	Width A.S.(2)	Length B.S.(1)	Length A.S.(2)	Tensile Strength(1), psi	Elongation(1), %
369(3)	0	1.3	1.3	1.9	7.0	15.0	8.0	-4.0	1587	>100
369(4)	0	1.5	1.4	1.9	12.0	16.0	9.5	-3.0	1410	>100
370(3)	2	1.1	1.2	1.9	6.7	9.6	6.0	-7.5	1661	>100
370(4)	2	1.2	1.2	1.9	9.1	10.1	8.0	-7.0	1596	>100
371(3)	5	1.1	1.2	2.0	5.1	4.0	3.5	-10.0	2328	>100
371(4)	5	1.1	1.2	1.9	5.0	4.0	5.2	-8.5	2050	>100
372(3)	8	1.1	1.1	2.0	6.1	6.1	4.0	-9.5	2215	>100
372(4)	8	1.1	1.2	2.0	6.1	4.1	4.0	-9.5	2406	>100

Note
Resistance is average of 12 samples. Thickness is average of 16 samples. Dimensional change is average of 4 samples. Tensile strength and elongation are average of 4 samples. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Neutralized and washed at 80°C.
(4) Neutralized and washed at 97°C.
(5) Change from dry dimension.

TABLE 49. EFFECT OF VARIOUS CHAIN TERMINATORS GRAFTED ONLY

Grafting Solution Composition, Wt %
25 Acrylic acid
75 Benzene plus terminator

Experimental Conditions for Grafting
Dose Rate: 0.012 Mrad/hr
Total Dose: 0.815 Mrad
Atmosphere: Nitrogen
Roll Length: 30 feet

Sample No.	Terminator and Concentration (wt %)	Footage	Resistance, milliohm-inch^2	Grafting Temperature (°F)	Time to Exotherm (hrs)	Time to Maximum Temperature (hrs)	Maximum Temperature (°F)	Remarks
138	Carbon tetra- bromide, 5.0	2	>3000	78		No exotherm		IR scan indicates some grafting occurred.
		16	>3000					
		26	>3000					
148	Carbon tetra- bromide, 1.0	6	41, 41	77	7.0	9.0	86	--
		16	175, 107					
		26	220, 346					
213	Acetone, 1.0	15	11, 13	87	3.0	4.5	104	No homopolymer precipitated. Outer 12 feet did not graft.
		25	25, 24					
		30	13, 13					
214	Acetone, 2.5	6	22, 22	87	4.0	5.0	106	Outer 5 feet did not graft.
		15	35, 47					
		25	23, 29					
178	Acetone, 5.0	6	9, 15	74	3.0	4.5	90	Small amount of homopolymer precipitated. Intermittent grafting on outer two feet.
		16	15, 15					
		26	15, 13					
215	Carbon disulfide, 2.5	6	9, 9	87	3.0	4.0	102	Intermittent grafting on outer 5 feet.
		15	14, 16					
		25	15, 16					
197	Carbon disulfide, 5.0	5	9, 9	80	7.0	9.0	87	Outer three feet and top edge did not graft.
		15	11, 10					
		25	13, 12					
183	Benzene saturated with sulfur	6	15, 10	74	2.0	5.0	85	No homopolymer precipitated. Intermittent grafting on outer 2 feet.
		16	18, 18					
		26	21, 20					
199	CCl_4 (5.0) plus benzene saturated with sulfur	10	>3000, 2597	80	7.0	9.0	85	No homopolymer precipitated. Intermittent grafting on outer 10 feet.
		15	50, 45					
		25	149, 26					

TABLE 49. (Continued)

Sample No.	Terminator and Concentration (wt %)	Footage	Resistance, milliohm-inch^2	Grafting Temperature (°F)	Time to Exotherm (hrs)	Time to Maximum Temperature (hrs)	Maximum Temperature (°F)	Remarks
149	Dodecyl mercaptan 1.0	6 16	56, 61 29	77	2.0	4.0	89	Outer six feet did not graft.
139	Dodecyl mercaptan 5.0	Random	9 11	78	0.5	3.5	90	Film tore during processing. Random sampling.
150	Dodecyl mercaptan 10.0	4 14 24	>3000 >3000 >3000	77	0.5	2.5	104	KOH solution did not wet film.

Note
Prepared from JPL film with paper toweling interlayer. Neutralized and washed at 80°C.

TABLE 50. EFFECT OF VARIOUS CHAIN TERMINATORS GRAFTED AND CROSSLINKED

Crosslinking Solution Composition, vol %		Experimental Conditions for Crosslinking	
1	Divinylbenzene	Dose Rate:	0.025 Mrad/hr
1	Benzene	Total Dose:	0.55 Mrad
98	Methanol	Atmosphere:	Nitrogen
		Temperature:	Ambient

A. Electrical Properties

Sample No.	Chain Terminator and Concentration, wt %	Average Resistance, milliohm-inch^2		Standard Deviation		Standard Deviation % of Average	
		B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)
178	Acetone - 5.0	23	11	6.5	1.4	28.5	13.0
183	Benzene saturated with sulfur	22	10	5.9	3.0	27.2	29.7
197	Carbon disulfide - 5.0	17	11	1.4	1.2	8.5	10.5
123	None	13	12	2.4	1.5	18.8	12.6

B. Physical Properties

Sample No.	Thickness, mil			Dimensional Changes(3), % Width		Dimensional Changes(3), % Length		Tensile Strength(1), psi	Elongation(1), %
	Dry	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)		
178	1.4	1.5	1.6	6.1	9.2	5.5	2.0	1238	85
183	1.2	1.3	1.6	7.1	9.1	6.5	2.0	1347	72
197	1.6	1.7	1.8	5.9	8.9	6.9	6.9	1147	78
123	1.5	1.7	1.8	10.2	10.2	10.1	8.1	1161	80

Note

Prepared from JPL film with paper toweling interlayer. Neutralized and washed at 80°C. Resistance is average of 18 samples. Thickness is average of 24 samples. Dimensional change is average of 6 samples. Tensile strength and elongation are average of 6 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH
(3) Change from dry dimensions.

TABLE 51. ACETONE TERMINATOR GRAFTED ONLY

Grafting Solution Composition, Wt %		Experimental Conditions for Grafting	
Acrylic acid	15	Dose Rate:	0.012 Mrad/hr
Benzene	to 100	Total Dose:	0.815 Mrad
Acetone	as indicated	Temperature:	77°F
		Atmosphere:	Nitrogen
		Roll Length:	25 feet

Sample No.	Acetone, %	Resistance, milliohm-inch^2 5 ft	15 ft	20 ft
417	1	13	9	11
418	2	10	9	9
419	4	11	10	10
420	6	8	9	9
421	8	9	9	10
422	10	11	8	8

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.
Neutralized and washed at 97°C.

TABLE 52. DIVINYLBENZENE CROSSLINKING STUDIES

A. Electrical Properties

Sample No.	Divinylbenzene in Crosslinking Solution, vol %	Atmosphere	Average Resistance, milliohn-inch^2 B. S.(1)	Average Resistance, milliohn-inch^2 A. S.(2)	Standard Deviation B. S.(1)	Standard Deviation A. S.(2)	Standard Deviation % of Average B. S.(1)	Standard Deviation % of Average A. S.(2)
1-120-24	1	N_2	7	9	1.9	0.9	27.2	10.0
2-120-55	1	air	21	16	6.3	8.1	30.0	50.6
2-120-39	2	N_2	17	17	2.1	5.5	12.3	32.3
2-120-23	2	air	12	13	4.1	12.2	34.1	93.7
3-120-26	4	N_2	15	9	2.6	1.9	17.3	21.2
3-120-17	4	air	21	36	15.7	61.8	74.6	171.0
3-120-49	8	N_2	18	16	5.6	4.3	31.1	26.9
3-120-37	8	air	15	9	1.4	2.0	9.3	22.2
3-120-63	16	N_2	20	13	4.9	5.2	24.5	40.0

B. Physical Properties

Sample No.	Thickness, mil Dry	Thickness, mil B. S.(1)	Thickness, mil A. S.(2)	Dimensional Changes(3), % Width B. S.(1)	Dimensional Changes(3), % Width A. S.(2)	Dimensional Changes(3), % Length B. S.(1)	Dimensional Changes(3), % Length A. S.(2)	Tensile Strength, psi B. S.(1)	Elongation, % B. S.(1)
120-24	1.3	1.4	1.7	6.1	8.2	4.0	2.0	1107	64
120-55	1.4	1.5	1.8	6.0	6.0	6.0	-3.0	1634	100
120-39	1.1	1.3	1.6	8.2	8.2	4.0	0	1318	69
120-23	1.2	1.5	1.7	10.2	10.2	6.1	-2.0	1117	62
120-26	1.1	1.3	1.6	10.0	10.0	7.0	-0.5	1138	67
120-17	1.1	1.2	1.5	10.2	8.2	5.5	-1.0	1154	59
120-49	1.2	1.3	1.7	9.1	8.1	7.5	-0.5	1411	98
120-37	1.1	1.3	1.6	9..1	8.1	7.0	-0.5	1461	83
120-63	1.3	1.4	1.8	9.2	10.2	6.5	2.0	1675	95

Note
Resistance is average of six samples. Thickness is average of 7 samples. Dimensional change is single determination. Tensile strength and elongation are average of 2 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization wet with 40% KOH.
(3) Change from dry dimensions.

TABLE 53. PROPERTIES OF GRAFTED AND ELECTRON BEAM-CROSSLINKED FILM

A. Electrical Properties

Sample No.	Crosslinking Conditions	Average Resistance, milliohm-inch² B.S.[1]	Average Resistance, milliohm-inch² A.S.[2]	Standard Deviation B.S.[1]	Standard Deviation A.S.[2]	Standard Deviation % of Average B.S.[1]	Standard Deviation % of Average A.S.[2]
120-81	None	9	7	2.1	2.0	23.3	28.6
120-59	0.40 Mrad[3]	11	7	2.0	1.8	18.8	26.0
120-56	0.91 Mrad[3]	9	8	2.3	2.0	25.3	25.6
120-62	2.34 Mrad[3]	6	4	3.0	3.3	47.5	79.5
120-65	2.98 Mrad[3]	9	9	2.9	2.7	30.8	30.7
120-80	4.71 Mrad[3]	11	9	2.3	2.4	21.4	27.2
120-68	7.56 Mrad[3]	12	11	2.4	2.5	21.1	23.5
120-69	15.43 Mrad[3]	10	12	3.6	5.8	37.4	49.9
120-74	38.5 Mrad[3]	15	21	5.1	11.9	34.9	58.0
120-77	77.0 Mrad[3]	16	35	4.7	24.6	30.5	69.5
120-24	1% DVB[4] in nitrogen	7	9	1.9	0.9	27.2	10.0
120-55	1% DVB[4] in air	21	16	6.3	8.1	30.0	50.6

TABLE 53. (Continued)

B. Physical Properties

Sample No.	Thickness, mil			Dimensional Changes[5], %				Tensile Strength[1], psi		Elongation[1], %	
				Width		Length					
	Dry	B.S.[1]	A.S.[2]	B.S.[1]	A.S.[2]	B.S.[1]	A.S.[2]	B.S.[1]	A.S.[2]	B.S.[1]	A.S.[2]
120-81	1.1	1.4	1.8	4.0	0	7.9	-3.0	1065	623	95	89
120-59	1.2	1.4	1.8	8.0	6.0	7.0	-3.0	1570	823	>100	>100
120-56	1.2	1.4	1.8	8.2	6.1	8.0	-2.0	1545	1253	>100	>100
120-62	1.0	1.2	1.7	6.0	0	5.0	-1.0	1190	933	>100	>100
120-65	1.1	1.2	1.7	6.0	2.0	5.9	-4.0	1215	798	95	95
120-80	1.1	1.2	1.6	8.2	2.0	3.0	-3.0	1425	905	>100	79
120-68	1.1	1.2	1.6	4.0	-8.0	5.0	-3.0	1263	718	>100	85
120-69	1.1	1.2	1.6	2.0	-12.0	6.0	-7.0	1200	738	91	79
120-74	1.2	1.2	1.5	6.1	0	3.0	-6.0	1068	925	81	85
120-77	1.2	1.2	1.2	4.1	-4.1	5.1	-1.0	1043	763	44	44
120-24	1.3	1.4	1.7	6.1	8.2	4.0	2.0	1107	-	64	-
120-55	1.4	1.5	1.8	6.0	6.0	6.0	-3.0	1634	-	>100	-

Note

Resistance is average of six samples. Thickness is average of 7 samples. Dimensional change is single determination. Tensile strength and elongation are average of 2 samples.

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(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Dosage from electron beam.
(4) Divinylbenzene.
(5) Change from dry dimensions.

TABLE 54. GRAFTED AND CROSSLINKED FILM.

Crosslinking Solution Composition, vol %	
1	Divinyl sulfone
1	Benzene
98	Methanol

Experimental Conditions for Crosslinking	
Dose Rate:	0.025 Mrad/hr
Total Dose:	0.550 Mrad
Temperature:	71°F
Atmosphere:	Nitrogen
Roll Length:	15 feet

A. Electrical Properties

	Resistance, milliohm-inch²		Average		Standard Deviation		Standard Deviation % of Average	
Sample No.	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)
120-87	17	7						
	11	28						
	14	8						
	12	9						
	20	33						
	12	26	14	19	3.5	11.7	24.4	63.5

B. Physical Properties

	Thickness, mil			Dimensional Changes(3), % Width		Dimensional Changes(3), % Length		Tensile Strength(1), psi	Elongation(1) %
Sample No.	Dry	B S (1)	A S. (2)	B.S. (1)	A.S. (2)	B.S. (1)	A.S. (2)		
120-87	1.2	1.4	2.0	10.0	8.0	7.0	-2.0	1350	>100.
	1.2	1.3	1.6					1325	>100
	1.2	1.4	1.8						
	1.3	1.5	1.9						
	1.4	1.4	2.0						
	1.4	1.5	1.6						
	1.3	1.5	1.8						
Average	1.3 .	1.5	1.8					1338	>100

(1) Before sterilization - wet with 40% KOH
(2) After sterilization - wet with 40% KOH
(3) Change from dry dimensions.

TABLE 55. METHACRYLIC ACID GRAFTED POLYETHYLENE FILM GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %		
137	146	
25	25	Methacrylic acid
70	75	Benzene
5	-	Carbon tetrachloride

Experimental Conditions for Grafting	
Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	77°F
Atmosphere:	Nitrogen
Roll Length:	30 feet

A. Electrical Properties

Sample No.	Average Resistance, milliohm-inch² B.S.(1)	A.S.(2)	Standard Deviation B.S.(1)	A.S.(2)	Standard Deviation % of Average B.S.(1)	A.S.(2)
137	59	69	20.0	65.7	34.1	95.4
146	26	7	4.1	2.8	16.0	39.2

B. Physical Properties

Sample No.	Thickness, mils Dry	B.S.(1)	A.S.(2)	Dimensional Changes(3), % Width B.S.(1)	Width A.S.(2)	Length B.S.(1)	Length A.S.(2)	Tensile Strength(1) (psi)	Elongation(1 (%)
137	1.2	1.3	2.2	8.2	4.1	4.0	-9.4	2237	>100
146	2.0	1.5	2.1	7.0	14.0	6.0	-4.5	1992	>100

Note

Prepared from JPL polyethylene film with paper toweling interlayer. Neutralized and washed at 80°C. Resistance is average of 18 samples. Thickness is average of 16 samples. Dimensional change is average of 6 samples. Tensile strength and elongation are average of 6 samples.

(1) Before sterilization - wet with 40% KOH.

(2) After sterilization - wet with 40% KOH.

(3) Change from dry dimension.

TABLE 56. METHACRYLIC ACID GRAFTED POLYETHYLENE FILM GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Methacrylic acid	Dose Rate:	0.012 Mrad/hr
75 Benzene	Total Dose:	0.815 Mrad
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Sample No.	Resistance Range, milliohm-inch^2	Grafting Temperature ^oF	Time to Exotherm, (hrs)	Time to Maximum Exotherm, (hrs)	Maximum Temperature ^oF
226[(1)]	6-7	81	2.0	5.0	89
227[(2)]	3-6	81	2.0	5.0	89
291[(3)]	6-9	72	2.0	3.5	77
292[(3)]	10-13	72	2.0	3.5	77
295[(4)]	8-12	72	No exotherm		
296[(4)]	8-12	72	No exotherm		

Note
Neutralized and washed at 97^oC.

(1) Prepared from JPL polyethylene film with St. Regis paper interlayer.
(2) Prepared from Dow 710M (1 mil) polyethylene film with St. Regis paper interlayer.
(3) Prepared from Dow 510M (1 mil) polyethylene film with St. Regis paper interlayer.
(4) Prepared from Dow 560E (1 mil) polyethylene film with St. Regis paper interlayer.

TABLE 57. EFFECT OF METHACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION - SAMPLE NOS. 339-342 GRAFTED ONLY

Grafting Solution Composition, Wt %

339	340	341	342	
25	20	15	10	Methacrylic acid (MA)
75	80	85	90	Benzene

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	72°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

Electrical Properties

	Resistance, milliohm-inch^2			
Footage	25 wt % MA 339	20 wt % MA 340	15 wt % MA 341	10 wt % MA 342
5[1]	13, 14	14, 12	14, 16	19, 18
14[1]	13, 14	12, 13	24, 26	24, 23
20[2]	8, 8	10, 10	14, 13	11, 10
25[2]	8, 7	10, 10	12, 9	14, 11

Exotherm Data

Time to exotherm, hr:	--	2.0	2.0	2.0
Time to max. exotherm, hr:	--	4.0	4.0	4.0
Maximum temperature, °F:	--	76	76	76

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

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(1) Neutralized and washed at 80°C.
(2) Neutralized and washed at 97°C.

