DEVELOPMENT OF BATTERY SEPARATOR MATERIAL PROCESS

by

L. M. Adams
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INTERIM REPORT

SwRI Project No. 01-2015
Contract No. 951718

Prepared for

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

May 29, 1969

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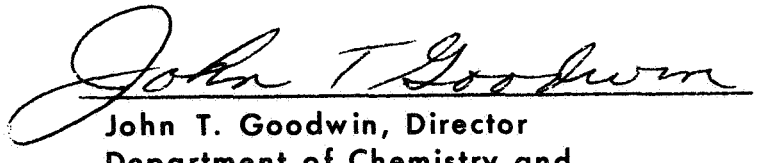

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I. SUMMARY

The dose rate and total dose for irradiation grafting of polyethylene film with acrylic acid have been standardized at 0.012 Mrad/hr and 0.815 Mrad, respectively.

High carbon tetrachloride (chain terminator) concentrations in the acrylic acid grafting solution cause a decrease in uniformity of the properties of battery separator material on sterilization. Of the chain terminators evaluated, carbon tetrachloride, acetone, sulfur, and carbon disulfide appear to be the most satisfactory.

The replacement of benzene in the grafting solution with toluene or xylene results in poorly grafted films.

Polyethylene film can be grafted with acrylic acid using aqueous grafting solutions if a gelation inhibitor is employed.

Polyethylene film has been grafted in 600-foot lengths. However, 100-foot lengths are more convenient to handle in the laboratory.

The temperature during neutralization and washing has an effect on the properties of acrylic acid grafted polyethylene film. On treatment at 97°C, the product has a lower electrical resistance than when treated at 80°C.

Dow 400 polyethylene film yields much more uniformly grafted film than the previously supplied polyethylene film. It is also possible

to obtain a good product using lower concentrations of acrylic acid in the grafting solution with the new film.

Cotton cheesecloth and coarse nylon mesh are superior to paper toweling as an interlayer material. More uniform grafting and lower concentrations of acrylic acid can be used in the grafting solution with these materials.

A machine has been constructed for neutralizing, washing, and drying long lengths of grafted polyethylene film. Operation of the machine has been satisfactory.

The potassium content of the neutralized and washed grafted film decreases with increase of carbon tetrachloride in the grafting solution. It increases on soaking in forty percent potassium hydroxide solution and increases further on sterilization in forty percent potassium hydroxide solution.

The quantity of divinylbenzene in the battery separator material increases with an increase in concentration in the crosslinking solution.

The results of crosslinking acrylic acid grafted polyethylene film in an electron beam are inconclusive.

Polyethylene film has been grafted with methacrylic acid, acrylic acid/N-vinylpyrrolidone mixtures, acrylic acid/vinylpyridine mixtures, and acrylic acid/sodium vinylsulfonate mixtures.

Equipment for ion migration studies has been assembled.

II. INTRODUCTION

A sterilizable battery separator material for silver-zinc batteries can be prepared by grafting polyethylene film with acrylic acid and subsequently crosslinking with divinylbenzene. A study of the processing parameters is being conducted to determine optimum conditions for preparing the best battery separator material having good uniformity and a low electrical resistance.

Other factors such as the monomer or monomers used for grafting the polyethylene film, method of crosslinking the grafted film, and solvents used in the grafting and crosslinking solutions are also being studied.

Studies for scale-up procedures for the production of quantities of long lengths of battery separator material are being made so that sufficient material can be supplied for the battery programs. The scale-up includes fabrication of reactors for grafting and crosslinking large quantities of polyethylene film and construction of equipment for neutralizing, washing and drying large quantities of material.

This report covers the period of August 15, 1967 through March 31, 1969.

III. EXPERIMENTAL

A. Grafting of Low-Density Polyethylene Film

1. Effect of Irradiation Dose Rate and Total Dose

The effects of irradiation dose rate and total dose used for grafting low-density polyethylene film with acrylic acid are discussed in the August 25, 1967 Interim Report. It was reported that dose rates of 0.0125 to 0.021 Mrad/hr and total doses of 0.671 to 1.7 Mrad had little if any effect on the properties of the grafted film produced. Lowering the dose rate to 0.006 Mrad/hr and the total dose to 0.408 Mrad gives borderline results in some cases (Table 1); in all cases, the outer portion of the roll did not graft.

A higher total dose at this low dose rate would probably be satisfactory, but irradiation time would become excessive. Two of the samples were crosslinked with divinylbenzene and analyzed (Table 2). The grafting procedure has been standardized at a dose rate of 0.012 Mrad/hr and a total dose of 0.815 Mrad.

2. Terminator Studies

On varying the carbon tetrachloride (chain terminator) from 0 to 10 weight percent of the grafting solution while maintaining the acrylic acid at 25 weight percent, the average electrical resistance of the unsterilized films (Table 3) was the same. However, there was a decrease in uniformity of the electrical resistances throughout the roll as the tetrachloride concentration was increased. On sterilization, the

film prepared with 10 weight percent carbon tetrachloride increased appreciably in average resistance in contrast to a decrease for films prepared with the lower concentrations. Also, the uniformity of the electrical resistance values of the film prepared with 10 weight percent carbon tetrachloride decreased considerably on sterilization, while the electrical resistance values of the films prepared with lower concentrations exhibited only slight changes.

The only significant effect on dimensional changes with carbon tetrachloride concentration occurred in the length on sterilization. The increase in length varied inversely with carbon tetrachloride concentration in the grafting solution.