TABLE 58. EFFECT OF METHACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION - SAMPLE NOS. 339-342 GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

339	340	341	342	
25	20	15	10	Methacrylic acid
75	80	85	90	Benzene

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	72°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

A. Electrical Properties

Sample No.	Methacrylic Acid, wt %	Average Resistance, milliohm-inch² B.S.(1)	A.S.(2)	Standard Deviation B.S.(1)	A.S.(2)	Standard Deviation % of Average B.S.(1)	A.S.(2)
339(3)	25	16	6	1.5	0.4	9.7	6.7
339(4)	25	8	6	0.7	1.2	9.2	20.5
340(3)	20	15	7	4.3	2.0	27.6	29.2
340(4)	20	10	7	1.2	2.0	11.9	27.7
341(3)	15	25	12	4.5	1.2	17.9	10.1
341(4)	15	15	13	2.6	3.9	16.8	29.9
342(3)	10	27	10	3.3	4.8	12.2	45.5
342(4)	10	13	11	2.4	1.3	18.0	12.3

B. Physical Properties

Sample No.	Methacrylic Acid, wt %	Average Thickness, mil Dry	B.S.(1)	A.S.(2)	Average Dimensional Changes(5), % Width B.S.(1)	Width A.S.(2)	Length B.S.(1)	Length A.S.(2)	Tensile Strength(1), psi	Elongation(1), %
339(3)	25	1.2	1.3	1.6	11.0	19.0	5.5	-4.5	1349	>100
339(4)	25	1.1	1.4	1.8	12.0	17.0	6.9	-5.5	1300	>100
340(3)	20	1.3	1.4	1.7	11.2	18.4	7.1	-4.0	1790	>100
340(4)	20	1.2	1.3	1.7	13.1	15.2	6.5	-7.5	1405	>100
341(3)	15	1.3	1.4	1.9	8.1	11.1	6.5	-5.0	1852	>100
341(4)	15	1.2	1.4	2.0	12.3	14.3	8.0	-6.0	1694	>100
342(3)	10	1.3	1.3	1.8	10.1	17.2	6.5	-5.5	1993	>100
342(4)	10	1.3	1.4	1.9	11.0	13.0	7.5	-8.5	1356	>100

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer. Resistance is average of 12 samples. Thickness is average of 16 samples. Dimensional change is average of 4 samples. Tensile strength and elongation are average of 4 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Neutralized and washed at 80°C
(4) Neutralized and washed at 97°C.
(5) Change from dry dimension.

TABLE 59. METHACRYLIC ACID-GRAFTED POLYETHYLENE
SAMPLE NO. 433 - GRAFTED AND CROSSLINKED

Grafting Solution Composition

15 wt % Methacrylic acid
85 wt % Benzene

Crosslinking Solution Composition

1.0 vol % Divinylbenzene
1.0 vol % Benzene
98.0 vol % Methanol

Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	72 °F
Atmosphere:	Nitrogen
Roll Length:	100 feet (5 rolls/reactor)

Conditions for Crosslinking

Dose Rate:	0.025 Mrad/hr
Total Dose:	0.550 Mrad
Temperature:	Ambient
Atmosphere:	Nitrogen

Exotherm During Grafting

Time to exotherm, hr:	0.5
Time to maximum exotherm, hr:	30.0
Maximum temperature, °F:	113

Neutralization and Washing Temperature

97°C

Note

Dow 400 (1 mil), Chicopee No. 44 interlayer
Footage shipped: 471

Results

Footage	Resistance, milliohm-inch^2
1-433-5	8
	9
1-433-25	12
	12
1-433-50	12
	14
1-433-75	15
	12
1-433-93	17
	19
2-433-5	9
	10
2-433-94	17
	13
3-433-5	7
	8
3-433-94	14
	14
4-433-5	11
	13
4-433-95	17
	16
5-433-5	8
	12
5-433-95	12
	13

TABLE 60, GRAFTING WITH ACRYLIC ACID-METHACRYLIC ACID MIXTURES GRAFTED ONLY

Grafting Solution Composition, Wt %

343	353	354	
12.5	8.2	16.8	Acrylic acid
12.5	16.8	8.2	Methacrylic acid
75.0	75.0	75.0	Benzene

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	72-75°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2 343	353	354
5(1)	15, 14	8, 7	11, 11
15(1)	12, 13	9, 9	9, 9
20(2)	12, 13	9, 9	9, 9
25(2)	14, 13	9, 9	11, 12

Exotherm Data

Time to exotherm, hr:	2.0	4.0	4.0
Time to maximum exotherm, hr:	14.0	14.0	14.0
Maximum temperature, °F:	76	78	78

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

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(1) Neutralized and washed at 80°C.
(2) Neutralized and washed at 97°C.

TABLE 61. GRAFTING WITH ACRYLIC ACID-METHACRYLIC ACID MIXTURES

GRAFTED AND CROSSLINKED

Grafted Solution Composition, Wt %

343	353	354	
12.5	8.2	16.8	Acrylic acid
12.5	16.8	8.2	Methacrylic acid
75.0	75.0	75.0	Benzene

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	72-75°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

A. Electrical Properties

Sample No.	Acrylic Acid Wt %	Methacrylic Acid Wt %	Average Resistance, milliohm-inch B.S.(1)	A.S.(2)	Standard Deviation B.S.(1)	A.S.(2)	Standard Deviation % of Average B.S.(1)	A.S.(2)
343(3)	12.5	12 5	13	8	2.6	2.1	20 4	25.4
343(4)	12.5	12.5	13	11	1.0	1.1	7.6	9.5
353(3)	8.2	16.8	13	9	1.8	1.5	13.7	17.7
353(4)	8.2	16.8	12	11	1.2	1.3	9.3	11.7
354(3)	16.8	8.2	15	10	2.6	2.1	20.4	25.4
354(4)	16.8	8.2	16	13	1.6	1.0	9.9	7.8

B. Physical Properties

Sample No.	Average Thickness, mil Dry	B.S.(1)	A.S.(2)	Average Dimensional Changes(5), % Width B.S.(1)	Width A.S.(2)	Length B.S.(1)	Length A.S.(2)	Tensile Strength(1), psi	Elongation(1), %
343(3)	1.3	1.6	1.7	12.0	10.0	10.9	8.4	1196	--
343(4)	1.4	1.6	1.7	12.9	10.9	12.4	17.8	634	47
353(3)	1.3	1.5	1.7	12.3	15.3	9.5	5.0	1005	--
353(4)	1.3	1.5	1.6	11.2	9.2	10.5	8.5	1337	>100
354(3)	1.3	1.5	1.7	9.0	9.0	10.6	6.7	1206	>100
354(4)	1.5	1.7	1.8	13.0	11.0	10.9	11.9	1005	--

Note
Resistance is average of 12 samples. Thickness is average of 16 samples. Dimensional change is average of 4 samples. Tensile strength and elongation are average of 4 samples. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Neutralized and washed at 80°C.
(4) Neutralized and washed at 97°C.
(5) Change from dry dimension.

TABLE 62. GRAFTING WITH ACRYLIC ACID-VINYLTOLUENE MIXTURES - SAMPLE NOS. 390-393 - GRAFTED ONLY

Grafting Solution Composition, Wt %

390	391	392	393	
20	15	10	5	Acrylic acid
5	10	15	20	Vinyltoluene
75	75	75	75	Benzene

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	75°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

Sample No.	Acrylic Acid, wt %	Vinyltoluene, wt %	Footage	Resistance, milliohm-inch^2
390	20	5	18	8, 9
390	20	5	23	8, 9
391	15	10	18	13, 14
391	15	10	23	14, 15
392	10	15	18	89, 80
392	10	15	23	83, 88
393	5	20	18	376, 341
393	5	20	24	380, 367

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.
Neutralized and washed at 97°C.

TABLE 63. GRAFTING WITH ACRYLIC ACID-VINYLTOLUENE MIXTURES - SAMPLE NOS. 394-397 - GRAFTED ONLY

Grafting Solution Composition, Wt %

394	395	396	397	
20	15	10	5	Acrylic acid
5	10	15	20	Vinyltoluene
70	70	70	70	Benzene
5	5	5	5	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	~75°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

Sample No.	Acrylic Acid, wt %	Vinyltoluene, wt %	Footage	Resistance, milliohm-inch^2
394	20	5	18	7, 12
394	20	5	23	6, 12
395	15	10	18	52, 63
395	15	10	23	65, 64
396	10	15	18	>3000
396	10	15	23	1507, 1531
397	5	20	18	>3000
397	5	20	24	>3000

Note

Prepared from Dow 400 (1 mil) polyethylene with cheesecloth interlayer.
Neutralized and washed at 97°C.

TABLE 64. GRAFTING WITH ACRYLIC ACID-VINYLTOLUENE MIXTURES

(NO TERMINATOR) - GRAFTED AND CROSSLINKED

Composition of Grafting Solution Wt %

390	391	392	
20	15	10	Acrylic acid
5	10	15	Vinyltoluene
75	75	75	Benzene

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	75°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

A. Electrical Properties

Sample No.	Acrylic Acid, wt %	Vinyltoluene, wt %	Average Resistance, milliohm-inch^2 B.S.(1)	Average Resistance, milliohm-inch^2 A.S.(2)	Standard Deviation B.S.(1)	Standard Deviation A.S.(2)	Standard Deviation % of Average B.S.(1)	Standard Deviation % of Average A.S.(2)
390	20	5	14	13	2.1	1.6	14.7	12.4
391	15	10	56	34	5.5	3.0	9.8	8.8
392	10	15	547	295	147.2	23.7	26.9	8.0

B. Physical Properties

Sample No.	Average Thickness, mil Dry	Average Thickness, mil B.S.(1)	Average Thickness, mil A.S.(2)	Average Dimensional Changes(3), % Width B.S.(1)	Width A.S.(2)	Length B.S.(1)	Length A.S.(2)	Tensile Strength(1), psi	Elongation(1), %
390	2.5	1.8	1.9	12.2	12.2	12.7	20.2	1445	>100
391	6.3	5.2	1.9	8.2	11.2	5.5	11.0	646	>100
392	6.1	6.1	2.8	1.0	10.1	1.5	-4.5	281	>100

Note

Resistance is average of 12 samples. Thickness is average of 16 samples. Hard texture of film prevented meaningful measurements in some cases. Dimensional change is average of 4 samples. Tensile strength and elongation are average of 4 samples. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Before sterilization (B.S.) - wet with 40% KOH.
(2) After sterilization (A.S.) - wet with 40% KOH.
(3) Change from dry dimension.

TABLE 65. GRAFTING WITH ACRYLIC ACID—VINYLTOLUENE MIXTURES

(WITH TERMINATOR) - GRAFTED AND CROSSLINKED

Composition of Grafting Solution Wt %

394	395	
20	15	Acrylic acid
5	10	Vinyltoluene
70	70	Benzene
5	5	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	~75 °F
Atmosphere:	Nitrogen
Roll Length:	25 feet

A. Electrical Properties

Sample No.	Acrylic Acid, wt %	Vinyltoluene, wt %	Average Resistance, milliohm-inch^2		Standard Deviation		Standard Deviation % of Average	
			B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)
394	20	5	45	52	82.6	47.0	183.8	89.7
395	15	10	310	high	212.7	--	68.6	--

B. Physical Properties

Sample No.	Average Thickness, mil			Average Dimensional Changes(3), %				Tensile Strength(1), psi	Elongation(1), %
				Width		Length			
	Dry	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)		
394	2.4	1.4	1.6	10.2	10.2	11.6	7.5	1267	>100
395	2.1	1.3	1.7	5.1	9.2	5.0	-10.0	1825	>100

Note
Resistance is average of 12 samples. Thickness is average of 16 samples. Hard texture of film prevented meaningful measurement of dry sample. Dimensional change is average of 4 samples. Tensile strength and elongation are average of 4 samples. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

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(1) Before sterilization (B.S.) - wet with 40% KOH.
(2) After sterilization (A.S.) - wet with 40% KOH.
(3) Change from dry dimension.

TABLE 66. ACONITIC AND ITACONIC ACID FOR GRAFTING POLYETHYLENE - GRAFTED ONLY

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	77°F
Atmosphere:	Nitrogen
Roll Length:	5 feet

Sample No.	Grafting Solution Composition, Wt %: Acrylic Acid	Aconitic Acid	Itaconic Acid	Methanol	Resistance, milliohm-inch^2
432	10	--	--	90	>3000
428	--	25	--	75	>3000
431	--	--	25	75	>3000
425	5	15	--	80	730
413	10	15	--	75	14
426	10	15	--	75	18
427	15	15	--	70	9
430	10	--	15	75	112

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.
Neutralized and washed at 97°C.

TABLE 67. GRAFTING WITH VINYLPYRROLIDONE MIXTURES GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %		
160	161	
12.5	18.75	Acrylic acid
12.5	6.25	N-vinyl-2-pyrrolidone
75.0	75.0	Benzene

Experimental Conditions for Grafting	
Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	74°F
Atmosphere:	Nitrogen
Roll Length:	30 feet

A. Electrical Properties

Sample No.	Average Resistance, milliohm-inch² B.S.[1]	A.S.[2]	Standard Deviation B.S.[1]	A.S.[2]	Standard Deviation % of Average B.S.[1]	A.S.[2]
160	26	13	3.3	1.4	12.6	10.7
161	20	10	4.3	4.2	21.3	41.6

B. Physical Properties

Sample No.	Thickness, mils Dry	B.S.[1]	A.S.[2]	Dimensional Changes[3], % Width B.S.[1]	Width A.S.[2]	Length B.S.[1]	Length A.S.[2]	Tensile Strength[1] (psi)	Elongation[1] (%)
160	1.8	1.5	1.7	7.0	14.0	5.0	-2.5	2064	>100
161	1.4	1.5	1.7	6.0	12.0	6.5	2.0	1701	>100

Note
Prepared from JPL polyethylene film with St. Regis paper interlayer. Neutralized and washed at 80°C. Resistance is average of 18 samples. Thickness is average of 24 samples. Dimensional change is average of 6 samples. Tensile strength and elongation are average of 6 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Change from dry dimensions.

TABLE 68. GRAFTING WITH VINYLPYRIDINE MIXTURES GRAFTED ONLY

Grafting Solution Composition, Wt %

184	239	240	
20	24	20	Acrylic acid
5	1	-	4-Vinylpyridine
-	-	5	2-Vinylpyridine
75	75	75	Water

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Atmosphere:	Nitrogen
Roll Length:	30 feet

Electrical Properties

Sample No.	Resistance Range, milliohm-inch^2	Grafting Temperature (^{o}F)	Time to Exotherm (hrs)	Time to Maximum Exotherm (hrs)	Maximum Temperature (^{o}F)
184	3-5	74	8.0	12.0	87
239	2-3	89	2.0	7.0	137
240	1-2	89	No exotherm		

Note

Prepared from JPL film with paper toweling interlayer.
Sample No. 184 neutralized and washed at 80^{o}C.
Sample Nos. 239 and 240 neutralized and washed at 97^{o}C.

TABLE 69. GRAFTING WITH VINYLPYRIDINE MIXTURE
SAMPLE NO. 184
GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
20 Acrylic acid	Dose Rate:	0.012 Mrad/hr
5 4-Vinylpyridine	Total Dose:	0.815 Mrad
75 Water	Atmosphere:	Nitrogen
	Roll Length:	30 feet

A. Electrical Properties

Average Resistance, milliohm-inch^2		Standard Deviation		Standard Deviation % of Average	
B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)
4	3	1.0	1.5	23.4	52.9

B. Physical Properties

Thickness, mils			Dimensional Changes(3), %				Tensile Strength(1) (psi)	Elongation(1) (%)
			Width		Length			
Dry	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)		
1.7	2.1	2.2	20.2	28.6	18.1	19.1	900	>100

Note
Resistance is average of 18 samples. Thickness is average of 24 samples. Dimensional change is average of 6 samples. Tensile strength and elongation are average of 6 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Change from dry dimension.

TABLE 70. GRAFTING WITH SODIUM VINYLSULFONATE MIXTURE SAMPLE NOS. 297 AND 310 GRAFTED ONLY

Grafting Solution Composition, Wt %

4.7 Sodium vinylsulfonate
18.8 Acrylic acid
76.5 Water plus 5.2 g $FeSO_4 \cdot 7H_2O$ per 2 kilo of grafting solution

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	72°F
Atmosphere:	Nitrogen
Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2 297	Resistance, milliohm-inch^2 310
6	5, 3	5, 6
15	6, 6	6, 6
25	5, 6	7, 6

Exotherm Data

	297	310
Time to exotherm, hrs:	4.0	2.5
Time to maximum exotherm, hrs:	5.5	3.5
Maximum temperature, °F:	77	78

Note

Neutralized and washed at 97°C. Prepared from Dow 560 E (1 mil) polyethylene film with St. Regis paper interlayer. Homopolymer precipitated.

TABLE 71. GRAFTING WITH AQUEOUS METHACRYLIC ACID SAMPLE NO. 299 - GRAFTED ONLY

Grafting Solution Composition, Wt %

25 Methacrylic acid
75 Water plus 5.2 g $FeSO_4 \cdot 7H_2O$ per 2 kilo of grafting solution

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	75°F
Atmosphere:	Nitrogen
Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	5, 4
15	7, 6
25	9, 9

Exotherm Data

Time to exotherm, hr:	No exotherm
Time to maximum exotherm, hr:	
Maximum exotherm, °F:	

Note

Prepared from Dow Resin 560E (1 mil) polyethylene film with St. Regis paper interlayer.
Neutralized and washed at 97°C.

TABLE 72. GRAFTING WITH AQUEOUS METHACRYLIC ACID SAMPLE NO. 345 - GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Methacrylic acid	Dose Rate:	0.012 Mrad/hr
75 Water plus 7.1 g $K_3Fe(CN)_6$ per 2 kilo of grafting solution	Total Dose:	0.815 Mrad
	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch²
Random sampling	5, 4

Exotherm Data

Time to exotherm, hr:	2.0
Time to maximum exotherm, hr:	8.0
Maximum temperature, °F:	94

Note

Neutralized and washed at 97°C. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

TABLE 73. POTASSIUM CONTENT OF GRAFTED FILM

Sample No.	Moisture Content %	Potassium, Film as Processed %	Potassium, Washed Film %	Potassium, Film after 40% KOH Soak %	Potassium, Sterilized Film %
1-120-29	12.5	11.5	9.4	11.3	-
2-120-8	11.6	8.8	7.7	9.8	-
3-120-114	10.1	9.1	7.3	9.0	-
1-120-28	8.4	-	-	-	13.5
2-120-7	7.1	-	-	-	11.1
3-120-115	8.2	-	-	-	10.2

Note
All potassium contents are based on dry film.

TABLE 74. EFFECT OF CHAIN TERMINATOR ON POTASSIUM CONTENT OF GRAFTED AND NEUTRALIZED FILM

Sample No.	CCl_4[1]	Potassium Content, %[2]
123-28	0	21.1
223-6	1.0	19.0
124-26	2.5	10.0
155-9	5.0	10.8
127-26	10.0	8.9

(1) Percent carbon tetrachloride in 25 wt % acrylic acid grafting solution.

(2) On dry-film basis.