Other chain terminators (Table 4) that were evaluated are carbon tetrabromide, acetone, carbon disulfide, sulfur, and dodecyl mercaptan. Carbon tetrabromide is known to be a more effective terminator than carbon tetrachloride, and concentrations as low as one weight percent in the acrylic acid grafting solution are excessive. Grafted films prepared with dodecyl mercaptan were not wet well by the 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 without terminator were crosslinked. From the limited data obtained (Table 5), carbon disulfide produced the most uniform film as determined by the electrical

resistance. However, the film prepared with the acetone chain terminator was almost as uniform after sterilization. All three are acceptable. The material prepared without chain terminator was also satisfactory.

3. Effect of Organic Solvents

None of the organic solvent systems evaluated improved the grafting of polyethylene over that obtained with benzene as the solvent (Table 6). Toluene produced poorly grafted films. The replacement of part of the toluene with methanol improved grafting, but the mixture was not as satisfactory as benzene.

The replacement of part of the benzene with methanol (Table 7) produced a grafted and crosslinked film which increased in width and length on sterilization to a greater extent than did films grafted in benzene with an equal carbon tetrachloride concentration.

4. Aqueous Acrylic Acid Grafting Solutions

Polyethylene film is readily radiation grafted in aqueous acrylic acid solutions to yield a material 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 the grafted film. It was found that the addition of ferrous sulfate to the aqueous acrylic acid solution prevents gelation, and the grafted film can 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 8).

After sterilization, some of the specimens had an electrical resistance less than 0.5 milliohm-inch^2. The physical properties of the grafted film are good.

It has also been found that potassium ferrocyanide and potassium ferricyanide will prevent gelation of the aqueous acrylic acid grafting solution. These materials have an advantage over ferrous sulfate as they can be completely washed from the grafted film. This eliminates the possibility of iron contamination of the product. The grafted films have uniform and very low electrical resistance (Table 9). These samples were overgrafted as they tore into 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 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.

5. Scale-up Studies

In preparing rolls of polyethylene film for grafting, the film is backed with a porous material and is 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. The initial work was done with paper toweling obtained from two different suppliers.

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:

a. 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.

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

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

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

e. The grafting solution was added.

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

g. The reactor was then rotated and irradiated at a dose rate of 0.22 Mrad/hr for a total dose of 1.530 Mrads 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 10) 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 No. 118 was also recovered. Crosslinking of these grafted materials with divinylbenzene produced battery separator material of low electrical resistance (Tables 11-15). 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 No. 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. Other work conducted with 100-foot rolls is discussed in several of the following sections.

6. Processing Conditions

To determine the effect of neutralization and washing temperature on battery separator properties, preliminary experiments were conducted with separator material on hand (Sample No. 187, grafted and crosslinked, Tables 1 and 2). Portions of the separator material were treated for one hour in boiling water, boiling five percent aqueous potassium hydroxide solution, boiling forty percent aqueous potassium hydroxide solution, or five percent aqueous potassium hydroxide solution at 80°C. The treated samples were washed with distilled water at room temperature and air dried. All of the procedures, with the exception of the one using five percent aqueous potassium hydroxide at 80°C, produced wet materials that expanded in all directions over the dry dimensions on sterilization at 135°C in 40 percent aqueous potassium hydroxide solution (Table 16).

Additional samples were treated with boiling distilled water, boiling five percent potassium hydroxide solution, or five percent potassium hydroxide solution at 80°C. After washing and drying the treated samples, they were heated under sterilization conditions (135°C for 64 hours) in the respective treating liquids. As expected, these samples swelled more (Table 17) than they did when treated

in forty percent potassium hydroxide solution. The sample sterilized in distilled water surprisingly withstood the treatment, and the electrical resistance decreased to 2 milliohm-inch^2.

One hundred-foot rolls of polyethylene film were grafted in two large reactors with 50 liters of grafting solution in each reactor. 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. The remainder were neutralized and washed at 80°C. The grafted and washed film was then crosslinked with divinylbenzene.

Data from the analysis of these samples are presented in Table 18. 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 is encountered periodically, and it is found that these areas of high resistance are difficult to neutralize. This phenomenon is discussed further under the section on polyethylene film studies.

The specimens taken from the separator material processed at 80°C exhibited a slight decrease in length from the dry length on

sterilization, while the material processed at the boiling points of the processing solutions exhibited an increase in length. The effect of neutralization and washing temperature is discussed further in later sections.

7. Polyethylene Film Studies

During the optimization of the acrylic acid grafting procedure, polyethylene film (1 mil) supplied by the Jet Propulsion Laboratory from stock on hand was utilized. The results obtained varied from roll to roll and throughout each roll. This was further illustrated during scale-up studies (Tables 19-21).

Because of the lack of uniformity obtained with the above film, other polyethylene films were examined. In one run made in the large reactor, five rolls of JPL polyethylene film and one roll of film prepared from Dow polyethylene 110E were grafted simultaneously. The Dow film grafted very uniformly while the grafted JPL film was not uniform (Table 22).

Films prepared from Dow polyethylene resins 710M, 110E, 510M, and 560E were obtained from the Freeport Laboratories of the Dow Chemical Company. These films were free of additives. Results obtained on grafting 30-foot rolls of these materials with acrylic acid are presented in Table 23 and compared with one run from the film which was used in most of the studies up to this time. The electrical resistance of all of the grafted aforementioned Dow films was uniformly low.

A sample of Dow 400 polyethylene film from a production run was obtained from the Dow Findlay, Ohio plant. This film was 2.0 mils thick and contained 1000 ppm of calcium carbonate as an additive. This material also produced a uniform film of low electrical resistance on grafting with acrylic acid (Sample Nos. 282 throug 285, Table 23).

Data for grafted and crosslinked Dow films are presented in Table 24. The electrical resistances in all cases are uniformly low before and after sterilization. In all cases, the length after sterilization (wet with 40% KOH) is greater than the dry length before sterilization whether the grafted film was neutralized and washed at 80°C or 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.

Previous work has shown that lowering the acrylic acid concentration in the grafting solution resulted in very nonuniform grafting with the JPL film. Lowering the concentration of the acrylic acid in the grafting solution to as low as 15 weight percent with most of the Dow polyethylene films resulted in satisfactory grafting. Dow 710M film grafted satisfactorily at 20 and 15 weight percent acrylic acid (Table 25). With 15 weight percent acrylic acid in the grafting solution, the grafted Dow 110E film was borderline. Further studies with the various polyethylene films are discussed in the section on interlayer material studies.

8. Interlayer Material Studies

A study of various interlayer materials indicates that cloth is superior to the paper toweling materials evaluated. Of the materials evaluated, two of the best from a standpoint of effect on grafting and convenience in handling 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. Also, there is less chance of impurities which might affect the grafting of the polyethylene film. More uniform and lower electrical resistances were obtained using cheesecloth and nylon mesh (Tables 26 and 27).

It was also possible to lower the acrylic acid concentration of the grafting solution to as low as 15 weight percent and still obtain uniformly grafted film when using the cheesecloth interlayer and the JPL film (Table 28). At 10 weight percent acylic acid, nonuniform grafting occurs.

Because of the excellent results obtained with the 2-mil Dow 400 polyethylene film, a quantity of 1-mil Dow 400 polyethylene film was purchased by JPL from Dow's Fresno, California plant. This material is being used in all future battery separator preparation. Using this new material with a cheesecloth interlayer, satisfactory grafting is obtained with acrylic acid concentrations as low as 10 weight percent in the grafting solution (Table 29). The effect of neutralization and washing at 80°C and 97°C is not as pronounced as with the previous JPL film.

The first scale-up run using cheesecloth interlayer was made with the old JPL film and a grafting solution containing 25 weight percent acrylic acid. The grafted film had uniformly low electrical resistance (Table 30) throughout the roll. Although a cooling coil was used in the grafting solution, the film was highly grafted, and sections were white from homopolymer that could not be washed from the film. The grafted film is always clear when prepared in 30-foot rolls in individual glass reactors. It is believed that the difference is due to the higher maximum temperature occurring in the large reactor.

On lowering the acrylic acid concentration to 15 weight percent and using the cooling coil in the grafting solution, the grafted film had excellent clarity, but the electrical resistance was not very uniform throughout the roll (Table 31). When the cooling coil was not used, a higher temperature was obtained in the reactor, but there was little if any improvement in the uniformity of the grafted film (Table 32).

A comparison between Dow 560E film and the JPL film was made by grafting rolls of both simultaneously in the same reactor. The grafted JPL film was much more uniform than from the previous runs, but the grafted Dow 560E film had lower and more uniform electrical resistance throughout the roll (Table 33).

On receipt of the new JPL film (Dow 400), runs under similar conditions were made with the new film. Uniformly low electrical resistances were obtained for the grafted film with and without a cooling coil in the grafting solution (Tables 34 and 35, respectively).

9. Film Washing Machine

All of the previously grafted film was processed in a small stainless steel kettle in the laboratory. As long lengths of grafted film are difficult to handle in the laboratory, a machine has been constructed to neutralize, wash, and dry long lengths of film. The grafted film passes through a hot five percent potassium hydroxide solution followed by hot deionized water. On leaving the water bath, the film is dried by passing through a high velocity air stream and rolled up with a paper interlayer for crosslinking. With a film movement of one foot per minute, the residence time for the film in each bath is approximately 45 minutes.

The effect of prolonged times in the bath was found to be slight (Table 36). This was determined by running a portion of a 100-foot roll through the machine at a rate such that a residence time of 31 minutes was obtained for each bath. The machine was then shut down and allowed to cool over the weekend. On reheating the baths, the remainder of the film was processed.

The data from a typical complete run are presented in Table 37. The residence time of the film in each bath was approximately 45 minutes. The electrical resistance values are uniformly low.

10. Potassium Content of Neutralized Grafted Film

The potassium content of grafted polyethylene film which had been neutralized and washed was determined by boiling 0.2 gram of the film in 50 milliliters of four percent hydrochloric acid, rinsing the film with 50 milliliters of four percent hydrochloric acid, and then rinsing with

distilled water until the combined acid and water 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 before analysis. This is illustrated by the data presented in Table 38.

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

The potassium content of neutralized grafted film is much higher when no terminator or a low concentration of terminator is used in the grafting solution than when higher chain terminator concentrations are used (Table 39).

11. Crosslinking Studies-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 with nitrogen or oxygen atmospheres. This was determined from ultraviolet spectra of the crosslinked films.

However, there is no proof that the degree of crosslinking is increased, as the ultraviolet spectra indicate only the aromatic content of the films.

On increasing the divinylbenzene content of the crosslinking solution from one to sixteen volume percent (Table 40), 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 and remain on the film. These deposits are very difficult to remove.

12. Crosslinking Studies-Electron Beam

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 A. Properties of the crosslinked material are presented in Table 41 along with the uncrosslinked film and the film crosslinked in the standard one 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 total doses above this value, the resistance increased on sterilization.

Increase in film thickness on sterilization varied inversely with total dose, and at the highest total dose, no increase occurred.

There was no correlation between total dose and other dimensional changes. The elongation of the film was lowest 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 in nitrogen 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 increased.