TABLE 75. GRAFTING OF VARIOUS POLYETHYLENE FILMS IN 30-FOOT ROLLS

GRAFTED ONLY

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Atmosphere:	Nitrogen

Sample No.	Film	Footage[1]	Resistance, milliohm-inch^2	Grafting Temperature, °F[2]	Time to Exotherm, hr	Time to Maximum Temperature, hr	Maximum Temperature, °F
237	JPL - 1.0 mil	5[4]	7, 7	89	1.0	5.5	118
		15[4]	60, 48				
		25[4]	12, 36				
193	Dow 710M - 0.5 mil	5[3]	8, 6	80	7.0	9.0	89
		15[3]	7, 4				
		25[3]	6, 4				
194	Dow 710M - 1.0 mil	5[3]	12, 11	80	7.0	9.0	88
		15[3]	11, 13				
		25[3]	10, 9				
233	Dow 710M - 1.0 mil	5[3]	7, 4	89	2.0	3.5	118
		15[3]	5, 5				
		16[4]	3, 2				
		28[4]	3, 1				
234	Dow 710M - 1.0 mil	12[3]	11, 11	89	3.0	5.0	118
		23[3]	8, 7				
		4[4]	4, 4				
		11[4]	3, 5				
257	Dow 110E - 1.5 mil	5[3]	14, 16	77	2.5	3.5	100
		15[3]	16, 18				
		25[3]	16, 18				
258	Dow 110E - 1.5 mil	5[4]	22, 22	77	2.5	4.0	121
		15[4]	19, 17				
		25[4]	16, 16				

TABLE 75. (Continued)

Sample No.	Film	Footage[1]	Resistance, milliohm-inch^2	Grafting Temperature, °F[2]	Time to Exotherm, hr	Time to Maximum Temperature, hr	Maximum Temperature, °F
259	Dow 110E - 1.5 mil	5[4] 15[4] 25[4]	14, 13 15, 16 15, 16	77	2.5	4.5	106
282	Dow 400 - 2.0 mil	5[4] 15[4] 25[4]	12, 14 12, 13 11, 12	80	2.0	4.0	100
283	Dow 400 - 2.0 mil	5[4] 15[4] 25[4]	14, 15 10, 11 12, 12	80	4.5	5.0	84
284	Dow 400 - 2.0 mil	5[4] 15[4] 25[4]	17, 16 13, 13 12, 13	80	-	-	-
285	Dow 400 - 2.0 mil	5[4] 15[4] 25[4]	15, 14 10, 10 11, 11	80	3.5	5.5	100
289	Dow 510M - 1.0 mil	5[4] 15[4] 25[4]	11, 12 9, 8 8, 8	72	2.5	3.5	95
290	Dow 510M - 1.0 mil	5[4] 15[4] 25[4]	9, 10 7, 6 7, 7	72	2.0	3.5	102
293	Dow 560E- 1.0	5[4] 15[4] 25[4]	10, 10 8, 8 8, 8	72	2.0	3.5	77
294	Dow 560E- 1.0 mil	5[4] 15[4] 25[4]	9, 10 8, 8 9, 8	72	2.5	4.0	91

Note Interlayer material was St. Regis paper.

(1) Distance into roll.
(2) Ambient temperature in radiation cell.
(3) Neutralized and washed at 80°C.
(4) Neutralized and washed at 97°C.

TABLE 76. GRAFTING OF VARIOUS POLYETHYLENE FILMS IN 30-FOOT ROLLS

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Grafting Conditions

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Atmosphere:	Nitrogen

Crosslinking Conditions

Dose Rate:	0.025 Mrad/hr
Total Dose:	0.55 Mrad
Temperature:	Ambient
Atmosphere:	Nitrogen

Crosslinking Solutions, vol %

1 Divinylbenzene
1 Benzene
98 Methanol

A. Electrical Properties

Sample No.	Film	Average Resistance, milliohm-inch^2		Standard Deviation		Standard Deviation % of Average	
		B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)
234(3)	Dow 710M 1.0 mil	13	10	1.7	1.4	12.4	15.1
234(4)	Dow 710M 1.0 mil	7	7	1.0	0.6	13.4	9.2
257(3)	Dow 110E 1.5 mil	28	30	3.1	8.4	11.0	27.8
258(4)	Dow 110E 1.5 mil	16	18	2.8	4.2	17.8	22.6
282(4)	Dow 400 2.0 mil	17	16	1.8	1.9	10.5	11.9
283(4)	Dow 400 2.0 mil	18	15	2.7	2.5	15.2	16.8
284(4)	Dow 400 2.0 mil	19	17	3.4	3.1	18.2	18.7
285(4)	Dow 400 2.0 mil	18	16	1.4	1.7	7.8	11.0

TABLE 76. (Continued)

B. Physical Properties

	Thickness, mil			Dimensional Changes[5], %				Tensile	
				Width		Length		Strength[1]	Elongation[1]
Sample No.	Dry	B.S.[1]	A.S.[2]	B.S.[1]	A.S.[2]	B.S.[1]	A.S.[2]	(psi)	(%)
234[3]	1.5	1.8	2.1	13.0	12.0	12.9	9.9	883	95
234[4]	2.1	2.0	2.1	18.0	13.0	19.7	20.7	694	95
257[3]	2.2	2.5	3.2	12.2	10.2	9.0	2.0	1421	>100
258[4]	3.1	3.3	3.8	17.3	13.3	16.1	10.1	1214	>100
282[4]	2.6	2.8	3.1	8.0	6.0	8.9	5.5	1576	>100
283[4]	2.5	2.7	3.0	9.9	8.9	7.9	3.4	1598	>100
284[4]	2.8	3.0	3.3	10.9	8.9	8.8	6.8	1586	>100
285[4]	2.7	2.8	3.0	14.3	11.2	12.5	7.5	1524	>100

Note
Resistance is average of 18 samples. Thickness is average of 24 samples. Dimensional change is average of 6 samples. Tensile strength and elongation are average of 6 samples.

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(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Neutralized and washed at 80°C.
(4) Neutralized and washed at 97°C.
(5) Percent change from dry dimensions.

TABLE 77. GRAFTING OF DOW 400 POLYETHYLENE (New Supply)
SAMPLE NO. 328 - GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 Benzene	Total Dose:	0.815 Mrad
5 Carbon tetrachloride	Temperature:	77°F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	8, 10
15	8, 9
25	8, 8

Note

Prepared from Dow 400 (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and washed at 97°C.

TABLE 78. GRAFTING OF DOW 710M POLYETHYLENE
SAMPLE NOS. 235 AND 236 - GRAFTED ONLY

Grafting Solution Composition, Wt %

235	236	
20	15	Acrylic acid
76	82	Benzene
4	3	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	89°F
Atmosphere:	Nitrogen
Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2 20 wt % Acrylic Acid 235	Resistance, milliohm-inch^2 15 wt % Acrylic Acid 236
5	6, 4	8, 8
15	6, 5	7, 10
20	-	6, 5
25	5, 6	--

Exotherm Data

Time to exother, hrs:	3.0	3.0
Time to maximum exotherm, hrs:	6.0	6.0
Maximum temperature, °F:	92	94

Note

Prepared from Dow 710M (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and washed at 97°C.

TABLE 79. GRAFTING OF DOW 110E POLYETHYLENE
SAMPLE NOS. 260-263 - GRAFTED ONLY

Grafting Solution Composition, Wt %

260-261	262-263	
20	15	Acrylic acid
76	82	Benzene
4	3	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	77°F
Atmosphere:	Nitrogen
Roll Length:	30 feet

Electrical Properties

	Resistance, milliohm-inch^2			
	20 wt % Acrylic Acid		15 wt % Acrylic Acid	
Footage	260	261	262	263
5	25, 27	24, 20	39, 41	118, 278
15	18, 23	22, 22	37, 45	45, 49
25	36, 30	24, 27	39, 35	39, 33

Exotherm Data

	260	261	262	263
Time to exotherm, hr:	2.5	2.5	2.5	2.5
Time to maximum exotherm, hr:	4.0	4.0	4.0	4.0
Maximum temperature, °F:	100	100	98	97

Note

Prepared from Dow 110E (1.5 mil) polyethylene film with St. Regis paper interlayer. Neutralized and washed at 80°C.

TABLE 80. EVALUATION OF NYLON MESH INTERLAYER
SAMPLE NOS. 275-276 - GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 Benzene	Total Dose:	0.815 Mrad
5 Carbon tetrachloride	Temperature:	75°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2 275	276
5	8, 7	8, 7
15	8, 7	9, 9
25	7, 7	7, 10

Note

Prepared from JPL polyethylene film with white nylon mesh interlayer. Neutralized and washed at 97°C.

TABLE 81. EVALUATION OF CHEESECLOTH INTERLAYER SAMPLE NO. 270 - GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 Benzene	Total Dose:	0.815 Mrad
5 Carbon tetrachloride	Temperature:	82°F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	8, 8
15	8, 9
25	8, 9

Exotherm Data

Time to exotherm, hr:	5.0
Time to maximum exotherm, hr:	9.0
Maximum temperature, °F:	93

Note

Prepared from JPL polyethylene film with cheesecloth interlayer. Neutralized and washed at 97°C.

TABLE 82. EVALUATION OF CHEESECLOTH INTERLAYER SAMPLE NOS. 271-273 - GRAFTED ONLY

Grafting Solution Composition, Wt %

271	272	273	
20	15	10	Acrylic acid
76	82	88	Benzene
4	3	2	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	82°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2		
	20 wt % Acrylic Acid 271	15 wt % Acrylic Acid 272	10 wt % Acrylic Acid 273
5	9, 8	16, 15	36, 40
15	11, 9	17, 15	113, 45
25	11, 12	15, 15	136, 23

Exotherm Data

	271	272	273
Time to exotherm, hr:	3.5	3.0	3.0
Time to maximum exotherm, hr:	8.0	9.0	15.0
Maximum temperature, °F:	1000	99	88

Note

Prepared from JPL polyethylene film with cheesecloth interlayer. Neutralized and washed at 97°C.

TABLE 83. EVALUATION OF CHEESECLOTH INTERLAYER SAMPLE NOS. 329-333 & 335 - GRAFTED ONLY

Grafting Solution Composition, Wt %

329-330	331-332	333	335	
25	20	15	10	Acrylic acid
70	76	82	88	Benzene
5	4	3	2	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	77°F
Atmosphere:	Nitrogen
Roll Length:	30 feet

Electrical Properties

	Resistance, milliohm-inch^2					
	25 wt % Acrylic Acid		20 wt % Acrylic Acid		15 wt % Acrylic Acid	10 wt % Acrylic Acid
Footage	329	330	331	332	333	335
5	5, 6$^{(2)}$	6, 8$^{(1)}$	6, 8$^{(2)}$	9, 10$^{(1)}$	19, 17$^{(1)}$	15, 17$^{(1)}$
15	5, 6$^{(2)}$	6, 7$^{(1)}$	8, 8$^{(2)}$	8, 8$^{(1)}$	18, 20$^{(1)}$	17, 22$^{(1)}$
25	5, 6$^{(2)}$	5, 7$^{(2)}$	7, 7$^{(2)}$	8, 8$^{(2)}$	11, 12$^{(2)}$	14, 14$^{(2)}$
30	--	6, 7$^{(2)}$	--	7, 8$^{(2)}$	16, 10$^{(2)}$	14, 15$^{(2)}$

Exotherm Data

	329	330	331	332	333	335
Time to exotherm, hr:	--	5.0	7.0	7.0	8.5	--
Time to max. exotherm, hr:	--	13.5	14.0	15.0	14.0	--
Maximum temperature, °F:	--	90	100	99	81	--

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

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(1) Neutralized and washed at 80°C.
(2) Neturalized and washed at 97°C.

TABLE 84. BATTERY SEPARATOR MATERIAL PREPARED WITH CHEESECLOTH INTERLAYER

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

330	332	333	335	
25	20	15	10	Acrylic acid
70	76	82	88	Benzene
5	4	3	2	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	77°F
Atmosphere:	Nitrogen
Roll Length:	30 feet

A. Electrical Properties

Sample No.	Acrylic Acid concentration, wt %	Average Resistance, milliohm-inch² B.S.(1)	Average Resistance, milliohm-inch² A.S.(2)	Standard Deviation B.S.(1)	Standard Deviation A.S.(2)	Standard Deviation % of Average B.S.(1)	Standard Deviation % of Average A.S.(2)
330(3)	25	12	7	3.0	0.6	25.7	8.6
330(4)	25	7	6	0.6	0.5	9.2	8.0
332(3)	20	12	10	1.1	-2.1	9.3	20.5
332(4)	20	10	8	1.7	1.2	16.7	13.8
333(3)	15	24	18	3.0	3.1	12.3	17.8
333(4)	15	16	16	1.6	2.0	9.9	12.7
335(3)	10	33	85	8.5	58.5	26.0	69.0
335(4)	10	22	38	1.6	6.5	7.4	16.9

B. Physical Properties

Sample No.	Acrylic Acid conc. wt %	Average Thickness, mil Dry	Average Thickness, mil B.S.(1)	Average Thickness, mil A.S.(2)	Average Dimensional Changes(5), % Width B.S.(1)	Width A.S.(2)	Length B.S.(1)	Length A.S.(2)	Tensile Strength(1), psi	Elongation(1), %
330(3)	25	1.1	1.4	1.5	10.0	10.0	8.8	4.4	1026	80
330(4)	25	1.1	1.3	1.5	10.0	8.0	8.8	2.5	975	89
332(3)	20	1.2	1.4	1.6	9.0	7.0	8.9	3.0	1000	75
332(4)	20	1.1	1.3	1.5	11.0	10.0	8.4	1.5	1093	87
333(3)	15	1.2	1.3	1.5	7.0	6.0	6.4	-0.5	1445	95
333(4)	15	1.2	1.3	1.5	10.0	9.0	6.9	-0.5	1334	95
335(3)	10	1.1	1.2	1.7	5.9	2.9	5.4	-3.5	1514	80
335(4)	10	1.1	1.2	1.6	8.0	6.0	6.4	-1.0	1351	83

Note
Resistance is average of 12 samples throughout roll. Thickness is average of 16 samples throughout roll. Dimensional change is average of 4 samples throughout roll. Tensile strength and elongation are average of 4 samples throughout roll.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Neutralized and washed at 80°C
(4) Neutralized and washed at 97°C.
(5) Change from dry dimension.

TABLE 85. EFFECT OF ACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION ON COMPOSITION OF THE GRAFTED FILM

Sample No.	Grafting Solution Composition, wt %: Acrylic Acid	CCl_4	C_6H_6	Footage	Degree of Grafting[1]	Degree of Grafting (Avg.)	Poly(acrylic acid) in Product (wt %)	(Avg.)	(meq[2]/g)	(Avg.)
S-81	25	5	75	5	0.85	0.87	45.9	46.4	6.38	6.44
				10	0.86		46.2		6.42	
				15	0.89		47.1		6.54	
				20	0.86		46.2		6.42	
S-82	20	4	76	5	0.75	0.70	42.9	41.1	5.96	5.70
				10	0.69		40.8		5.67	
				15	0.64		39.0		5.42	
				20	0.71		41.5		5.76	
S-83	15	3	82	5	0.51	0.52	33.8	34.0	4.69	4.72
				10	0.50		33.3		4.63	
				15	0.51		33.8		4.69	
				20	0.54		35.1		4.88	
S-84	10	2	88	5	0.40	0.38	28.6	27.5	3.97	3.82
				10	0.37		27.0		3.75	
				15	0.37		27.0		3.75	
				20	0.38		27.5		3.82	

Note

Dow 400 (1 mil) polyethylene film with Chicopee No. 44 cheesecloth interlayer. Neutralized and washed at 97°C.

(1) Weight of poly(acrylic acid) in grafted polyethylene film/weight of starting polyethylene film.
(2) Theoretical milliequivalent.

TABLE 86. EFFECT OF ACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION ON RESISTANCE OF GRAFTED FILM SAMPLE NOS. S-81 - S-84 - GRAFTED ONLY

Grafting Solution Composition, Wt %

S-81	S-82	S-83	S-84	
25	20	15	10	Acrylic acid
70	76	82	88	Benzene
5	4	3	2	Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	79°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2			
	25 wt % Acrylic Acid S-81	20 wt % Acrylic Acid S-82	15 wt % Acrylic Acid S-83	10 wt % Acrylic Acid S-84
5	7	9	12	14
10	7	9	13	14
15	7	10	11	16
20	6	9	10	16

Exotherm Data

Time to exotherm, hr:	12.0	12.0	12.0	12.0
Time to max. exotherm, hr:	20.0	20.0	20.0	20.0
Maximum temperature, °F:	82	81	81	82

Note

Prepared from Dow 400 (1 mil) polyethylene film with Chicopee No. 44 cheesecloth interlayer. Neutralized and washed at 97°C.

TABLE 87. EXOTHERM AND ROLL CONFIGURATION

Sample No.	Initial Temperature (°F)	Time to Exotherm (hrs)	Time to Maximum Exotherm (hrs)	Maximum Temperature (°F)	Roll Configuration	Comments
114	72	2	12	149	600-ft roll, 1/4" pipe core	Air conditioner on in cell. Recovered 120 feet of film.
115	61	4	7	142	600-ft roll, 1/4" pipe core	Air conditioner on in cell. Recovered 220 feet of film.
117	73	6	10	107	600-ft roll, 1/4" pipe core	Air conditioner on in cell. Recovered 310 feet of film.
118	61	4	30	149	600-ft roll, 6" core.	Air conditioner on in cell; 550 feet of film recovered.
119	66	4	18	136	600-ft roll, 4" core with aluminum screen at 200 and 400 ft.	Air conditioner on in cell; all film recovered.
120	61	5	30	85	8 100-ft rolls on 1/4" pipe core	Air conditioner on in cell; all film recovered.
152	72	2	52	117	600-ft roll, 1/4" pipe core with screen at 100, 200, 300, 400, and 500 ft.	Air conditioner on in cell. All of film recovered.
153	72	4	58	97	600-ft roll, 1/4" pipe core with screen at 33, 100, 200, 300, 400, and 500 ft.	Air conditioner on in cell. St. Regis paper used; all of film recovered.
162	77	6	20	140	Same	Air conditioner on in cell. 100 ft of film recovered.
163	78	6	20	127	Same	Air conditioner on in cell. All of film recovered.

Note Brenner-Filmark paper used unless otherwise noted.