13. Other Crosslinking Studies

Divinyl sulfone was used to crosslink acrylic acid grafted polyethylene film. The infrared spectrum of the crosslinked film indicated the presence of the sulfone group. On sterilization of this film in a stainless steel chamber, a large amount of black material which could not be removed deposited on some of the resistance specimens. It is believed that this deposit is partially responsible for the decrease in uniformity of the electrical resistance of the sterilized specimens (Table 42).

14. Additives for Grafting Solutions

A considerable amount of homopolymer precipitates during the grafting of polyethylene film with acrylic acid. A number of metal salts and organic materials were evaluated in an attempt to reduce or eliminate homopolymer precipitation. Of the metal salts evaluated (Table 43), only iron, cerium, cobalt, and nickel salts accomplished the

desired results. It is known that iron is 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 this extra effort would 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 homopolymer precipitation. 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.

15. Grafting with Other Monomers

Several different types of polyethylene film have been grafted with methacrylic acid. The initial work was conducted with the JPL polyethylene film. The grafted and crosslinked film was not uniform when carbon tetrachloride was present in the grafting solution, and the resistance increased on sterilization. Grafting without terminator yielded a uniformly grafted film of low resistance which decreased considerably on sterilization. The comparison is given in Table 45.

Data for grafting other polyethylene films with methacrylic acid are presented in Table 46.

The new Dow 400 polyethylene film can be grafted with methacrylic acid concentrations as low as 10 weight percent to yield a grafted film of uniformly low electrical resistance (Table 47).

Polyethylene film grafted with vinylpyrrolidone was of high resistance (greater than 3000 milliohm-inch^2), but grafting with acrylic acid/vinylpyrrolidone mixtures resulted in products having uniformly low resistance (Table 48).

When polyethylene film is grafted with mixtures of acrylic acid and vinylpyridine, the product is of very low resistance (Table 49). One sample was crosslinked with one volume percent divinylbenzene (Table 50). This material expands considerably more than acrylic acid grafted films when wet with water or potassium hydroxide solutions.

Polyethylene film can be grafted with mixtures of acrylic acid and sodium vinylsulfonate to yield grafted films of very low and uniform electrical resistances (Table 51). The infrared spectrum indicates the presence of sulfonic acid groups in the grafted film.

16. Ion Migration Studies

Equipment has been constructed for the determination of silver ion, zincate ion, and hydroxyl ion migration. This equipment will be used to evaluate the various battery separator membranes prepared.

IV. CONCLUSIONS

Dow 400 polyethylene film is an excellent base stock for the preparation of sterilizable battery separator material.

Cheesecloth and nylon mesh are superior to paper toweling for use as the interlayer material.

Aqueous grafting solutions appear promising if the degree of grafting can be decreased.

Battery separator materials can be prepared by cografting polyethylene film with mixtures of acryic acid and N-vinylpyrrolidone, 2-vinylpyridine, 4-vinylpyridine, or sodium vinylsulfonate.

A dose rate of 0.012 Mrad/hr and a total dose of 0.815 Mrad appear to be satisfactory for the grafting procedure.

Acetone, sulfur, carbon tetrachloride, and carbon disulfide are good chain terminators for the acrylic acid grafting of polyethylene film.

Large quantities of battery separator material can be prepared in the equipment constructed for scale-up work.

Homopolymer precipitation can be minimized or eliminated by the addition of certain metal salts or organic compounds to the acrylic grafting solution.

V. FUTURE WORK

Preparation of large quantities of battery separator material will be continued.

Optimization of grafting procedures using the new polyethylene film will be continued.

Further studies will be conducted with other grafting and cross-linking monomers.

Ion migration studies will be conducted.

TABLE 1. EFFECT OF LOW DOSE RATE AND LOW TOTAL DOSE GRAFTED ONLY

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

Sample No.	Resistance, milliohm-inch^2	
	Average	Range
185	33	14-62
186	45	11-117
187	13	10-18
188	20	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.

TABLE 2. EFFECT OF LOW DOSE RATE AND LOW TOTAL DOSE ON GRAFTING GRAFTED AND CROSSLINKED

A. Electrical Properties

Sample No.	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)
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, mil			Dimensional Changes(3), % Width		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	Experimental Conditions for Crosslinking	
1 vol % Divinylbenzene	Dose Rate:	0.025 Mrad/hr
1 vol % Benzene	Total Dose:	0.55 Mrad
98 vol % Methanol	Atmosphere:	Nitrogen
	Temperature:	Ambient

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

TABLE 3. EFFECT OF CARBON TETRACHLORIDE CONCENTRATION IN GRAFTING SOLUTION GRAFTED ONLY

Grafting Solution Composition

25 wt % Acrylic acid
75-65 wt % Benzene
0-10 wt % Carbon tetrachloride

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.	Carbon Tetrachloride, %	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)
123	0	14	11	2.0	1.8	14.5	16.9
124	2.5	14	10	3.6	2.5	26.4	25.0
127	10	14	22	5.5	25.6	38.1	115.6

B. Physical Properties

Sample No.	Thickness, mil			Dimensional Changes(3), % Width		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)		
123	1.5	1.7	1.8	11.0	10.0	10.5	8.6	1386	>100
124	1.2	1.4	1.7	12.1	10.1	9.0	4.0	1298	>100
127	1.4	1.5	1.9	11.0	9.0	9.0	0	1693	>100

Note

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

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

TABLE 4. EFFECT OF VARIOUS CHAIN TERMINATORS GRAFTED ONLY

Grafting Solution Composition

25 wt % Acrylic acid
75 wt % 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, hr	Time to Maximum Temperature, hr	Maximum Temperature, °F
138[1]	Carbon tetrabromide, 5.0	2	>3000	78	No exotherm		
		16	>3000				
		26	>3000				
148	Carbon tetrabromide, 1.0	6	41, 41	77	7.0	9.0	86
		16	175, 107				
		26	220, 346				
213[2]	Acetone, 1.0	15	11, 13	87	3.0	4.5	104
		25	25, 24				
		30	13, 13				
214[3]	Acetone, 2.5	6	22, 22	87	4.0	5.0	106
		15	35, 47				
		25	23, 29				
178[4]		6	9, 15	74	3.0	4.5	90
		16	15, 15				
		26	15, 13				
215[5]	Carbon disulfide, 2.5	6	9, 9	87	3.0	4.0	102
		15	14, 16				
		25	15, 16				
197[6]	Carbon disulfide, 5.0	5	9, 9	80	7.0	9.0	87
		15	11, 10				
		25	13, 12				

TABLE 4 (Continued)

Sample No.	Terminator and Concentration, wt %	Footage	Resistance, milliohm-inch^2	Grafting Temperature, °F	Time to Exotherm, hr	Time to Maximum Temperature, hr	Maximum Temperature, °F
183[7]	Benzene saturated with sulfur	6 16 26	15, 10 18, 18 21, 20	74	2.0	5.0	85
199[8]	CCl_4 (5.0) plus benzene saturated with sulfur	10 15 25	3000, 2597 50, 45 149, 26	80	7.0	9.0	85
149[9]	Dodecyl mercaptan 1.0	6 16	56, 61 29	77	2.0	4.0	89
139[10]	Dodecyl mercaptan 5.0	random	9 11	78	0.5	3.5	90
150[11]	Dodecyl mercaptan 10.0	4 14 24	3000 3000 3000	77	0.5	2.5	104

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

(1) IR scan indicates some grafting occurred.
(2) No homopolymer precipitated. Outer twelve feet did not graft.
(3) Outer five feet did not graft.
(4) Small amount of homopolymer precipitated. Intermittent grafting on outer two feet.
(5) Intermittent grafting on outer five feet.
(6) Outer three feet and top edge did not graft.
(7) No homopolymer precipitated. Intermittent grafting on outer two feet.
(8) No homopolymer precipitated. Intermittent grafting on outer ten feet.
(9) Outer six feet did not graft.
(10) Film tore during processing. Random sampling.
(11) KOH solution did not wet film.

TABLE 5. EFFECT OF VARIOUS CHAIN TERMINATORS GRAFTED AND CROSSLINKED

Crosslinking Solution Composition

1 vol % Divinylbenzene
1 vol % Benzene
98 vol % Methanol

Experimental Conditions for Crosslinking

Dose Rate:	0.025 Mrad/hr
Total Dose:	0.55 Mrad
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(4)	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(4)	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.

(1) Before sterilization - wet with 40% KOH.
(2) After sterilization - wet with 40% KOH.
(3) Change from dry dimensions.
(4) See Table 3 for grafting conditions.

TABLE 6. EFFECT OF SOLVENTS
GRAFTED ONLY

Grafting Solution Composition

25 wt % Acrylic Acid
75 wt % As shown in table

Experimental Conditions for Grafting

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

Sample No.	Solvent wt %	Carbon Tetrachloride, wt %	Average Resistance milliohm-inch^2	Standard Deviation	Standard Deviation % of Average
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.

TABLE 7. EFFECT OF SOLVENTS GRAFTED AND CROSSLINKED

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

A. Electrical Properties

Sample No.	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)
134	10	5	2.3	1.8	22.3	39.3

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)		
134	1.2	1.5	1.9	13.4	18.6	9.6	5.6	1390	>100

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

TABLE 8. GRAFTING WITH AQUEOUS ACRYLIC ACID GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
75 wt % Distilled water plus 5.2 g $FeSO_4 \cdot 7H_2O$ per 2 kilo of solution	Total Dose:	0.815 Mrad
	Temperature:	82°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

A. Electrical Properties

Sample No.	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)
125	16	3	1.6	1.6	10	53.3

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)		
125	1.2	1.4	1.6	11.0	25.0	10.0	3.5	1361	>100

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

TABLE 9. GRAFTING WITH AQUEOUS ACRYLIC ACID
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
As listed.	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, (grams)(1)	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.

(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 10. EXOTHERM AND ROLL CONFIGURATION

Sample No.	Initial Temp., °F	Time to Exotherm, hr	Time to Maximum Exotherm, hr	Maximum Temp., °F	Roll Configuration	Comments
114	72	2	12	149	600-foot roll 1/4" pipe core	Air conditioner on in cell. Recovered 120 ft of film.
115	61	4	7	142	600-foot roll 1/4" pipe core	Air conditioner on in cell. Recovered 220 ft of film.
117	73	6	10	107	600-foot roll 1/4" pipe core	Air conditioner on in cell. Recovered 310 ft of film.
118	61	4	30	149	600-foot roll 6" core	Air conditioner on in cell; 550 ft of film recovered.
119	66	4	18	136	600-foot 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 foot rolls on 1/4" pipe core	Air conditioner on in cell; all film recovered.
152	72	2	52	117	600-foot 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-foot 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 11. SAMPLE NO. 118
GRAFTED AND CROSSLINKED

Grafting Solution Composition

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Crosslinking Solution Composition

1.0 vol % Divinylbenzene
1.0 vol % Benzene
98.0 vol % 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, hr: 4.0
Time to maximum exotherm, hr: 30.0
Maximum temperature, °F: 149

Neutralization and Washing Temperature

80°C

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

TABLE 11 (Continued)

Results	
Footage	Resistance, milliohm-inch^2
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 12. SAMPLE NO. 119
GRAFTED AND CROSSLINKED

Grafting Solution Composition

25 wt % Acrylic acid
70 wt % Benzene
5 wt % 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

	Resistance, milliohm-inch^2		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]
119-300	10	4						
	15	19						
	19	25						
	14	17						
	12	12						
	15	19	14	16	3.1	7.2	21.6	45.1

TABLE 12 (Continued)

B. Physical Properties

Sample No.	Thickness, mil			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]		
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.