TABLE 88. SAMPLE NUMBER 118

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Crosslinking Solution Composition, vol %

1.0 Divinylbenzene
1.0 Benzene
98.0 Methanol

Experimental Conditions for Grafting

Dose rate:	0.012 Mrad/hr
Total dose:	0.815 Mrad
Temperature:	61°F
Atmosphere:	Nitrogen
Roll length:	500 feet

Experimental Conditions for Crosslinking

Dose rate:	0.022 Mrad/hr
Total dose:	0.550 Mrad
Temperature:	78°F
Atmosphere:	Nitrogen

Exotherm During Grafting

Time to exotherm, hrs:	4.0
Time to maximum exotherm, hrs:	30.0
Maximum temperature, °F	149

Neutralization and Washing Temperature

80°C

Footage Shipped

500

Results

Footage	Resistance, milliohm-inch^2
10	14
20	18
28	13
30	15
40	13
50	12
60	12
70	15
80	9
90	10
100	13
110	6
120	7
130	12
140	10
160	12
170	6
180	9
190	13
200	6
210	7
220	10
230	9
240	5
250	11
260	10
270	8
280	12
290	7
300	4
310	8
320	5
330	5
340	7
350	9
360	5
370	6
380	12
390	8
400	7
410	11
420	15
430	7
440	20
450	14
460	11
470	9
480	17
490	12
500	20
510	20

TABLE 89. SAMPLE NO. 119

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	66°F
Atmosphere:	Nitrogen
Roll Length:	600 feet

A. **Electrical Properties**

Sample No. - Footage	Resistance, milliohm-inch² B.S.(1)	A.S.(2)	Average B.S.(1)	A.S.(2)	Standard Deviation B.S.(1)	A.S.(2)	Standard Deviation % of Average B.S.(1)	A.S.(2)
119-300	10	4						
	15	19						
	19	25						
	14	17						
	12	12						
	15	19	14	16	3.1	7.2	21.6	45.1

B **Physical Properties**

Sample No. - Footage	Thickness, mil Dry	B.S.(1)	A.S.(2)	Dimensional Changes(3), % Width B.S.(1)	Width A.S.(2)	Length B.S.(1)	Length A.S.(2)	Tensile Strength(1), psi	Elongation(1) %
119-300	1.3	1.4	1 8	8.0	10.0	2.0	-9.9	1555	67
	1.3	1.5	2.0	8.0	8.0	4.0	-5.9	1790	75
	1.0	1.3	1 5	10.0	12.0	4.0	-5.0		
	1 1	1.3	1.6	9 1	11.1	3.0	-4.0		
	1.1	1 3	1.7	8.0	8.0	4.0	-4.0		
	1 5	1 7	2.3	12 3	14.3	5.0	-3 0		
	1 3	1 5	1 9	10.0	10.0	5 0	-1.0		
	1.0	1.2	1.6	10.0	8.0	4.0	-3.0		
	1 1	1 3	1.8	8.0	8.0	2 0	-5.0		
	1 2	1.3	1.8	14 0	10.0	5.0	-5.9		
	1.0	1.2	1.5	10.0	12.0	4.0	-2.0		
	1 5	1.6	1 6	8.0	10 0	5.0	-4.0		
Average	1.2	1.4	1 8	9.6	10.1	3.9	-4.4		

Note

Neutralized and washed at 80°C
Footage shipped - 742

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Change from dry dimensions.

TABLE 90. SAMPLE NUMBER 152

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Crosslinking Solution Composition, vol %

1.0 Divinylbenzene
1.0 Benzene
98.0 Methanol

Experimental Conditions for Grafting

Dose rate: 0.012 Mrad/hr
Total dose: 0.815 Mrad
Temperature: 72°F
Atmosphere: Nitrogen
Roll Length: 600 feet

Experimental Conditions for Crosslinking

Dose rate: 0.025 Mrad/hr
Total dose: 0.550 Mrad
Temperature: 75°F
Atmosphere: Nitrogen

Exotherm During Grafting

Time to exotherm, hrs: 2.0
Time to maximum exotherm, hrs: 52.0
Maximum temperature, °F: 117

Neutralization and Washing Temperature

80°C

Footage Shipped

500

Results

Footage	Resistance, milliohm-inch^2
120	12
140	9
160	35, 27
180	40, 29
200	11
220	13
240	14
260	11
280	20
300	19
320	11
340	14
360	16
380	25
400	18
420	16
440	12
460	11
480	15, 11
500	29, 27
520	38, 36
540	17
560	43, 32
580	19
590	19

TABLE 91. SAMPLE NUMBER 153

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Crosslinking Solution Composition, vol %

1.0 Divinylbenzene
1.0 Benzene
98.0 Methanol

Experimental Conditions for Grafting

Dose rate:	0.012 Mrad/hr
Total dose:	0.815 Mrad
Temperature:	72°F
Atmosphere:	Nitrogen
Roll length:	600 feet

Experimental Conditions for Crosslinking

Dose rate:	0.025 Mrad/hr
Total dose:	0.550 Mrad
Temperature:	75°F
Atmosphere:	Nitrogen

Exotherm During Grafting

Time to exotherm, hrs:	4.0
Time to maximum exotherm, hrs:	58.0
Maximum temperature, °F:	97

Neutralization and Washing Temperature

80° F

Footage Shipped

575

Results

Footage	Resistance, milliohm-inch^2
10	10
20	14
40	17
60	12
80	12
100	12
120	10
140	23
160	17
180	13
200	10
220	22
240	15
260	16
280	15
300	15
320	11
340	14
360	18
380	25
400	12
420	15
440	19
460	14
480	12
500	21
520	12
540	14
560	20
580	19
600	31

TABLE 92. SAMPLE NUMBER 163

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Crosslinking Solution Composition, vol %

1.0 Divinylbenzene
1.0 Benzene
98.0 Methanol

Experimental Conditions for Grafting

Dose rate:	0.012 Mrad/hr
Total dose:	0.815 Mrad
Temperature:	78°F
Atmosphere:	Nitrogen
Roll length:	600 feet

Experimental Conditions for Crosslinking

Dose rate:	0.025 Mrad/hr
Total dose:	0.550 Mrad
Temperature:	75°F
Atmosphere:	Nitrogen

Exotherm During Grafting

Time to exotherm, hrs:	6.0
Time to maximum exotherm, hrs:	20.0
Maximum temperature, °F:	127

Neutralization and Washing Temperature

80°C

Footage Shipped

600

Results

Footage	Resistance, milliohm-inch^2
60	9
80	12
100	10
120	11
140	15
160	18
180	17
200	9
220	8
240	23
260	22, 16
280	6
300	15
320	10
340	22
360	24
380	22
400	9
420	21
440	20
460	74, 96
480	7
500	24
520	16
540	27, 28
560	26
580	16
600	12
620	24
640	18
660	20

TABLE 93. SAMPLE NUMBER 249
GRAFTED ONLY

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	61°F
Atmosphere:	Nitrogen
Roll Length:	100 feet (6 rolls)

Electrical Properties

Footage	Resistance, milliohm-inch^2 1-249 (roll no. 1)	6-249 (roll no. 2)
1	12	34, 85
10	10	70, 167
20	19	1871, >3000
30	28, 32	52
40	11	158
50	42, 42	2479, >3000
60	45	46
70	13	98
80	29	>3000
90	32	114
100	19	78
114	16	-

Exotherm Data

Time to exotherm, hrs:	3.0
Time to maximum exotherm, hrs:	14.0
Maximum temperature, °F:	143

Note

Prepared from JPL polyethylene film with St. Regis paper interlayer. Neutralized and washed at 80°C. Sample No. 1-249 from JPL polyethylene roll no. 1. Sample No. 6-249 from JPL polyethylene film roll no. 2.

TABLE 94. SAMPLE NOS. 251 AND 252
GRAFTED ONLY

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	69°F
Atmosphere:	Nitrogen
Roll Length:	100 feet (6 rolls per reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2		
	4-251 (roll no. 1)	5-251 (roll no. 2)	1-252 (roll no. 1)
1	12	12	12
10	220, 67	76	422, 225
20	85	70	124
30	96	16	173, 297
40	165, 455	49	>3000
50	60	33	165, 466
60	60	35	117
70	48	35	124
80	59	63	346
90	61	535, 836	>3000
100	36	57	19

Note
Prepared from JPL polyethylene film with St. Regis paper interlayer. Neutralized and washed at 80°C. Sample No. 4-251 from JPL polyethylene roll no. 1. Sample No. 5-251 from JPL polyethylene roll no. 2. Sample No. 1-252 from JPL polyethylene roll no. 1.

TABLE 95. SAMPLE NOS. 253 & 254
GRAFTED ONLY

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	74°F
Atmosphere:	Nitrogen
Roll Length:	100 feet (6 rolls in each reactor)

Electrical Properties

Footage	Resistance, milliohm-inch² 2-253	3-254
10	21	62, 104
20	39, 11	88
30	60	132
40	485	120
50	264, 85	61
60	55	>3000
70	181	205
80	>3000	52
90	>3000	>3000
100	158	89

Exotherm Data

	2-253	3-254
Maximum temperature indicated by thermotabs at 40 and 80 feet, °F:	180, 175	170, 180

Note

Prepared from JPL polyethylene film (roll no. 1) with St. Regis paper interlayer. Neutralized and washed at 80°C.

TABLE 96. SAMPLE NUMBER 265
GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
25 Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 Benzene	Total Dose:	0.815 Mrad
5 Carbon tetrachloride	Temperature:	67°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (6 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2 1-265 (110E)	2-265 (JPL no. 2)
20	16, 16	45, 111
30	14, 11	223, 109
40	20, 16	69, 37
50	22, 16	85, 141
60	19, 13	344, 368
70	17, 13	101, 40
80	26, 17	53, 43
90	16, 15	67, 234
100	15, 14	966, 28

Exotherm Data

Time to exotherm, hrs:	4.0
Time to maximum exotherm, hrs:	20.0
Maximum temperature, °F:	148

Note

St. Regis paper interlayer used. Neutralized and washed at 80°C.
Sample No. 1-265 prepared from Dow polyethylene 110E (1.5 mil),
Sample No. 2-265 prepared from JPL polyethylene roll no. 2.

TABLE 97. EFFECT OF TEMPERATURE ON GRAFTING SAMPLE NO. 300 - GRAFTED ONLY

Grafting Solution Composition, Wt %

25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	64°F
Atmosphere:	Nitrogen
Roll Length:	100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
30	6
40	9
50	8
60	7
70	8
80	12
90	10
100	13
110	10

Exotherm Data

Thermotabs at 50-ft level in the roll indicated a temperature of 165°F.

Note

Neutralized and washed at 97°C. Prepared from JPL polyethylene film with cheesecloth interlayer. Used cooling coil in solution. Highly grafted with homopolymer impregnated in the film.

TABLE 98. EFFECT OF TEMPERATURE ON GRAFTING SAMPLE NOS. 305-306 - GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
15 Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 Benzene	Total Dose:	0.815 Mrad
3 Carbon tetrachloride	Temperature:	64°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	305	306
5	51, 28	22, 25
15	26, 17	--
25	42, 59	29, 20
35	26, 15	--
45	29, 33	27, 50
55	16, 29	--
65	21, 17	30, 117
75	64, 59	--
85	67, 109	35, 43
95	255, 50	--
105	44, 28	39, 70
115	186, >3000	139

Exotherm Data

Thermotabs at 50-ft level in the roll indicated a temperature less than 100°F.

Note

Neutralized and washed at 97°C. Prepared from old JPL polyethylene film with cheesecloth interlayer. Film has excellent clarity. Cooling coil in solution.

TABLE 99. EFFECT OF TEMPERATURE ON GRAFTING
SAMPLE NO. 337 - GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
15 Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 Benzene	Total Dose:	0.815 Mrad
3 Carbon tetrachloride	Temperature:	60°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
10	12, 14
20	9, 12
30	10, 18
40	12, 11
50	12, 7
60	10, 11
70	14, 12
80	19, 19
90	12, 13
100	14, 13

Exotherm Data

Thermotabs at 50-ft level in the roll indicated a temperature less than 100°F.

Note

Neutralized and washed at 97°C. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer. Cooling coil in solution.

TABLE 100. EFFECT OF TEMPERATURE ON GRAFTING SAMPLE NO. 309 - GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
15 Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 Benzene	Total Dose:	0.815 Mrad
3 Carbon tetrachloride	Temperature:	60°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet

Electrical Properties

Footage	Resistance, milliohm-inch²: JPL 2	Resistance, milliohm-inch²: Dow 560E
1	13	8
	15	7
5	15	8
	12	12
25	20	7
	20	9
45	21	8
	18	9
65	21	10
	25	10
85	26	11
	25	12
95	29	12
	34	14

Exotherm Data

Thermotabs at 50-ft level indicated a temperature of 130°F in JPL 2, and more than 140°F but less than 150°F in the Dow 560E.

Note

Neutralized and washed at 97°C. Prepared from 3 rolls of JPL No. 2 polyethylene film and 1 roll of Dow 560E (1 mil) polyethylene film; cheesecloth interlayer.

TABLE 101. EFFECT OF TEMPERATURE ON GRAFTING SAMPLE NO. 338 - GRAFTED ONLY

Grafting Solution Composition, Wt%	Experimental Conditions for Grafting	
15 Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 Benzene	Total Dose:	0.815 Mrad
3 Carbon tetrachloride	Temperature:	60°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
10	9, 10
20	10, 12
30	8, 8
40	10, 10
50	10, 10
60	8, 10
70	9, 9
80	11, 10
90	13, 10
100	10, 11

Exotherm Data

Thermotabs at 50-ft level in the roll indicated a temperature less than 150°F but more than 140°F.

Note

Neutralized and washed at 97°C. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

TABLE 102. EVALUATION OF NEW CHEESECLOTH SAMPLE NO. 356 - GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
15 Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 Benzene	Total Dose:	0.815 Mrad
3 Carbon tetrachloride	Temperature:	63°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (5 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
10	7, 9
20	7, 8
30	9, 9
40	8, 9
50	9, 9
60	9, 9
70	9, 9
80	7, 8
90	10, 9
100	7, 8

Exotherm Data

Time to exotherm, hr:	6.0
Time to maximum exotherm, hr:	18.0
Maximum temperature, °F:	113

Note

Prepared from Dow 400 (1 mil) polyethylene film with Chicopee No. 44 cheesecloth interlayer. Neutralized and washed at 97°C.

TABLE 103. EFFECT OF NEUTRALIZATION AND WASHING TEMPERATURE - SAMPLE NOS. 231 AND 232

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %
25 Acrylic acid
70 Benzene
5 Carbon tetrachloride

Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	72°F
Atmosphere:	Nitrogen
Roll Length:	100 feet (6 rolls per reactor)

Crosslinking Solution Composition, vol %
1 Divinylbenzene
1 Benzene
98 Methanol

Conditions for Crosslinking

Dose Rate:	0.025 Mrad/hr
Total Dose:	0.550 Mrad
Atmosphere:	Nitrogen

A. Electrical Properties

Sample No.	Average Resistance, milliohm-inch² B.S.(1)	A.S.(2)	Standard Deviation B.S.(1)	A.S.(2)	Standard Deviation % of Average B.S.(1)	A.S.(2)
231(3)	21	15	8.2	8.9	38.3	60.8
231(4)	15	18	9.1	19.6	60.9	108.8
232(3)	17	12	6.4	9.1	38.3	74.9
232(4)	11	11	3.9	6.4	36.2	58.4

B. Physical Properties

Sample No.	Thickness, mils Dry	B.S.(1)	A.S.(2)	Dimensional Changes(5), % Width B.S.(1)	Width A.S.(2)	Length B.S.(1)	Length A.S.(2)	Tensile Strength(1) (psi)	Elongation(1) (%)
231(3)	1.3	1.3	1.6	6.1	6.1	6.5	-2.0	1467	95
231(4)	1.7	1.4	1.6	9.1	6.1	9.0	2.0	1455	>100
232(3)	1.7	1.5	1.8	7.1	8.1	7.5	-1.0	1521	>100
232(4)	2.4	1.6	1.8	10.1	9.1	10.5	3.0	1394	>100

Note Prepared from JPL polyethylene film with paper toweling interlayer. Resistance is average of 30 samples. Thickness is average of 40 samples. Dimensional change is average of 10 samples. Tensile strength and elongation are averages of 10 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Neutralized and washed at 80°C.
(4) Neutralized and washed at 97°C.
(5) Change from dry dimensions.

TABLE 104. EFFECT OF NEUTRALIZING AND WASHING CONDITIONS
SAMPLE NO. 338 - GRAFTED ONLY

Grafting Solution Composition, Wt %	Experimental Conditions for Grafting	
15 Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 Benzene	Total Dose:	0.815 Mrad
3 Carbon tetrachloride	Temperature:	60°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
10(1)	12, 11
20(1)	13, 13
30(1)	14, 12
40(2)	12, 12
50(2)	15, 15
60(2)	16, 16
65(2)	16, 16
75(3)	17, 17
89(3)	17, 17
95(3)	18, 18

Exotherm Data

Time to exotherm, hr:	14.0
Time to maximum exotherm, hr:	22.0
Maximum temperature, °F:	157

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer. Machine neutralized and washed at 197°C.

(1) Left in 5% KOH solution over weekend.
(2) 31 minutes in KOH solution and left in water over weekend.
(3) 31 minutes in KOH solution and 31 minutes in water.

TABLE 105. CONDITIONS FOR PREPARATION OF MATERIAL SHIPPED TO JPL

Grafting Solution Composition

15 wt % Acrylic acid
82 wt % Benzene
3 wt % Carbon tetrachloride

Crosslinking Solution Composition

1.0 vol % Divinylbenzene
1.0 vol % Benzene
98.0 vol % Methanol

Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	as indicated
Atmosphere:	Nitrogen
Roll Length:	100 feet (5 rolls per reactor)

Conditions for Crosslinking

Dose Rate:	0.025 Mrad/hr
Total Dose:	0.550 Mrad
Temperature:	Ambient (75-85°F)

Neutralization and Washing Temperature

97°C

Remarks

Prepared from Dow 400 (1 mil) polyethylene with Chicopee No. 44 cheesecloth interlayer.

TABLE 106. MATERIAL SHIPPED TO JPL
SAMPLE NOS. 356 & 358

Roll-Sample-Footage	Resistance, milliohm-inch^2	Roll-Sample-Footage	Resistance, milliohm-inch^2
2-356-5	10, 12	1-358-3	10, 11
2-356-110	18, 17	1-358-100	16, 16
3-356-5	15, 15	2-358-3	11, 9
3-356-110	11, 11	2-358-100	14, 15
4-356-5	11, 11	3-358-3	12, 11
4-356-110	8, 9	3-358-100	16, 17
5-356-5	9, 10	4-358-3	15, 12
5-356-110	11, 10	4-358-60	19, 17
		4-358-100	16, 18
		5-358-3	16, 18
		5-358-100	13, 8

	Sample 356	Sample 358
Cell Temperature, °F:	63	59
Time to exotherm, hr:	6.0	6.0
Time to max. exotherm, hr:	18.0	15.0
Maximum temperature, °F:	113	161
Footage shipped:	410	530

TABLE 107. MATERIAL SHIPPED TO JPL
SAMPLE NOS. 359 & 360

Roll-Sample-Footage	Resistance, milliohm-inch^2	Roll-Sample-Footage	Resistance, milliohm-inch^2
1-359-4	12, 14	1-360-4	19, 16
1-359-65	12, 12	1-360-109	8, 9
1-359-110	14, 12	2-360-4	11, 11
2-359-4	10, 8	2-360-104	13, 9
2-359-110	20, 20	3-360-5	14, 14
3-359-4	13, 16	3-360-58	9, 8
3-359-110	10, 15	5-360-4	18, 18
4-359-6	11, 11	5-360-110	15, 16
4-359-110	8, 9		
5-359-4	9, 10		
5-359-110	11, 9		

	Sample 359	Sample 360
Cell Temperature, °F:	59	73
Time to exotherm, hr:	6.0	13.0
Time to max. exotherm, hr:	15.0	23.0
Maximum temperature, °F:	113	116
Footage shipped:	525	361

TABLE 108. MATERIAL SHIPPED TO JPL
SAMPLE NOS. 361 & 362

Roll-Sample-Footage	Resistance, milliohm-inch^2	Roll-Sample-Footage	Resistance, milliohm-inch^2
1-361-4	20, 21	1-362-6	18, 12
1-361-52	10, 11	1-362-125	14, 9
1-361-96	8, 8	2-362-6	12, 8
2-361-4	15, 16	2-362-124	12, 12
2-361-112	17, 13	3-362-6	17, 13
3-361-4	14, 11	3-362-122	8, 10
3-361-106	11, 12	4-362-6	20, 17
4-361-4	13, 13	4-362-122	11, 12
4-361-108	10, 12		
5-361-4	18, 19		
5-361-104	17, 14		

	Sample 361	Sample 362
Cell temperature, °F:	73	64
Time to exotherm, hr:	13.0	4.0
Time to max. exotherm, hr:	23.0	20.0
Maximum temperature, °F:	111	111
Footage shipped:	491	469

TABLE 109. MATERIAL SHIPPED TO JPL
SAMPLE NOS. 363 & 375

Roll-Sample-Footage	Resistance, milliohm-inch^2	Roll-Sample-Footage	Resistance, milliohm-inch^2
1-363-6	10, 9	1-375-8	16, 15
1-363-122	9, 10	1-375-100	11, 9
2-363-6	13, 9	2-375-8	11, 14
2-363-110	11, 17	2-375-100	14, 12
3-363-6	18, 14	3-375-5	18
3-363-118	10, 10	3-375-100	8
4-363-6	11, 9	4-375-5	12
4-363-123	10, 7	4-375-100	9
		5-375-5	11
		5-375-100	11

	Sample 363	Sample 375
Cell temperature, °F:	64	66
Time to exotherm, hr:	4.0	7.0
Time to max. exotherm, hr:	20.0	20.0
Maximum temperature, °F:	100	131
Footage shipped:	450	502

TABLE 110. MATERIAL SHIPPED TO JPL
SAMPLE NOS. 376 & 379

Roll-Sample-Footage	Resistance, milliohm-inch^2	Roll-Sample-Footage	Resistance, milliohm-inch^2
1-376-6	14, 14	1-379-6	13, 20
1-376-100	14, 14	1-379-100	8, 9
2-376-6	14, 16	2-379-6	14, 16
2-376-100	15, 18	2-379-90	7, 10
3-376-6	15, 10	3-379-6	17, 16
3-376-100	12, 19	3-379-103	7, 8
4-376-6	14, 13	4-379-6	13, 13
4-376-100	12, 16	4-379-100	10, 9
5-376-6	18, 17	5-379-6	21, 17
5-376-100	16, 13	5-379-100	6, 8

	Sample 376	Sample 379
Cell temperature, ^{o}F:	66	-
Time to exotherm, hr:	7.0	-
Time to max. exotherm, hr:	20.0	-
Maximum temperature, ^{o}F:	144	-
Footage shipped:	710	509

TABLE 111. MATERIAL SHIPPED TO JPL
SAMPLE NOS. 388 & 389

Roll-Sample-Footage	Resistance, milliohm-inch^2	Roll-Sample-Footage	Resistance, milliohm-inch^2
1-388-5	18, 17	2-389-6	11, 14
1-388-73	11, 13	2-389-80	9, 9
2-388-6	13, 12	3-389-6	13, 14
2-388-100	11, 12	3-389-100	11, 9
3-388-6	12, 12	4-389-6	15, 13
3-388-100	10, 11	4-389-100	9, 7
4-388-6	12, 15	5-389-6	10, 10
4-388-100	11, 14	5-389-100	11, 10
5-388-6	14, 13		
5-388-100	9, 8		

	Sample 388	Sample 389
Cell temperature, °F:	66	66
Time to exotherm, hr:	4.0	4.0
Time to max. exotherm, hr:	34.0	34.0
Maximum temperature, °F:	108	86
Footage shipped:	473	389

TABLE 112. MATERIAL SHIPPED TO JPL
SAMPLE NOS. 398 & 399

Roll-Sample-Footage	Resistance, milliohm-inch^2	Roll-Sample-Footage	Resistance, milliohm-inch^2
1-398-6	11, 15	1-399-6	12, 16
1-398-100	14, 11	1-399-100	11, 12
2-398-6	10, 13	2-399-6	9, 11
2-398-100	10, 12	2-399-100	10, 12
3-398-6	17, 20	3-399-6	14, 16
3-398-100	16, 14	3-399-100	13, 12
4-398-6	15, 18	4-399-6	17, 15
4-398-100	12, 10	4-399-100	11, 13
5-398-6	11, 14	5-399-6	10, 10
5-398-100	8, 12	5-399-100	11, 13

	Sample 398	Sample 399
Cell temperature, °F:	64	64
Time to exotherm, hr:	4.0	4.0
Time to max. exotherm, hr:	20.0	20.0
Maximum temperature, °F:	106	104
Footage shipped:	498	492

TABLE 113. MATERIAL SHIPPED TO JPL - SAMPLE NOS. 2-358 - 5-358

GRAFTED AND CROSSLINKED

Grafting Solution Composition, Wt %

15 Acrylic acid
82 Benzene
3 Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	59°F
Atmosphere:	Nitrogen
Roll Length:	100 feet (5 rolls per reactor)

A. Electrical Properties

Sample No.	Average Resistance, milliohm-inch[2] B.S.[1]	A.S.[2]	Standard Deviation B.S.[1]	A.S.[2]	Standard Deviation % of Average B.S.[1]	A.S.[2]
2-358	13	12	3.6	3.3	28.2	28.2
3-358	13	25	1.7	26.1	12.7	105.3
4-358	13	13	1.7	1.4	12.6	10.9
5-358	14	16	3.8	6.5	27.3	41.7

B. Physical Properties

Sample No.	Average Thickness, mil Dry	B.S.[1]	A.S.[2]	Average Dimensional Changes[3], % Width B.S.[1]	Width A.S.[2]	Length B.S.[1]	Length A.S.[2]	Tensile Strength[1], psi	Elongation[1], %
2-358	1.1	1.3	1.6	13.0	14.0	9.5	5.5	1286	>100
3-358	1.1	1.4	1.5	13.0	14.0	9.0	3.0	1270	>100
4-358	1.2	1.4	1.7	12.0	15.0	9.5	4.0	1398	>100
5-358	1.2	1.4	1.7	15.0	16.0	10.5	5.0	1302	>100

Note
Prepared from Dow 400 (1 mil) polyethylene film with Chicopee No. 44 cheesecloth interlayer. Neutralized and washed at 97°C. Resistance is average of 12 samples. Thickness is average of 16 samples. Dimensional change is average of 4 samples. Tensile strength and elongation are average of 4 samples.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Change from dry dimension.

TABLE 114. EFFECT OF DEGREE OF GRAFTING ON SILVER ION MIGRATION

GRAFTED ONLY

Sample No.	Grafting Solution Composition, Wt.% Acrylic Acid	CCl_4	C_6H_6	Degree of Grafting[(1)]	Resistance, milliohm-inch^2	Silver Pick-up, g x 10^6/hr-inch^2	Migration Rate, g Ag x 10^2/hr-inch^2-mol
S-81	25	5	70	0.87	7	0.42	4.50
S-82	20	4	76	0.70	9	0.49	3.31
S-83	15	3	82	0.52	11	1.00	1.55
S-84	10	2	88	0.38	15	1.05, 2.76	0.91, 0.79

NOTE: Neutralized and washed at 97°C.

(1) Weight of poly(acrylic acid) in grafted polyethylene film/weight of starting polyethylene film.

TABLE 115. EFFECT OF ACRYLIC ACID CONCENTRATION ON SILVER ION MIGRATION GRAFTED AND CROSSLINKED

Grafting Solution Composition, wt %

330	332	333	335	
25	20	15	10	Acrylic acid
70	76	82	88	Benzene
5	4	3	2	Carbon tetrachloride

Sample No.	Acrylic Acid wt %	Resistance, milliohm-inch^2	Silver Pick-Up, g x 10^6/hr-inch^2	Migration Rate, g Ag x 10^2/hr-inch^2-mol
330-6(1)	25	12	0.60	3.66
330-24(2)	25	7	0.65	4.85
332-6(1)	20	12	0.45	2.79
332-24(2)	20	10	0.72	3.69
333-6(1)	15	24	0.39	1.59
333-24(2)	15	16	0.78	1.95
335-6(1)	10	33	0.42	1.33
335-24(2)	10	22	0.58	1.61

(1) Neutralized and washed at 80°C.
(2) Neutralized and washed at 97°C.

TABLE 116. EFFECT OF TERMINATOR CONCENTRATION ON SILVER ION MIGRATION GRAFTED AND CROSSLINKED

Grafting Solution Composition, wt %

368	348	350	351	352	
15	15	15	15	15	Acrylic acid
85	82	80	77.5	70	Benzene
0	3	5	7.5	15	Carbon tetrachloride

Sample No.	CCl_4 Concentration, wt %	Resistance, milliohm-inch^2	Silver Pick-up, g x 10^6/hr-inch^2	Migration Rate, g Ag x 10^2/hr-inch^2-mol
368-6(1)	0	15	0.27	3.02
368-24(2)	0	11	1.13	1.98
348-6(1)	3	20	0.40	2.25
348-14(1)	3	20	0.52	1.90
348-24(2)	3	16	0.45, 0.48	2.18, 2.96
350-24(2)	5	19	1.76	1.59
351-6(1)	7.5	30	0.28	2.76
351-24(2)	7.5	36	0.34, 0.37	0.49, 1.18
352-6(1)	15	45	0.23	1.64
352-24(2)	15	89	0.27, 0.24	0.10, 0.50

(1) Neutralized and washed at 80°C.
(2) Neutralized and washed at 97°C.

TABLE 117. EFFECT OF DIVINYLBENZENE CONTENT OF CROSSLINKING SOLUTION ON SILVER ION MIGRATION

<u>GRAFTED AND CROSSLINKED</u>

Sample No.	Divinylbenzene in Crosslinking Solution, vol %	Atmosphere	Resistance, milliohm-inch^2	Silver Pick-up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol
2-120-55	1	Air	21	0.37	1.96
2-120-33	2	Air	12	1.13	3.83
3-120-26	4	Nitrogen	15	1.15	3.38
3-120-17	4	Air	12	0.40	1.91
3-120-49	8	Nitrogen	18	1.24	2.40
3-120-37	8	Air	21	0.39	2.18
3-120-63	16	Nitrogen	20	1.39	1.37
115-10[1]	1	Nitrogen	7	0.59	4.10

(1) Prepared in 600-foot roll.

TABLE 118. SILVER ION MIGRATION THROUGH POLYETHYLENE COGRAFTED WITH ACRYLIC ACID AND VINYLTOLUENE

Grafting Solution Composition, wt %

390	391	392	393	394	395	
20	15	10	5	20	15	Acrylic acid
5	10	15	20	5	10	Vinyltoluene
75	75	75	75	70	70	Benzene
0	0	0	0	5	5	Carbon tetrachloride

Sample No.	Acrylic Acid wt %	Vinyltoluene, wt %	CCl_4, wt %	Average Resistance, milliohm-inch^2		Silver Pick-Up, g x 10^6/hr/inch^2		Migration Rate, g Ag x 10^2/hr/inch^2/-mol	
				G	GX	G	GX	G	GX
390	20	5	0	9	14	0.40	1.01	5.40	9.45
391	15	10	0	14	56	0.17	1.92	4.16	3.36
392	10	15	0	85	547	0.16	1.28	0.05	3.96*
393	5	20	0	366	>3000	0.15	1.31	0.16	0.25
394	20	5	5	9	45	0.73	1.98	2.98	2.86
395	15	10	5	61	310	0.23	1.63	0.31	0.30

Note
All samples neutralized and washed at 97°C.
G - Grafted Only.
GX - Grafted and crosslinked with divinylbenzene.

*Sample rewashed at room temperature in 5% KOH and then soaked in 40% KOH before determining silver ion migration.

TABLE 119. SILVER ION MIGRATION THROUGH POLYETHYLENE FILM COGRAFTED WITH ACRYLIC ACID AND ANOTHER MONOMER

Sample No.	Resistance, milliohm-inch^2	Silver Pick-up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol	Grafting Solution
S-80	-	0.45	2.75	25 wt % acrylic acid, 5 wt % carbon tetrachloride, 5 wt % divinylbenzene in benzene
S-122	90	1.02, 1.00	0.49, 0.29	25 wt % acrylic acid, 5 wt % carbon tetrachloride, 10 wt % divinylbenzene in benzene
407G	10	0.66	4.17	25wt % acrylic acid, 5 wt % divinyl diethylene glycol diether, 5 wt % carbon tetrachloride in benzene
S-98	-	0.16	2.56	15 wt % acrylic acid, 10 wt % styrene in benzene
184GX	4	0.87	4.90	20 wt % acrylic acid, 5 wt % 4-vinylpyridine in water
240GX	2	1.09	8.78	20 wt % acrylic acid, 5 wt % 2-vinylpyridine in water
S-23	17	1.88	1.01	25 wt % 2-vinylpyridine in methanol and subsequently grafted with 25 wt % acrylic acid in benzene
413G	14	0.66	3.86	10 wt % acrylic acid, 15 wt % aconitic acid in methanol

TABLE 119. (Cont'd.)

Sample No.	Resistance, milliohm-inch^2	Silver Pick-Up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol	Grafting Solution
297G	6	0.34	6.30	18.8 wt % acrylic acid, 4.7 wt % sodium vinylsulfonate in water with 5.2 gm $FeSO_4 . 7H_2O$ per 2 kilo of grafting solution
343GX(1)	13	0.32	2.80	12.5 wt % acrylic acid, 12.5 wt % methacrylic acid in benzene
343GX	13	0.39	2.29	same as 343GX(1)
353GX(1)	13	0.30	3.53	8.2 wt % acrylic acid, 16.8 wt % methacrylic acid in benzene
353GX	12	0.36	2.97	same as 353GX(1)
354GX(1)	15	0.33	3.10	16.8 wt % acrylic acid, 8.2 wt % methacrylic acid in benzene
354GX	16	0.27	2.65	same as 354GX(1)

Note:
GX in sample number indicates grafted with acrylic acid and crosslinked with divinylbenzene. All others are grafted only.

(1)Neutralized and washed at 80°C; all others at 97°C.

TABLE 120. EFFECT OF TYPE OF TERMINATOR ON SILVER MIGRATION

Sample No.	Resistance, milliohm-inch^2	Silver Pick-up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol	Grafting Solution
213G	13	2.09	0.65	1 wt % acetone terminator and 25 wt % acrylic acid in benzene
213GX	25	2.51, 3.04, 2.81	0.28, 0.47, 0.52	same as 213G, but crosslinked with divinylbenzene
417G	13	0.70	3.10	1 wt % acetone terminator and 15 wt % acrylic acid in benzene
417GX	14	0.36	2.32	same as 417G, but crosslinked with divinylbenzene
214GX	26	1.57	0.33	2.5 wt % acetone terminator and 25 wt % acrylic acid in benzene
418 G	10	0.67	3.15	2 wt % acetone terminator and 15 wt % acrylic acid in benzene
419G	11	0.88	3.30	4 wt % acetone terminator and 15 wt % acrylic acid in benzene
422G	11	1.18	2.45	10 wt % acetone terminator and 15 wt % acrylic acid in benzene
422GX	12	1.42	1.96	same as 422G, but crosslinked with divinylbenzene

TABLE 120.(Cont'd.)

Sample No.	Resistance, milliohm-inch^2	Silver Pick-up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol	Grafting Solution
215G	15	2.34	0.80	2.5 wt % carbon disulfide terminator and 25 wt % acrylic acid in benzene
215GX	25	2.17, 2.60	0.91, 1.36	same as 215G, but crosslinked with divinylbenzene
183GX	22	2.58, 3.38	0.35, 0.47	25 wt % acrylic acid in benzene and saturated with sulfur
423G	11	1.07	2.62	15 wt % acrylic acid in benzene and saturated with sulfur
423GX	14	1.14	2.02	same as 423G, but crosslinked with divinylbenzene
149G	30	0.46	3.43	1 wt % dodecyl mercaptan terminator and 25 wt % acrylic acid in benzene

TABLE 121. EFFECT OF ADDITIVES IN ACRYLIC ACID GRAFTING SOLUTION ON SILVER ION MIGRATION

Sample No.	Average Resistance, milliohm-inch^2	Silver Pick-Up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol	Additive
128G	9	0.97	2.72	Iron naphthenate
144GX	11	1.31	1.53	Iron naphthenate
198G	21	2.90	0.38	Zinc naphthenate
210GX	15	2.98, 4.15	0.24, 0.22	Acenaphthene + CCl_4
220GX	13	2.17	1.59	Acenaphthene
211GX	35	3.10	0.23	Anthraquinone + CCl_4
274G	10	0.89	3.39	Calcium naphthenate
280G	11	1.82	2.56	Thiokol LP-6
315G	10	2.26	1.91	Ethynyl cyclohexanol
316G	12	5.70	0.73	Surfynol 104
189GX	--	2.04	0.23	Zinc naphthenate in methanol
317G	10	2.78	2.34	Surfynol 104 + CCl_4

Note

G in sample number indicates grafted with acrylic acid.
GX in sample number indicates grafted with acrylic acid and crosslinked with divinylbenzene.