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

TABLE 13. SAMPLE NO. 152
GRAFTED AND CROSSLINKED

Grafting Solution Composition

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Crosslinking Solution Composition

1.0 vol % Divinylbenzene
1.0 vol % Benzene
98.0 vol % 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, hr:	2.0
Time to maximum exotherm, hr:	52.0
Maximum temperature, °F:	117

Neutralization and Washing Temperature

80°C

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 14. SAMPLE NO. 153
GRAFTED AND CROSSLINKED

Grafting Solution Composition

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Crosslinking Solution Composition

1.0 vol % Divinylbenzene
1.0 vol % Benzene
98.0 vol % 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, hr:	4.0
Time to maximum exotherm, hr:	58.0
Maximum temperature, °F:	97

Neutralization and Washing Temperature

80°F

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 15. SAMPLE NO. 163
GRAFTED AND CROSSLINKED

Grafting Solution Composition

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Crosslinking Solution Composition

1.0 vol % Divinylbenzene
1.0 vol % Benzene
98.0 vol % 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, hr:	6.0
Time to maximum exotherm, hr:	20.0
Maximum temperature, °F:	127

Neutralization and Washing Temperature

80°C

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 16. TREATMENT OF SAMPLE NO. 187
GRAFTED AND CROSSLINKED

Treatment for One Hour	Thickness, mil			Dimensional Changes[3], %				Resistance, milliohm-inch^2
				Width		Length		
	Dry	B.S.[1]	A.S.[2]	B.S.[1]	A.S.[2]	B.S.[1]	A.S.[2]	A.S.[2]
Boiling water	1.4	1.5	1.9	8.3	4.2	10.5	2.1	10
	1.3	1.4	1.7	12.2	8.2	12.2	8.9	
5% KOH at	1.2	1.3	1.7	6.4	6.4	5.0	-1.0	15
80°C	1.4	1.4	1.9	4.1	2.0	5.0	-2.0	
5% KOH at	1.4	1.5	1.8	8.0	2.0	7.1	3.1	13
B.P. (97°C)	1.4	1.5	1.9	8.0	2.0	8.2	1.0	
40% KOH at	1.8	1.8	2.0	8.5	6.4	4.2	4.2	13
B.P. (121°C)	1.8	1.9	2.0	6.2	6.2	5.2	5.2	

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

TABLE 17. TREATMENT OF SAMPLE NO. 187 GRAFTED AND CROSSLINKED

Treatment for One Hour	Thickness, mil				Dimensional Changes, %						Resistance[4] milliohm-inch^2 After Sterilization in Treating Solution
					Width			Length			
	Dry	(1)	(2)	(3)	(1)	(2)	(3)	(1)	(2)	(3)	
Boiling water	1.4	1.8	1.8	2.1	18.0	32.0	58.0	18.0	20.0	28.0	2
	1.4	1.8	1.8	2.1	24.5	32.6	57.2	16.0	20.0	28.0	
5% KOH at	1.3	1.5	1.5	1.7	18.4	18.4	28.6	17.8	18.8	15.8	6
80°C	1.4	1.5	1.4	1.9	20.4	20.4	28.6	16.0	15.0	11.0	
5% KOH at	1.4	1.5	1.5	1.9	18.4	20.4	26.5	17.0	17.0	12.0	7
B.P. (97°C)	1.4	1.5	1.5	1.9	18.4	18.4	24.5	17.0	17.0	11.0	

(1) Wet with treating solution.
(2) Wet with treating solution after treatment.
(3) Wet with treating solution after sterilization at 135°C for 64 hours in treating solution.
(4) Measured in 40% KOH solution.

TABLE 18. EFFECT OF NEUTRALIZATION AND WASHING TEMPERATURE (Sample Nos. 231 and 232) GRAFTED AND CROSSLINKED

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet
Crosslinking Solution Composition	**Experimental Conditions for Crosslinking**	
1.0 vol % Divinylbenzene	Dose Rate:	0.025 Mrad/hr
1.0 vol % Benzene	Total Dose:	0.550 Mrad
98.0 vol % Methanol	Atmosphere:	Nitrogen

A. Electrical Properties

Sample No.	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)
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, mil			Dimensional Changes(5), % Width		Dimensional Changes(5), % 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)		
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.

(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 19. SAMPLE NO. 249
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	61°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (6 rolls)

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	1-249	6-249
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, hr:	3.0
Time to maximum exotherm, hr:	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 20. SAMPLE NOS. 251 AND 252 GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	69°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (6 rolls per reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2		
	4-251	5-251	1-252
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 21. SAMPLE NOS. 253 AND 254
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	74°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (6 rolls in each reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	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

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 22. SAMPLE NO. 265
GRAFTED ONLY

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

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	1-265	2-265
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, hr:	4.0
Time to maximum exotherm, hr:	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 23. GRAFTING OF VARIOUS POLYETHYLENE FILMS IN 30-FOOT ROLLS
GRAFTED ONLY