TABLE 122. EFFECT OF POLYETHYLENE ON SILVER ION MIGRATION

Sample No.	Resistance, milliohm-inch^2	Silver Pick-up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol	Polyethylene Grafted
193GX	11	0.82	2.72	Dow 710M - 0.5 mil
234GX	7	1.02	3.53	Dow 710M - 1.0 mil
258GX	16	1.14	2.32	Dow 110E - 1.5 mil
289G	9	0.82	2.35	Dow 510E - 1.0 mil
293GX	14	1.20	2.47	Dow 560E - 1.0 mil
282GX	19	1.55	0.86	Dow 400 - 2.0 mil
330GX	12	0.60	3.66	Dow 400 - 1.0 mil

Note

G in sample number indicates grafted with acrylic acid.
GX in sample number indicates grafted with acrylic acid and crosslinked with divinylbenzene.

TABLE 123 MISCELLANEOUS SILVER ION MIGRATION STUDIES

Sample No.	Resistance, milliohms-inch^2	Silver Pick-up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol	Comments
3-356GX	15	0.70, 1.08	4.65, 3.72	Standard film.
3-356GX (A)	5	1.04	7.85	Heated in air for 1 hr at 110°C followed by boiling in water for 1 hr. Evaluated without drying.
3-356GX (B)	205	1.72	0.55	Heated in air for 1 hr at 110°C.
3-356GX (C)	15	1.55	1.70	Boiled in dimethylformamide for 1 hr. Washed in water at room temperature and air dried.
3-356GX (D)	19	1.95	1.88	Boiled in acetone for 1 hr and air dried.
3-356GX (E)	22	1.80	1.11	Boiled in butyl alcohol for 1 hr and air dried.
5-398GX-100	12	0.84	3.26	First roll through fresh solution in washing machine.
1-399GX-100	11	1.03	2.73	Tenth roll through washing machine.
1-389G	--	0.95	1.53	Evaluated in free acid form.
326G	20	2.08	0.91	Grafting solution solvent was cyclohexane.

TABLE 123. (Cont'd.)

Sample No.	Resistance, milliohms-inch^2	Silver Pick-up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol	Comments
326GX	20	1.16	1.69	Same as 326G.
312G	5	0.34	13.10	Grafted in aqueous acrylic acid solution containing $K_4Fe(CN)_6$.
313G	5	0.27	5.70	Grafted in aqueous acrylic acid solution containing $K_3Fe(CN)_6$.
313△	12	1.33	2.86	Sample of 313G heated for 15 minutes in air at 110°C.
346G	6	0.72	4.80	Grafted in aqueous acrylic acid solution containing $K_3Fe(CN)_6$.
4-376GX	--	0.76	3.07	Sample of 4-376GX exposed to additional 0.815 Mrads in air.
2-120G	14	2,05,1.66	1.48,3.58	Crosslinked with divinyl sulfone.
5-356GX	11	1.89	3.50	From shipment to JPL.
5-356GX	--	0.31	2.54	Sterilized in 40% KOH at 135°C.

TABLE 123 (Cont'd.)

Sample No.	Resistance, milliohms-inch^2	Silver Pick-up, g x 10^6/hr/inch^2	Migration Rate, g Ag x 10^2/hr/inch^2/mol	Comments
5-356GX	11	0.59	4.35	Rewashed in water at room temperature followed by methanol and benzene wash.
5-356GX	11	0.38	4.15	Rewashed with water at room temperature and air dried.

TABLE 124. EFFECT OF METHACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION ON SILVER ION MIGRATION

GRAFTED AND CROSSLINKED

Grafting Solution Composition, wt %

339	340	341	342	
25	20	15	10	Methacrylic acid
75	80	85	90	Benzene

Sample No.	Methacrylic Acid, wt %	Resistance, milliohm-inch^2	Silver Pick-Up, g x 10^6/hr-inch^2	Migration Rate, g Ag x 10^2/hr-inch^2-mol
339-6(1)	25	16	0.27	3.49
339-24(2)	25	8	0.70	4.76
340-6(1)	20	15	0.32	4.57
340-24(2)	20	10	0.57	3.99
341-6(1)	15	25	0.25	1.40
341-24(2)	15	15	0.59	3.14
342-6(1)	10	27	0.27	2.24
342-24(2)	10	13	0.50	3.43

(1) Neutralized and washed at 80°C.
(2) Neutralized and washed at 97°C.

TABLE 125. EFFECT OF ACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION ON HYDROXYL ION MIGRATION

GRAFTED AND CROSSLINKED

Grafting Solution Composition, wt %

330	332	333	335	
25	20	15	10	Acrylic acid
70	76	82	88	Benzene
5	4	3	2	Carbon tetrachloride

Sample No.	Acrylic Acid, wt %	Resistance, milliohm-inch^2	Hydroxyl Ion Migration mol x 10^3/min/inch^2	
			(1)	(2)
330-6(3)	25	12	4.75	7.60
330-24(4)	25	7	6.95	10.40
332-6(3)	20	12	4.50	7.20
332-24(4)	20	10	6.15	9.85
333-6(3)	15	24	4.64	6.50
333-24(4)	15	16	5.20	7.80
335-6(3)	10	33	4.73	6.15
335-24(4)	10	22	4.22	5.50

(1) Film as prepared.
(2) Corrected to 1-mil thickness (wet).
(3) Neutralized and washed at 80°C.
(4) Neutralized and washed at 97°C.

TABLE 126 EFFECT OF METHACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION ON HYDROXYL ION MIGRATION

GRAFTED AND CROSSLINKED

Grafting Solution Composition, wt %

339	340	341	342	
25	20	15	10	Methacrylic acid
75	80	85	90	Benzene

Sample No.	Methacrylic Acid, wt %	Resistance, milliohm-inch^2	Footage	Hydroxyl Ion Migration mol x 10^3/min/inch^2 (1)	(2)
339(3)	25	16	6	6.92	9.70
339(4)	25	8	24	7.14	10.70
340(3)	20	15	6	5.55	9.41
340(4)	20	10	24	6.89	11.70
341(3)	15	25	6	3.94	6.30
341(4)	15	15	24	7.92	10.30
342(3)	10	27	6	5.99	7.80
342(4)	10	13	24	6.80	10.90

Note: At 25°C and 40% potassium hydroxide.

(1) Film as prepared.
(2) Corrected to 1-mil thickness (wet).
(3) Neutralized and washed at 80°C.
(4) Neutralized and washed at 97°C.

TABLE 127. EFFECT OF TERMINATOR CONCENTRATION ON HYDROXYL ION MIGRATION

GRAFTED AND CROSSLINKED

Grafting Solution Composition, wt %

368	348	350	351	352	
15	15	15	15	15	Acrylic acid
85	82	80	77.5	70	Benzene
0	3	5	7.5	15	Carbon tetrachloride

Sample No.	CCl_4 Concentration, wt %	Resistance, milliohm-inch^2	Footage	Hydroxyl Ion Migration mol x 10^3/min/inch^2	
				(1)	(2)
368(3)	0	15	6	4.13	7.45
368(4)	0	11	24	4.08	6.10
348(3)	3	20	6	3.72	4.45
348(4)	3	16	24	4.46	7.15
350(3)	5	38	6	2.74	3.56
350(4)	5	19	24	3.50	4.20
351(3)	7.5	30	6	2.74	3.56
351(4)	7.5	36	24	3.74	4.47
352(3)	15	45	6	2.85	4.00
352(4)	15	89	24	1.44	1.73

Note: At 25°C and 40% potassium hydroxide.

(1) Film as prepared.
(2) Corrected to 1-mil thickness (wet).
(3) Neutralized and washed at 80°C.
(4) Neutralized and washed at 97°C.

TABLE 128. HYDROXYL ION MIGRATION THROUGH COGRAFTED POLYETHYLENE

Grafting Solution Composition, wt %

390	391	392	393	394	395	
20	15	10	5	20	15	Acrylic acid
5	10	15	20	5	10	Vinyltoluene
75	75	75	75	70	70	Benzene
--	--	--	--	5	5	Carbon tetrachloride

Sample No.	Acrylic Acid, wt %	Vinyltoluene, wt %	CCl_4, wt %	Average Resistance, milliohm-inch^2		Hydroxyl Ion Migration, mol x 10^3/min/inch^2	
				G	GX	G	GX
390	20	5	0	9	14	7.65	6.13
391	15	10	0	14	56	7.45	6.50
392	10	15	0	85	547	5.80	6.40
393	5	20	0	366	>3000	4.65	3.56
394	20	5	5	9	45	6.90	6.12
395	15	10	5	61	310	1.87	2.04

Note:

All samples neutralized and washed at 97°C.

G - Grafted only.

GX - Grafted and crosslinked with divinylbenzene

TABLE 12.9. EFFECT OF TERMINATOR CONCENTRATION ON ZINCATE ION MIGRATION

GRAFTED AND CROSSLINKED

Grafting Solution Composition, wt %

368	348	350	351	352	
15	15	15	15	15	Acrylic acid
85	82	80	77.5	70	Benzene
0	3	5	7.5	15	Carbon tetrachloride

Sample No.	CCl_4 Concentration, wt %	Resistance, milliohm-inch^2	Zincate Ion Migration, mol x 10^6/inch^2/min (1)	(2)
368	0	11	1.96	3.14
348	3	16	2.39	3.82
350	5	19	2.22	2.64
351	7.5	36	0.51	0.56
352	15	89	1.25	1.37

Note: All samples neutralized and washed at 97°C.

(1) Film as prepared.
(2) Corrected to 1-mil thickness (wet).

TABLE 130. ZINCATE ION MIGRATION THROUGH COGRAFTED POLYETHYLENE

GRAFTED AND CROSSLINKED

Grafting Solution Composition, wt %

390	391	392	394	395	
20	15	10	20	15	Acrylic acid
5	10	15	5	10	Vinyltoluene
75	75	75	70	70	Benzene
--	--	--	5	5	Carbon tetrachloride

Sample No.	Acrylic Acid, wt %	Vinyltoluene, wt %	CCl_4, wt %	Resistance, milliohm-inch^2	Zincate Ion Migration, mol x 10^6/inch^2/min (1)	(2)
390	20	5	0	14	3.10	7.13
391	15	10	0	56	0.79	0.95
392	10	15	0	547	0.007	0.01
394	20	5	5	45	4.10	6.55
395	15	10	5	310	0.69	1.17

Note: All samples neutralized and washed at 97°C.

(1) Film as prepared.
(2) Corrected to 1-mil thickness (wet).

APPENDIX A

Analytical Procedures

APPENDIX A

ANALYTICAL PROCEDURES

Electrical Resistance

A Lucite cell having dimensions as shown in Figure 1 and equipped with platinized platinum electrodes was utilized in all electrical resistance measurements. The resistance is read to the nearest milliohm on a Leeds and Northrup No. 8067 precision bridge. Alternating current (1000 cycle) is supplied to the system by means of a Hewlett Packard oscillator No. 200CD. The signal from the bridge is amplified with a Hewlett Packard amplifier No. 466A and is read with a Hewlett Packard AC voltmeter Model HP403 to determine the null setting of the bridge. A schematic and photograph of the apparatus are presented in Figures 2 and 3, respectively.

The procedure for determining the membrane resistance is as follows:

1. The cell is opened. A washer (0.5-inch I.D. and 1.0-inch O.D.) of the separator material is placed into the cell. Care is taken to prevent the material from obstructing the hole.

2. The cell is closed, bolted, and filled with 40 weight percent potassium hydroxide solution. The solution temperature is determined to the nearest 0.1°C and recorded. The cell resistance (R_W) is determined.

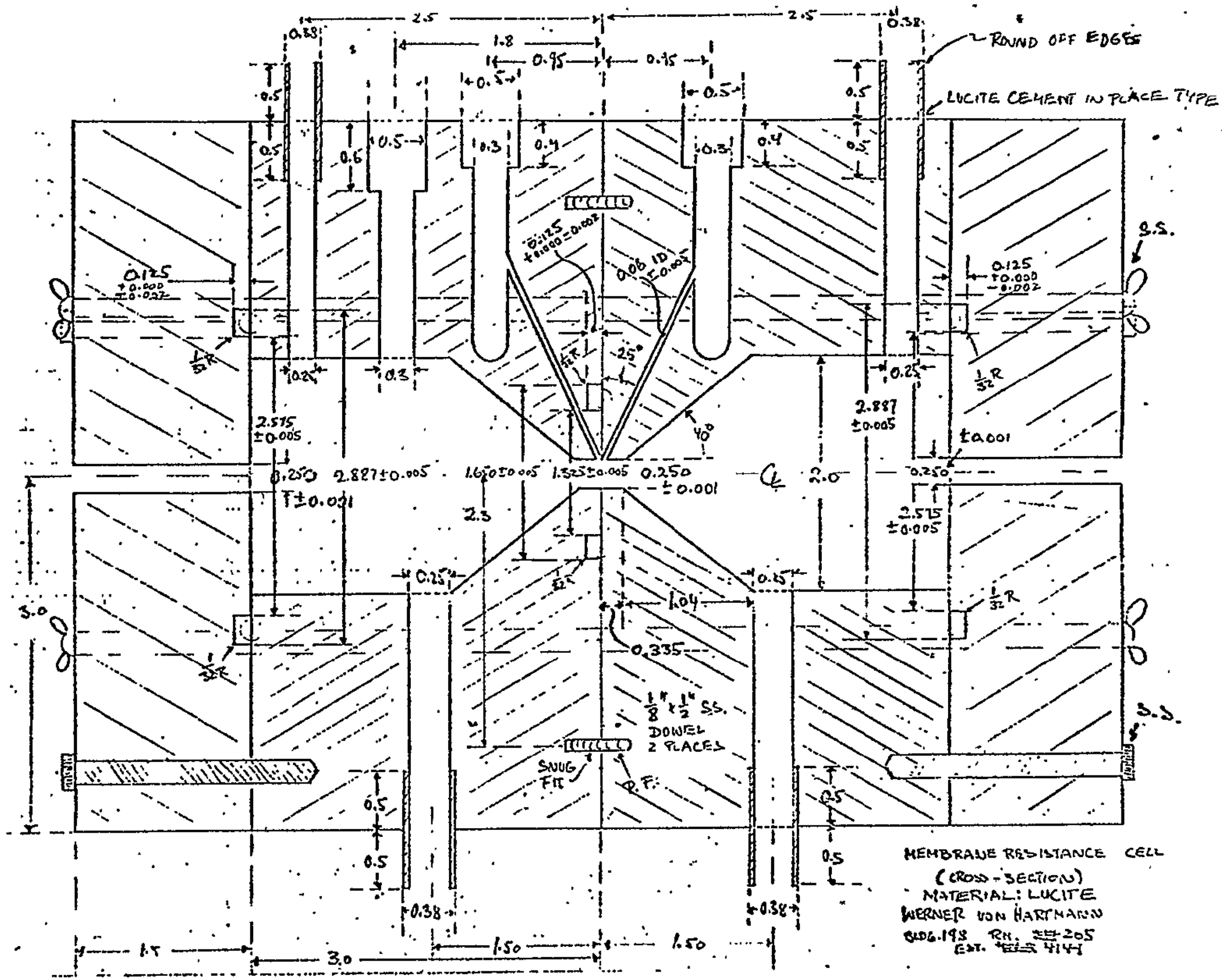

FIGURE 1. LUCITE RESISTANCE CELL

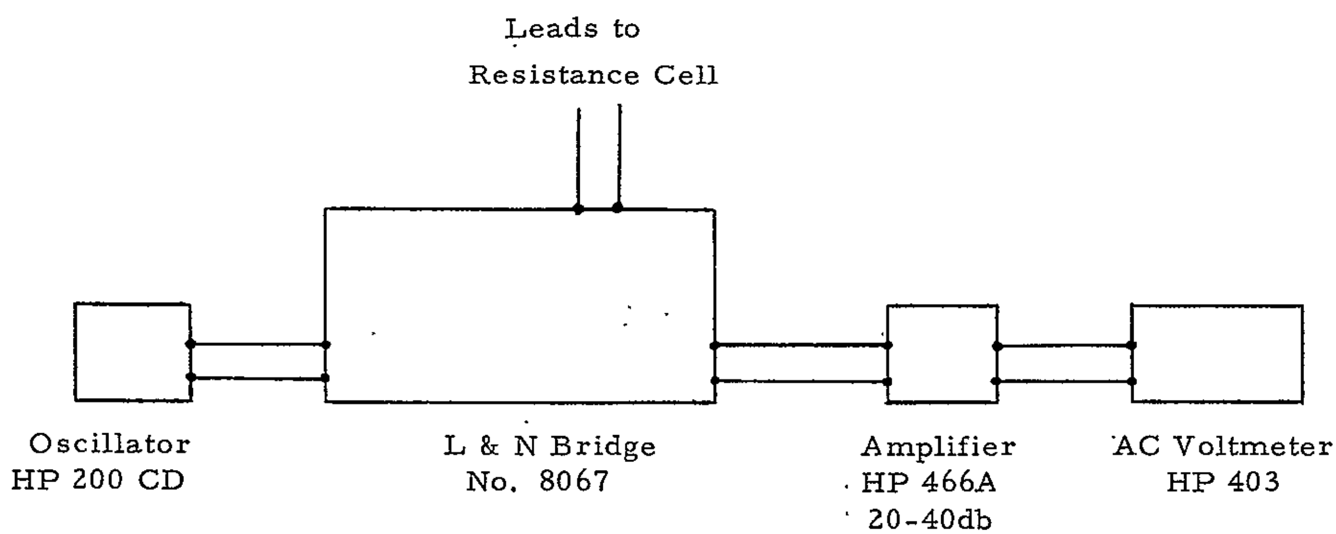

FIGURE 2. RESISTANCE APPARATUS SCHEMATIC

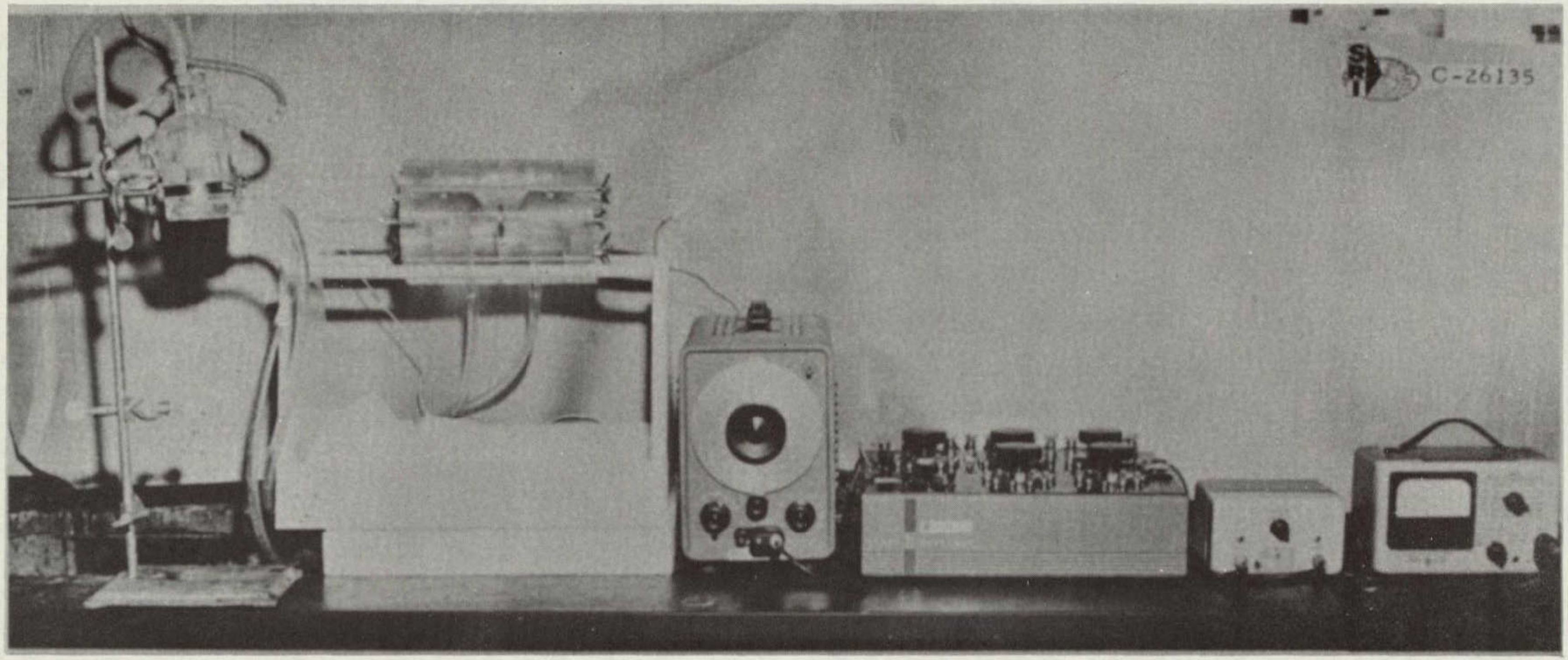

FIGURE 3. RESISTANCE APPARATUS

3. The potassium hydroxide solution is drained, the washer is removed, and the cell faces wiped with 5 percent acetic acid and dried.

4. A disc (0.5-inch diameter) of the separator material is inserted into the cell so that the material covers the 0.25-inch opening completely.

5. The cell is closed, bolted, and filled with 40 weight percent potassium hydroxide solution. The total resistance (R_T) of the membrane and cell is determined.

6. The membrane resistance in milliohm-inch^2 is obtained from the expression

$$\text{Membrane Resistance} = (R_T - R_W) \times A$$

where:

R_T = total resistance, milliohms

R_W = cell resistance, milliohms

A = film area, square inches

Thickness Measurements

Wet and dry thickness measurements are made with a Starrett thickness gauge which can be read to 0.0001 inch.

Dimensional Changes

The dimensional changes of the material are made on approximately 1-inch by 2-inch dry samples. All measurements are made to the nearest 1/50-inch with a stainless steel ruler and the changes calculated on the basis of the dry dimensions.

Wet dimensional changes before sterilization are determined after the samples are soaked for a minimum of 19 hours in 40 weight percent potassium hydroxide solution. The changes after sterilization are measured in a similar manner.

Sterilization

Sterilization chambers were fabricated from 12-inch lengths of 1-inch 316 stainless steel pipe and pipe caps. Each chamber contains a film holder consisting of a 316 stainless steel rod which has two pieces of 316 stainless steel attached to it. One piece of the screen is fastened to the bottom of the rod and the other about four inches above the bottom. Figure 4 is a photograph of the sterilization chamber.

Sterilization is conducted on samples which are cut for resistance and dimensional analysis. After making the necessary measurements on the samples, they are placed between the two screens and immersed in approximately 90 milliliters of 40 weight percent potassium hydroxide. The containers are flushed with nitrogen, sealed, and placed in a forced draft oven set at 135°C. The samples are held in the oven for 64 hours, cooled to room temperature, and transferred to plastic vials containing fresh 40 weight percent potassium hydroxide solution.

Tensile Strength and Elongation

The tensile strength and elongation are measured on 0.38-inch wide and 6-inch long strips which are wet with 40 weight percent potassium hydroxide solution using a Gardner Tensile Apparatus.

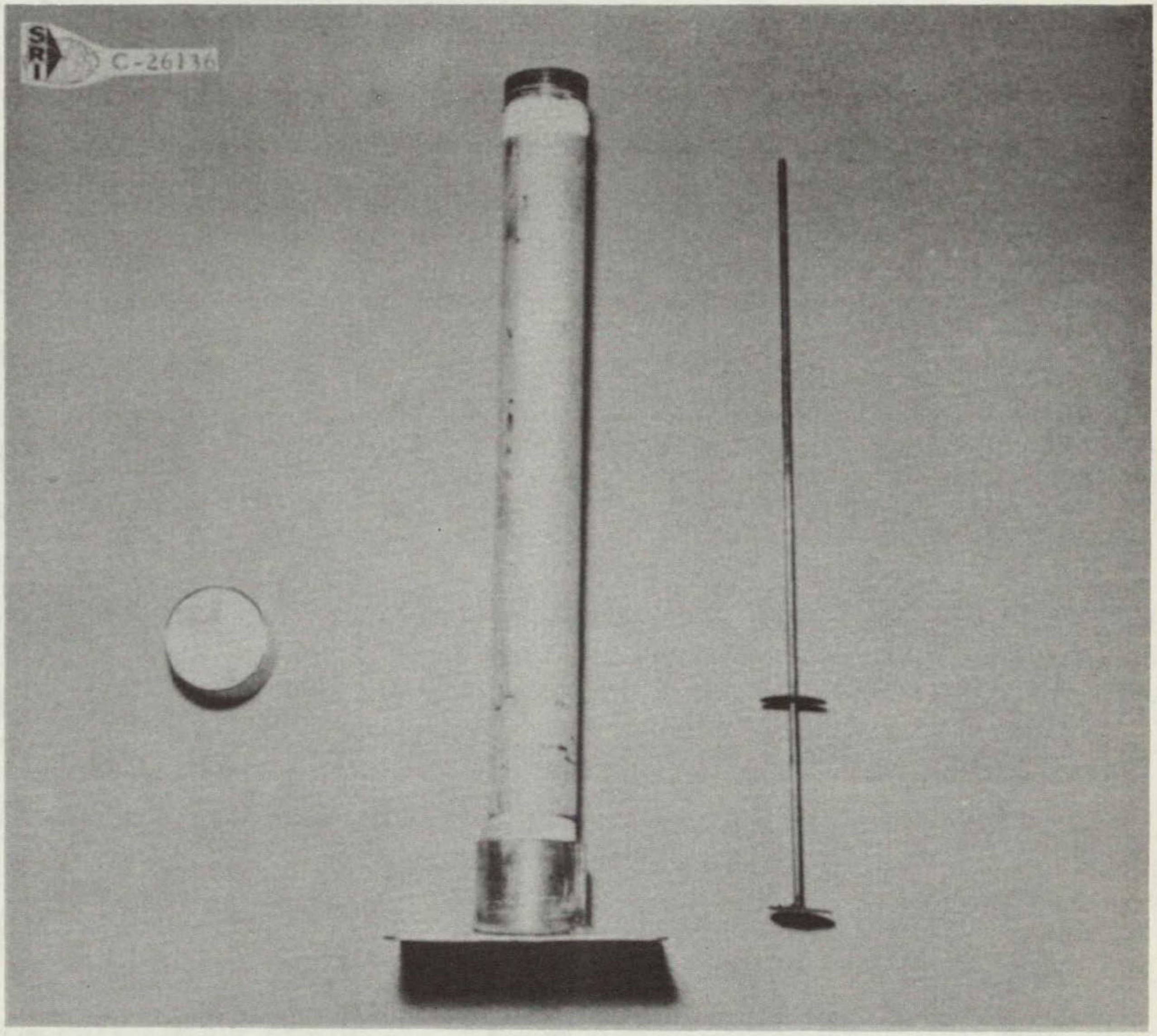

FIGURE 4. STERILIZATION CHAMBER

Silver Ion Migration

The method used for silver determination is the classical one for measuring the self-diffusion of ions utilizing the radioisotope tracer technique. The method used in this investigation is essentially that described by T. Dirkse (Chapter 10, J. E Cooper and Arthur Fleischer, Characteristics of Separators for Alkaline Silver Oxide-Zinc Secondary Batteries--Screening Methods)

The apparatus consists of wide mouth Erlenmeyer flasks (250 ml capacity) which were modified to accommodate the membrane between them, as shown in Figure 5. The counting equipment used in determining silver 110m concentration was a Packard Model 3002 Tri-Carb Scintillation Spectrometer.

The membrane to be tested is pre-soaked overnight in 40 weight percent potassium hydroxide. Prior to starting the run, the membrane is removed from the potassium hydroxide, blotted and sealed between Compartment A and B of the diffusion cell. Compartment A and B are then filled simultaneously with 200 ml of their respective solutions, the stirrers are started and the time recorded. Compartment A (hot side) solution contains 40 weight percent potassium hydroxide, a known amount of dissolved silver (as determined potentiometrically), and enough silver 110m isotope to produce approximately 36,000 cpm per 0.2 ml sample. Compartment B solution is 40 weight percent potassium hydroxide. Samples (0.2 ml) are taken from Compartments A and B after 0, 1, 2, 4, 8, and 24 hours.

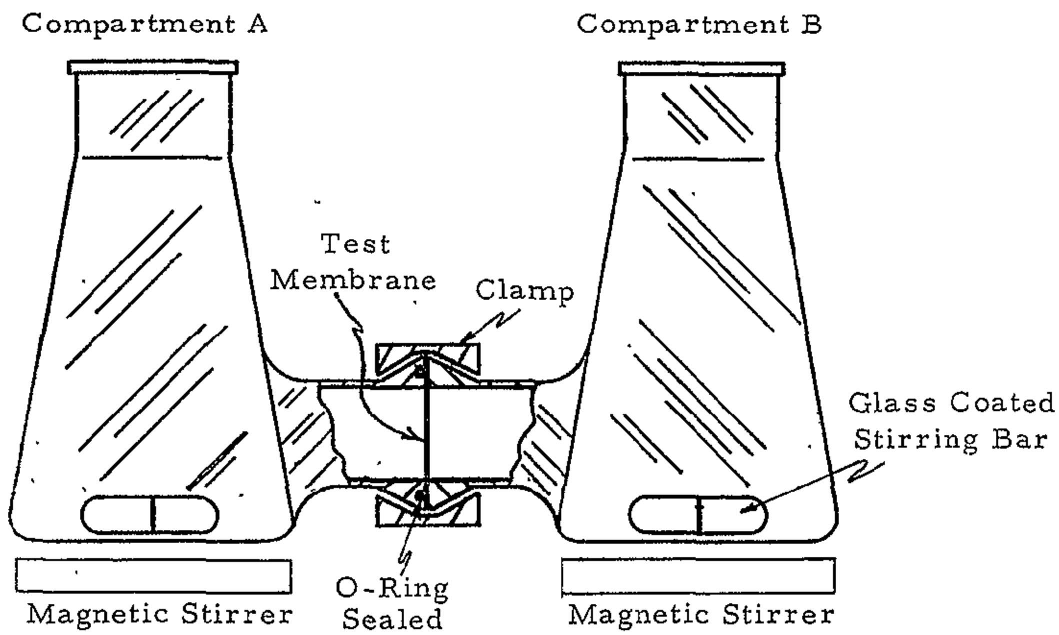

FIGURE 5. SILVER ION MIGRATION CELL

These samples are then neutralized with 0.2 ml of acetic acid and 15 ml of counting solution [4 grams of PPO(2, 5-Diphenyloxazole, Scintillation Grade) per liter of 2:1 toluene-Cellosolve mixture] added. The samples are then stirred and placed in the scintillation counter to cool. When cool, each sample is counted for 10 minutes. The silver migration rate is calculated using the 4-hour value.

At the end of the 24-hour run, the solutions from Compartments A and B are discarded and the membrane removed, washed with distilled water, placed in a scintillation vial with 15 ml of counting solution, and counted to determine deposited silver.

The silver pick-up and the rate of silver diffusion through the membrane are calculated by employing the following equations:

$$\text{Silver pick-up} = \frac{C_M}{C_{A_o}} \times \frac{A_o}{\Delta T} \times \frac{1}{a}$$

where

C_M = Counts per minute of the membrane

A_o = Grams of silver in 0.2 ml of solution in Compartment A at time zero.

C_{A_o} = Counts per minute for A_o.

a = Area of exposed membrane.

ΔT = Time in hours from start to test.

$$\text{Silver Rate} = \frac{C_B}{C_{A_o}} \times \frac{A_o}{\Delta T} \times \frac{1}{a} \times \frac{\text{Volume Factor}}{M_o}$$

where C_B = Corrected cpm in Compartment B (initially 0).

A_o = Grams of silver in 0.2 ml of solution in Compartment A at time zero.

C_{A_o} = Counts per minute for A_o.

a = Area of exposed membrane.

ΔT = Time in hours from start of test.

Volume factor = The factor needed to give the total amount of silver in Compartment B. (1000 when volume = 200 ml)

M_o = Molar concentration of silver in Compartment A at time zero.

Hydroxyl Ion Migration

The procedure used is described by E. L. Harris in Chapter 9, J. E. Cooper and Arthur Fleischer, Characteristics of Separators for Alkaline Silver Oxide-Zinc Secondary Batteries--Screening Methods.

Zincate Ion Migration

The procedure used is described by J. J. Lander in Chapter 11, J. E. Cooper and Arthur Fleischer, Characteristics of Separators for Alkaline Silver Oxide-Zinc Secondary Batteries--Screening Methods.

APPENDIX B

ELECTRON BEAM IRRADIATION OF THIN POLYETHYLENE FILM

APPENDIX B

ELECTRON BEAM IRRADIATION OF THIN POLYETHYLENE FILM

Thin polyethylene film supplied by Southwest Research Institute was irradiated by a beam of electrons for various total integrated doses. The film which was about 15 inches wide was cut into 36-inch lengths for irradiation. From each 36-inch piece a 2-inch piece was detached for use as a control. Each 34" piece and the corresponding 2-inch piece were coded for identification purposes. These 34" pieces were placed inside a 36" long by 29" wide by 1" deep nitrogen container with a 1-mil thick polypropylene window (approximately 35" x 28"). This chamber was flushed with nitrogen for approximately 4 minutes prior to the irradiation and during the irradiation. After irradiation, the 34" lengths were transferred to a polyethylene bag, purged of air with nitrogen, and stored for 15 hours in this nitrogen atmosphere.

The dose measurements were made using cellophane dosimetry.[1] The irradiated cellophane was read using a Fisher Spectrophotometer II, Model 81. One-foot squares were read prior to and subsequent to irradiation and the difference in light transmission was used to ascertain the integrated dose from the curves of reference 1. A cellophane foil was placed on either side of the sample to be irradiated and the actual integrated dose measured for each irradiated sample.

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1 E. J. Henley and D. Richman, Cellophane-Dye Dosimeter for 10^5 to 10^7 Roentgen Range, Analytical Chem., 28, No. 10, (1956).

These irradiations were performed using the Texas Nuclear Model 9800 Polycure accelerator. The operating voltage was maintained at 250 kV and the total dose varied by varying the current and/or making multiple passes under the electron beam. This electron beam was adjusted for a 28" scan, more than sufficient for the width of the material being irradiated. Some difficulty in maintaining the (low) current required for the lowest dose measurements was experienced which resulted in exposures which were in some cases quite different from the nominal doses requested.

The following table lists the integrated dose each sample received. No changes in the physical characteristics of this film were observed except a slight darkening in color of those samples which received very high dosages. The irradiated samples were stored in a nitrogen atmosphere for approximately 15 hours and then packaged and shipped to Southwest Research Institute for tests and evaluation.

Sample No.	Total Integrated Dose, megarads
1	0.91
2	0.40
3	2.34
4	2.98
5	7.56
6	15.43
7	38.5
8	77.0
9	4.71

APPENDIX C

PROCEDURE FOR PREPARATION OF BATTERY SEPARATOR MATERIAL

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I. Equipment

A. Reactor - The reactor is a flanged cylindrical chamber fabricated from aluminum sheet (1/8-inch thick). The inside diameter is 12-1/2 inches and the height is 30 inches. The end plates, 1/2-inch thick, are bolted on with a Buna N rubber gasket. A safety valve set for 15 psig and sufficient valving for conducting the necessary operations are mounted on the top plate.

B. Cobalt-60 Source - Any source capable of giving a uniform overall dose of gamma radiation to the reactor as specified for grafting and crosslinking. The irradiation configuration and reactors are shown in Figures 1 and 2. Approximately 10,000 curies of cobalt-60 is used in the parabolic holder.

C. Rolling Equipment - Any device can be used to roll 100 feet of polyethylene film which is backed with cheesecloth or paper onto a 1/2-inch capped aluminum pipe.

D. Neutralizing and Washing Device - Any device capable of washing long lengths of grafted polyethylene film may be used if a minimum contact time of 45 minutes in each bath (neutralizing and washing) can be obtained. A description of the device used at the Institute is given at the end of this preparatory procedure.

II. Grafting Procedure

A. Stock polyethylene film in one-hundred (100) foot length is backed with cheesecloth and rolled onto a 1/4-inch capped aluminum pipe.

B. Five (5) of the rolls are placed in aluminum reactor, and the reactor is sealed.

C. Two (2) reactors containing 5 rolls each of material are attached to a vacuum manifold.

D. The reactors are then evacuated for one and one-half hours to approximately 8 mm of Hg.

E. Nitrogen is then admitted to the reactors to a pressure of 5 psig.

F. The reactors are evacuated to 10-15 mm of Hg pressure and then pressured to 5 psig with nitrogen. This step is repeated three times.