Grafting Solution Composition

25 wt % Acrylic acid
70 wt % Benzene
5 wt % 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] 15[4] 25[4]	7, 7 60, 48 12, 36	89	1.0	5.5	118
193	Dow 710M - 0.5 mil	5[3] 15[3] 25[3]	8, 6 7, 4 6, 4	80	7.0	9.0	89
194	Dow 710M - 1.0 mil	5[3] 15[3] 25[3]	12, 11 11, 13 10, 9	80	7.0	9.0	88
233	Dow 710M - 1.0 mil	5[3] 15[3] 16[4] 28[4]	7, 4 5, 5 3, 2 3, 1	89	2.0	3.5	118
234	Dow 710M - 1.0 mil	12[3] 23[3] 4[4] 11[4]	11, 11 8, 7 4, 4 3, 5	89	3.0	5.0	118
257	Dow 110E - 1.5 mil	5[3] 15[3] 25[3]	14, 16 16, 18 16, 18	77	2.5	3.5	100
258	Dow 110E - 1.5 mil	5[4] 15[4] 25[4]	22, 22 19, 17 16, 16	77	2.5	4.0	121

TABLE 23 (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

TABLE 23 (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
293	Dow 560E - 1.0 mil	5[4]	10, 10	72	2.0	3.5	77
		15[4]	8, 8				
		25[4]	8, 8				
294	Dow 560E - 1.0 mil	5[4]	9, 10	72	2.5	4.0	91
		15[4]	8, 8				
		25[4]	9, 8				

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 24. GRAFTING OF VARIOUS POLYETHYLENE FILMS IN 30-FOOT ROLLS GRAFTED AND CROSSLINKED

Grafting Solution Composition

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Grafting Conditions

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

Crosslinking Conditions

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

Crosslinking Solutions

1 vol % Divinylbenzene
1 vol % Benzene
98 vol % 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 24 (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]		
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

(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 25. EFFECT OF ACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION GRAFTED ONLY

Sample No.	Film	Acrylic Acid Concentration, wt %	Footage[1]	Resistance, milliohm-inch^2	Grafting Temperature, °F[2]	Time to Exotherm, hr	Time to Maximum Temp., hr	Maximum Temp., °F
235[3]	Dow 710M	20	5	6, 4	89	3.0	6.0	92
			15	6, 5				
			25	5, 6				
236[3]	Dow 710M	15	5	8, 8	89	3.0	6.0	95
			15	7, 10				
			25	6, 5				
260[3]	Dow 110E	20	5	25, 27	77	2.5	4.0	100
			15	18, 23				
			25	36, 30				
261[3]	Dow 110E	20	5	24, 20	77	2.5	4.0	100
			15	22, 22				
			25	24, 27				
262[3]	Dow 110E	15	5	39, 41	77	2.5	4.0	98
			15	37, 45				
			25	39, 35				
263[3]	Dow 110E	15	5	118, 278	77	2.5	4.0	97
			15	45, 49				
			25	39, 33				

Note: Nitrogen Atmosphere
Dose Rate: 0.012 Mrad/hr
Total Dose: 0.815 Mrad
St. Regis paper interlayer

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

TABLE 26. SAMPLE NO. 270
EFFECT OF CHEESECLOTH INTERLAYER
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % 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 27. SAMPLE NOS. 275 AND 276
EFFECT OF NYLON INTERLAYER
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % 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 nylon mesh interlayer. Neutralized and washed at 97°C.

TABLE 28. SAMPLE NOS. 271-273
EFFECT OF CHEESECLOTH INTERLAYER
GRAFTED ONLY

Grafting Solution Composition

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
Poll Length:	25 feet

Electrical Properties

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

Exotherm Data

Time to exotherm, hr:	3.5	3.0	3.0
Time to maximum exotherm, hr:	8.0	9.0	15.0
Maximum temperature, °F:	100	99	88

Note

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

TABLE 29. EFFECT OF ACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION GRAFTED ONLY

Grafting Solution Composition, wt %

329	330	331	332	333	335	
25	25	20	20	15	10	Acrylic acid
70	70	76	76	82	88	Benzene
5	5	4	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

Footage	Resistance, milliohm-inch^2					
	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)	9, 10(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

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	-
Max. temperature, °F:	-	90	100	99	81	-

Note

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

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

TABLE 30. SAMPLE NO. 300
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	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 No. 2 polyethylene film with cheesecloth interlayer. Used cooling coil in solution. Highly grafted with hompolymer impregnated in the film.

TABLE 31. SAMPLE NOS. 305 AND 306
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt % Benzene	Total Dose:	0.815 Mrad
3 wt % 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 in both samples.

Note

Neutralized and washed at 97°C. Prepared from JPL No. 2 polyethylene film with cheesecloth interlayer. Film has excellent clarity. Cooling coil in solution.

TABLE 32. SAMPLE NO. 307
GRAFTED ONLY

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

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	23, 41
25	27, 27
45	16, 31
65	48, 41
75	293, 67

Exotherm Data

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

Time to exotherm, hr:	16.0
Time to maximum exotherm, hr:	23.0
Maximum temperature, °F:	172

Note

Neutralized and washed at 97°C. Prepared from JPL No. 2 polyethylene film with cheesecloth interlayer.

TABLE 33. SAMPLE NO. 309
GRAFTED ONLY

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

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	JPL 2	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.

Time to exotherm, hr:	16.0
Time to maximum exotherm, hr:	23.0
Maximum temperature, °F:	168

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 34. SAMPLE NO. 337
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt % Benzene	Total Dose:	0.815 Mrad
3 wt % 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 35. SAMPLE NO. 338
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt % Benzene	Total Dose:	0.815 Mrad
3 wt % 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.