G. The reactors are then evacuated for one hour (to about 8 mm of Hg), and each reactor is isolated from the vacuum manifold. The grafting solution (40 kilograms or 43.7 liters) is then added to each reactor by allowing it to be drawn into the reactor by the vacuum.

H. The reactors are evacuated to about 50 mm of Hg pressure and then pressured to 5 psig with nitrogen.

I. Step H is repeated twice.

J. The nitrogen is bled off to atmospheric pressure, the reactor vents are closed, and the reactors are allowed to stand at room temperature for about 24 hours.

K. The reactors are then placed on rotating platforms and exposed to cobalt-60 source for 68 hours and 5 minutes at a dose rate of 0.012 Mrad/hour. The temperature of the room is maintained at 60-65°F, and air is circulated over the reactors.

L The reactors are removed from the source and allowed to stand 24 hours ± 2 hours. The rolls of film are then removed from the reactors, unrolled, and washed in 5 weight percent KOH at 97°C for a minimum residence time of 45 minutes followed by a rinse in distilled water at 97°C for a minimum residence time of 45 minutes and air drying. The dried film is rolled up backed with paper toweling on a 1/4-inch aluminum pipe for crosslinking.

III. <u>Crosslinking Procedure</u>

A. Four (4) rolls of grafted film are placed in each reactor.

B. Two (2) reactors are attached to a vacuum manifold.

C. The reactors are evacuated for one and one-half hours to approximately 8 mm of Hg.

D. Nitrogen is then admitted to the reactors to a pressure of 5 psig.

E. The reactors are evacuated to 10-15 mm of Hg pressure, and then pressured to 5 psig with nitrogen. This step is repeated three times.

F. The reactors are then evacuated for one hour (to about 8 mm of Hg), and each reactor is isolated from the vacuum manifold. The crosslinking solution (38 liters) is then added to each reactor by allowing it to be drawn into the reactor by the vacuum.

G. The reactors are evacuated to about 50 mm of Hg pressure and then pressured to 5 psig with nitrogen.

H. Step G is repeated twice.

I. The nitrogen is bled off to atmospheric pressure, the reactor vents are closed, and the reactors are allowed to stand at room temperature for about 24 hours.

J. The reactors are then placed on rotating platforms and exposed to cobalt-60 for 22 hours at a dose rate of 0.025 Mrad/hr at room temperature.

K. The reactors are removed from the source and allowed to stand 24 hours $\pm$ 2 hours. The film is then removed from the reactors, unrolled, and washed by passing through a methanol bath. The film is dried and rolled up with a paper backing for storage or shipment.

IV. <u>Grafting Solution Composition</u>

15 wt % Acrylic acid

82 wt % Benzene

3 wt % Carbon tetrachloride

V. Crosslinking Solution Composition

1.0 vol % Divinylbenzene

1.0 vol % Benzene

98.0 vol % Methanol

VI. Materials

Materials	Grade	Supplier
Glacial acrylic acid	Contains 200 ppm MEHQ	Rohm & Haas 4120 Southwest Freeway Suite 118 Houston, Texas 77027
Benzene	Nitration	Texas Solvents & Chem. Co 8401 Market Street Houston, Texas 77029
Carbon tetrachloride	ACS	Matheson Scientific P. O. Box 9389 Houston, Texas
Divinylbenzene	Approximately 55% as DVB	Dow Chemical Company 3636 Richmond Avenue Houston, Texas
Methanol	ACS reagent	McKesson & Robbins 4351 Director Drive San Antonio, Texas
Paper	St. Regis L-1166 semi-bleached crepe for saturating basis (27 lb)	St. Regis Paper Company 1822 Prudential Bldg. Jacksonville, Fla. 32207
	Paper toweling	Banner-Filmark 650 Hinsdale Street Brooklyn, New York 11207
Cheesecloth	Chicopee 44 x 36	Chicopee Mills 2300 Stemmons Freeway Dallas, Texas 75207

VI. Materials (Cont'd.)

Materials	Grade	Supplier
Nitrogen	Water pumped	Local
Potassium hydroxide pellets	U.S.P.	McKesson & Robbins 4351 Director Drive San Antonio, Texas
Water, deionized	200,000 ohms resistance	Local
Polyethylene	Dow 400 1000 ppm $CaCO_3$	Supplied by JPL

VII. Film Processing Machine

An assembly drawing of the film processing machine for neutralizing and washing the grafted polyethylene film is shown in Figure 3. A complete set of engineering drawings, and one reproducible set, were sent to the Jet Propulsion Laboratory. The machine consists of 2 small tanks, 2 large tanks, a drying section, and a take-up device which rolled up the dried film with a paper toweling interlayer. Each small tank has 2 stationary idler rollers at the top and one removable idler roller at the bottom. Each large tank contains 8 stationary idler rollers at the top and 9 removable idler rollers at the bottom. The removable idler rollers are fastened to a frame so that they can be removed from the top to aid in threading the film leader through the machine. All parts which contact the film or solutions are made of stainless steel or plastic.

In starting material through the machine, a Nylon cord leader was used. All of the removable idlers were taken out of the machine and the Nylon cord placed over the fixed idlers and fastened to the rod of the take-up device. Starting at this same end, the idlers were replaced one at a time allowing additional Nylon cord to be fed to the machine until all of the idlers were replaced. The Nylon cord leader was tied to the end of the grafted film and the tanks filled with the required solutions to a level above the top idlers and heated to the required temperature. On starting the machine, the film was slowly fed through the machine. Successive rolls were fastened together with Nylon cord.

In the trial runs, the film passed through the first small tank which contained cold 5 percent potassium hydroxide, through the first large tank of hot 5 percent potassium hydroxide, and then through a large tank of hot deionized water. On leaving the hot water tank, the film passed through a small tank of methanol and through an air drying chamber at the end of the machine. On leaving the drying chamber, the film was layered with paper toweling and rolled onto an aluminum rod to form a roll suitable for crosslinking.

It was found that the cold 5 percent potassium hydroxide tank was not essential, that the film had a tendency to stick to the divider in each large tank and to the end of each large tank as the film left the tank, and that the methanol bath was not essential. To eliminate the problem of film sticking, the last bottom idler roller was removed from each section of each tank.

The procedure adopted for neutralizing and washing the grafted film was to feed the film through the hot (97 °C) 5 percent potassium hydroxide solution and the hot (97 °C) deionized water. On leaving the water, the film passed through the air dryer and was wound up interlayered with paper toweling for crosslinking.

The grafted film was fed through the machine at a rate of 0.734 feet per minute. As there were approximately 38 feet of film in each of the larger tanks, the residence time of the film in each bath was about 52 minutes. Each bath was filled with 170 gallons of liquid.

Warm air to the dryer was supplied by two Tornado Model 420 blowers in parallel.

The machine could also be used for washing the crosslinked film with methanol by using methanol in one of the small baths. Because of ventilation problems in the room housing the machine, the laboratory rolling device was used instead, as it was located in a walk-in hood.

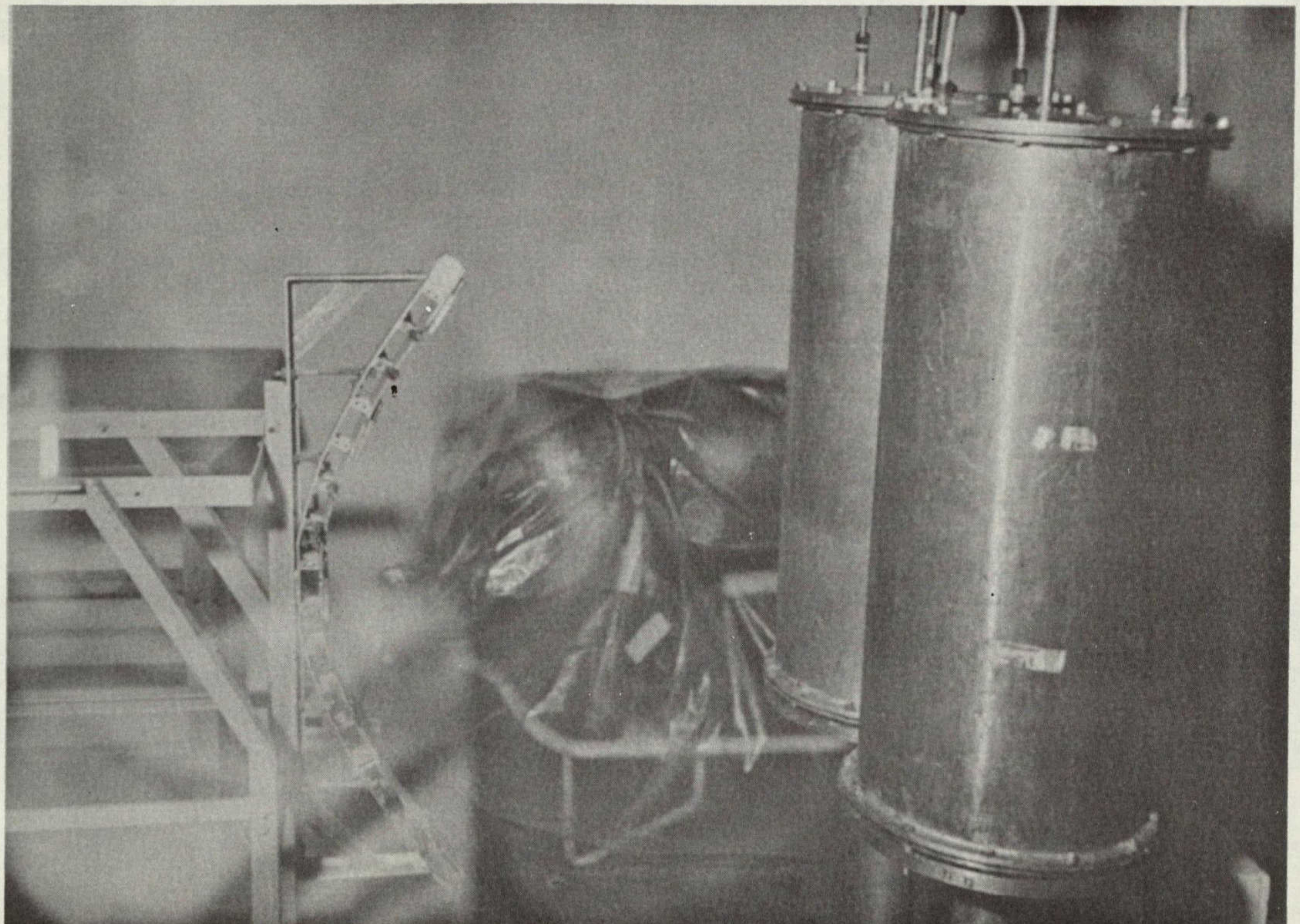

FIGURE 1. IRRADIATION CONFIGURATION

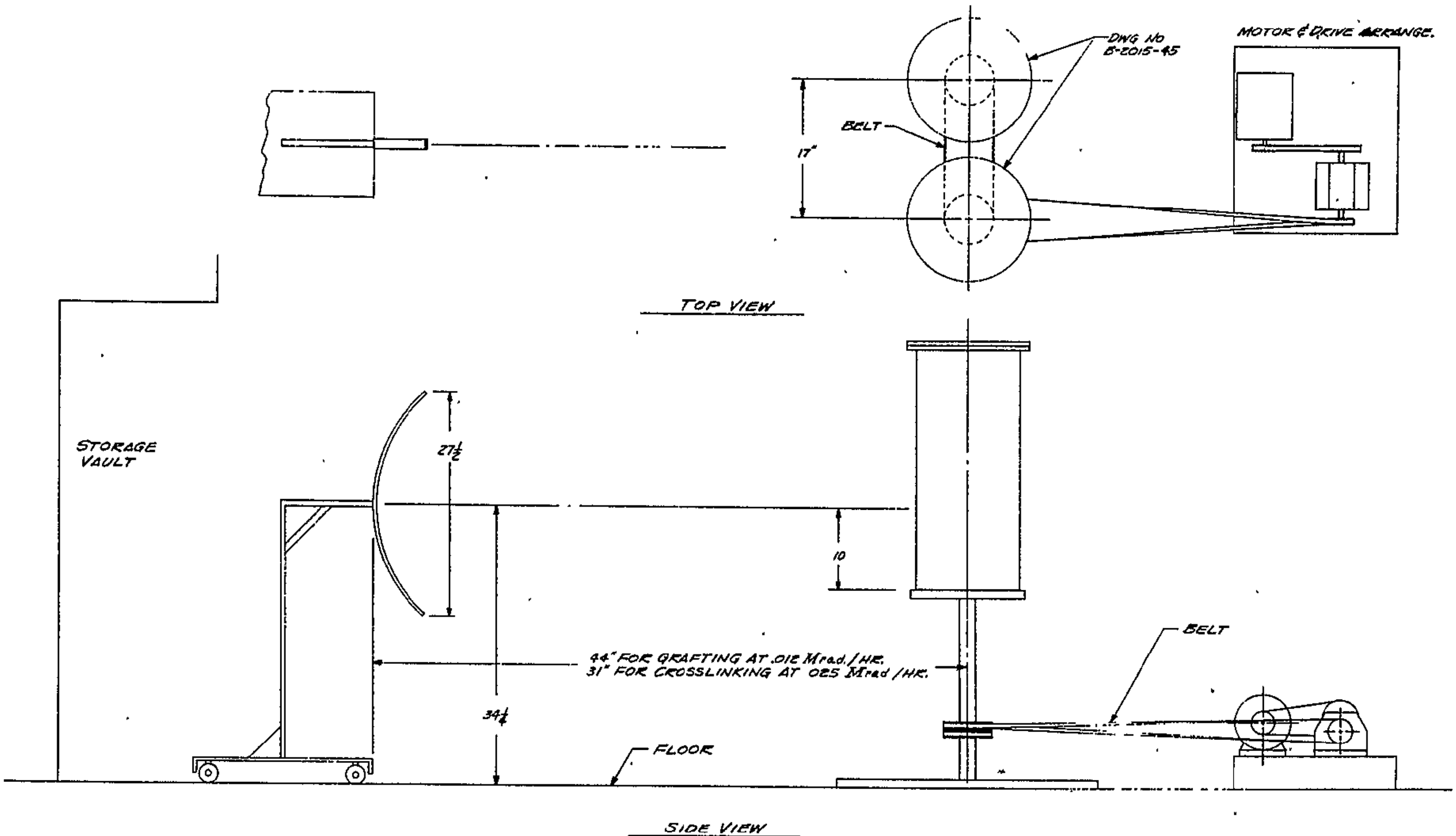

FIGURE 2. IRRADIATION CONFIGURATION

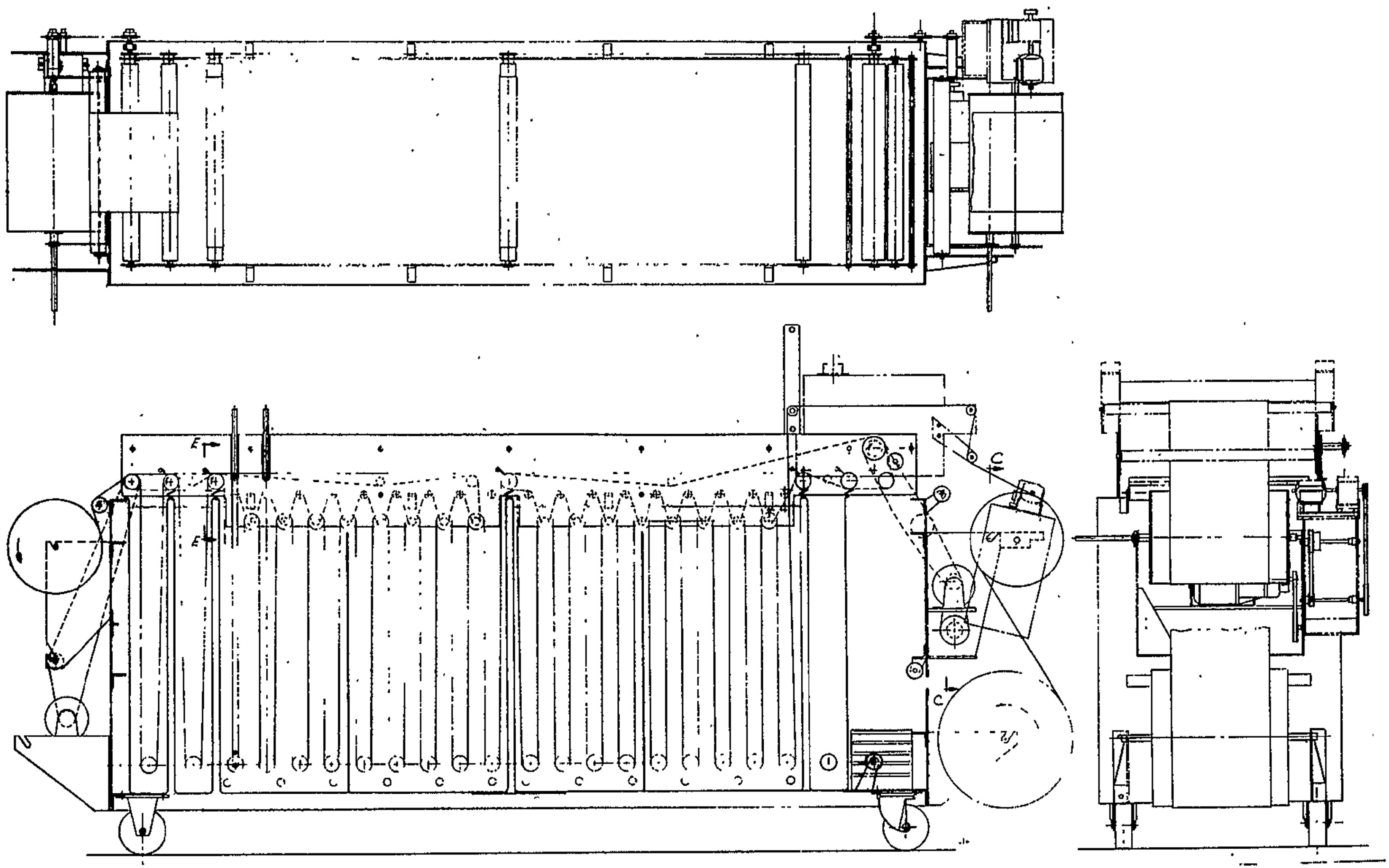

FIGURE 3. FILM PROCESSING MACHINE