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

Note

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

TABLE 36. SAMPLE NO. 338
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt % Benzene	Total Dose:	0.815 Mrad
3 wt % 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

(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 37. SAMPLE NO. 356
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt % Benzene	Total Dose:	0.815 Mrad/hr
3 wt % 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. Roll No. 1 processed.

TABLE 38. 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 39. 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 grafting solution.
(2) On dry-film basis.

TABLE 40. DIVINYLBENZENE CROSSLINKING STUDIES

A. Electrical Properties

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

TABLE 40 (Continued)

B. Physical Properties

Sample No.	Thickness, mil			Dimensional Changes[3], %				Tensile Strength, psi	Elongation, %
				Width		Length			
	Dry	B. S.[1]	A. S.[2]	B. S.[1]	A. S.[2]	B. S.[1]	A. S.[2]	B. S.[1]	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

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

TABLE 41. PROPERTIES OF GRAFTED AND CROSSLINKED FILM

A. Electrical Properties

Sample No.	Crosslinking Conditions	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]
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 41 (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	-

(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 42. GRAFTED AND CROSSLINKED FILM

Crosslinking Solution Composition

1 vol % Divinyl sulfone
1 vol % Benzene
98 vol % 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-inch^2		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 43. EFFECT OF ADDITIVES IN GRAFTING SOLUTION GRAFTED ONLY

Grafting Solution Composition

25 wt % Acrylic acid
75 wt % Benzene
0 wt % Carbon tetrachloride unless otherwise indicated

Crosslinked in 1 vol % divinylbenzene solution

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, gram[1]	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, gram[1]	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.	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)
144	17	10	6.5	2.5	38.9	23.9
147	15	11	2.3	2.9	15.4	26.4
156	14	13	4.9	2.7	35.1	21.4
157	18	11	3.2	3.1	17.7	27.2
166	14	11	2.8	2.0	20.2	17.4
167	14	11	1.3	1.3	9.5	11.7
168	17	12	2.6	2.3	16.0	18.7
171	20	15	3.1	1.6	15.7	10.7
179	17	10	3.2	1.7	18.8	17.3

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)		
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.

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

TABLE 45. METHACRYLIC ACID GRAFTED POLYETHYLENE FILM GRAFTED AND CROSSLINKED

Grafting Solution Composition			Experimental Conditions for Grafting	
137	146			
25	25	wt % Methacrylic acid	Dose Rate:	0.012 Mrad/hr
70	75	wt % Benzene	Total Dose:	0.815 Mrad
5	-	wt % Carbon tetrachloride	Temperature:	77°F
			Atmosphere:	Nitrogen
			Roll Length:	30 feet

A. Electrical Properties

Sample No.	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)
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, 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)		
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.

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

TABLE 46. METHACRYLIC ACID GRAFTED POLYETHYLENE FILM
GRAFTED ONLY

Grafting Solution Composition

25 wt % Methacrylic acid
75 wt % Benzene

Experimental Conditions for Grafting

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

Sample No.	Resistance Range, milliohm-inch^2	Grafting Temperature, °F	Time to Exotherm, hr	Time to Maximum Exotherm, hr	Maximum Temperature °F
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°C.

(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 47. METHACRYLIC ACID GRAFTED FILM GRAFTED ONLY

Grafting Solution Composition, wt %				Experimental Conditions for Grafting	
339	340	341	342		
25	20	15	10 Methacrylic acid	Dose Rate:	0.012 Mrad/hr
75	80	85	90 Benzene	Total Dose:	0.815 Mrad
				Temperature:	72°F
				Atmosphere:	Nitrogen
				Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2			
	339	340	341	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	2.6	2.0
Time to maximum 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.

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

TABLE 48. 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
Poll Length:	30 feet

A. Electrical Properties

Sample No.	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)
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, mil			Dimensional Changes(3), % Width		Length		Strength(1), psi	Elongation(1) %
	Dry	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)	B.S.(1)	A.S.(2)		
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.

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

TABLE 49. GRAFTING WITH VINYLPYRIDINE MIXTURES GRAFTED ONLY

Grafting Solution Composition, wt %				Experimental Conditions for Grafting	
184	239	240			
20	24	20	Acrylic acid	Dose Rate:	0.012 Mrad/hr
5	1	-	4-Vinylpyridine	Total Dose:	0.815 Mrad
-	-	5	2-Vinylpyridine	Atmosphere:	Nitrogen
75	75	75	Water	Roll Length:	30 feet

Electrical Properties

Sample No.	Resistance Range, milliohm-inch^2	Grafting Temperature, °F	Time to Exotherm, hr	Time to Maximum Exotherm, hr	Maximum Temperature, °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°C.
Sample Nos. 239 and 240 neutralized and washed at 97°C.

TABLE 50. GRAFTING WITH VINYLPYRIDINE MIXTURE
SAMPLE NO. 184
GRAFTED AND CROSSLINKED

Grafting Solution Composition	Experimental Conditions for Grafting	
20 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
5 wt % 4-Vinylpyridine	Total Dose:	0.815 Mrad
75 wt % 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. Electrical Properties

Thickness, mil			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

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

TABLE 51. SAMPLE NOS. 297 AND 310
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
4.7 wt % Sodium vinylsulfonate	Dose Rate:	0.012 Mrad/hr
18.8 wt % Acrylic acid	Total Dose:	0.815 Mrad
76.5 wt % Water plus 5.2 g $FeSO_4 \cdot 7H_2O$ per 2 kilo of grafting solution	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

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

Exotherm Data

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

Note

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

APPENDIX A

Crosslinking of Acrylic Acid Grafted Polyethylene Film with Electron Beam

ELECTRON 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" piece were coded for identification purposes. These 34" pieces were placed inside a 36" long by 29" wide x 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 was 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