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FABRICATION AND TESTING OF BATTERY SEPARATOR MATERIAL FOR THE JET PROPULSION LABORATORY

JPL Purchase Order CA 384807

Prime Contract NAS 7-100, Task Order RD-38

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LOCKHEED GEORGIA NUCLEAR LABORATORY
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In compliance with the requirements of Purchase Order CA-384807 issued by Jet Propulsion Laboratory to the Lockheed-Georgia Company, approximately 240 lineal feet of battery separator material were processed, tested, and supplied to Jet Propulsion Laboratory by the Lockheed Georgia Nuclear Laboratory.

This report describes the process used in preparing the material and presents results of tests of the battery separator material furnished under this purchase order.

3

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

1.0 INTRODUCTION

Continuing interest exists in development of specialized battery separator materials for use in a number of modern batteries, particularly for those employed in space applications. A previously conducted research and development study for the Jet Propulsion Laboratory (JPL) by Radiation Applications, Inc., indicated that an acceptable battery separator material could be prepared by crosslinking polyethylene film with divinylbenzene followed by grafting with acrylic acid using gamma irradiation to effect the crosslinking and grafting.

As part of its effort to establish sources of supply for this material, JPL issued a purchase order to Lockheed-Georgia Company for 200 lineal feet of the material to be processed according to JPL procedures. The suitability of the processed material for its intended application was to be evaluated by measurements of changes in physical dimensions resulting from multiple sterilization cycles in 40 percent potassium hydroxide solution at 145°C, by the absorbance ratio of the carboxyl group to the methylene group using infrared spectroscopy, by the wet tensile strength, and by the electrical resistance. The limiting criteria for acceptable material specified by JPL were a minimum tensile strength of 700 psi and a maximum electrical resistance of 0.060 ohms per square inch per mil thickness.

Material processed at the Lockheed Georgia Nuclear Laboratory (LGNL) in conformance with processes and procedures specified by JPL did not meet the criteria for maximum electrical resistance. Furthermore, the desired carboxyl salt peak did not appear in the IR spectrograms, but instead, a carboxylic acid peak was present. A modification of the JPL procedure was developed by LGNL which allowed consistent production of material that conformed to the electrical resistance and tensile requirements and had IR spectrograms exhibiting the desired carboxyl salt peak with the carboxylic acid peak eliminated.

Conferences with JPL technical personnel at LGNL on 10 March and 23 March resulted in technical approval of the LGNL processing modification and also in a change to the purchase order to permit the change in processing procedure and a reduction in the number of tests to be performed.

Details of the processing procedures and descriptions of special equipment used in preparing the approximately 240 lineal feet of battery separator material furnished under Purchase Order CA 384807 are presented followed by descriptions and results of tests performed.

2.0 PROCESSING PROCEDURES

The battery separator material was prepared from polyethylene film supplied by JPL. The density of the film was 0.917, its nominal thickness was 0.001 inch, and its width was 13 inches. Processing of the polyethylene film comprised crosslinking in divinylbenzene solution by exposure to gamma radiation followed by converting the crosslinked material into a grafted polymer by irradiating in an acrylic acid solution using a gamma source. During irradiations the film was wound in a helix using a special crepe paper toweling furnished by JPL as a separator. This paper separator also acted as a wick to maintain a supply of either the crosslinking or grafting solution in contact with the film surface. Between the crosslinking and grafting irradiations the film was rinsed in benzene; following the grafting irradiation it was washed first in hot potassium hydroxide solution and then in hot water. These operations required the special equipment described in Section 2.1; processing details are presented in Section 2.2.

2.1 Special Equipment

The handling equipment used for rolling the polyethylene film and paper toweling into a helix suitable for processing in standard test tubes (65 mm diameter X 500 mm long) is shown in Figure 1. The paper and film were concurrently passed from the supply rolls through the guide rolls onto a 0.5 inch diameter stainless steel tube used as a core for the helix. With this equipment the film, from the upper supply roll, was positioned so that the paper extended approximately 1.5 inches beyond the film at each end of the helix. In addition to its use in preparing the helix configurations for the various irradiations, this equipment was also used for supporting the roll of material during removal of the paper toweling and transfer of the film to the racks used for supporting the film during the rinsing or washing operations.

The wash racks were so constructed that the film was supported and separated from adjacent layers during rinse and wash operations by stainless steel wire cloth which permitted circulation of the rinse or wash liquids to all surfaces of the film. One rack loaded with film is shown being lowered into a wash tank in Figure 2.

For the benzene rinse an open topped stainless steel tank 12 inches deep, 22 inches wide and 36.5 inches long was used. Two similar tanks insulated on the outside with 2 inches of styrofoam with a 4-inch thick styrofoam cover and fitted with stainless steel steam coils on the inside of the bottom were used for the potassium hydroxide solution and water washes. Temperatures of 80 ± 5°C were maintained during the washing cycles.

apply a 25 psi load during all measurements. Length and width measurements were made with a stainless steel rule graduated to 0.01 inch.

Specimens were sterilized in stainless steel containers heated in an oven in which the temperature was maintained at 145 ± 2°C during each sterilization cycle by a Leeds and Northrup Speedomax G oven controller with a 1.5 millivolt span and a suppressed zero. Pre-sterilization warm-up and post-sterilization cool-down of the oven was controlled with a synchronous driven programmer that supplied a bucking voltage to the controller and also regulated oven dampers such that warm-up was effected uniformly over a 1-hour period and cool down was at a uniform rate over a 2-hour period.

3.2 Test Procedures

Although a few continuous infrared spectrograms were made, only spectral measurements between wave lengths of 2.5 and 4.0 microns and 5.5 and 7.0 microns were made on most specimens since the methylene and carboxyl peaks pertinent to evaluation of the battery separator material were defined within these wave length ranges. As specified in the purchase order the ratio of the carboxyl peak to the methylene peak was calculated. The method used to establish a base line for each peak is illustrated in Figure 9. In this figure the upper spectrogram is for unsterilized material and the lower spectrogram is for a specimen from the same sample of material after sterilization for 72 hours in 40 percent potassium hydroxide solution at 145°C. Both the carboxyl and methylene peaks of the two spectrograms are superimposed, but the base lines are quite different. The difference in ratios (0.6 for unsterilized material and 0.8 for sterilized material), therefore, is due to the change in base line rather than to any difference in energy transmission at the pertinent wave lengths. These spectrograms are typical for all material supplied. The energy transmission at the maximum absorbance for both the carboxyl and methylene peaks was in the range 0.1 to 1.5 percent transmission for all specimens tested.

Electrical resistance measurements were made using the previously described equipment in accord with the following typical procedure. Since the measurement cell was filled with demineralized water during overnight storage, before using the cell for measurements the water was drained and the cell was filled with 40 weight percent potassium hydroxide solution. To ensure that the electrolyte used for measurements was of the desired concentration, the cell was filled with 40 percent potassium hydroxide from the reservoir, allowed to soak for 5 minutes (minimum) and the electrolyte drained and discarded. This operation was repeated twice. After the third filling the cell resistance was measured. The cell was again drained, refilled with fresh electrolyte and the cell resistance again measured. If these two measurements agreed within 0.05 ohms the cell was ready for specimen

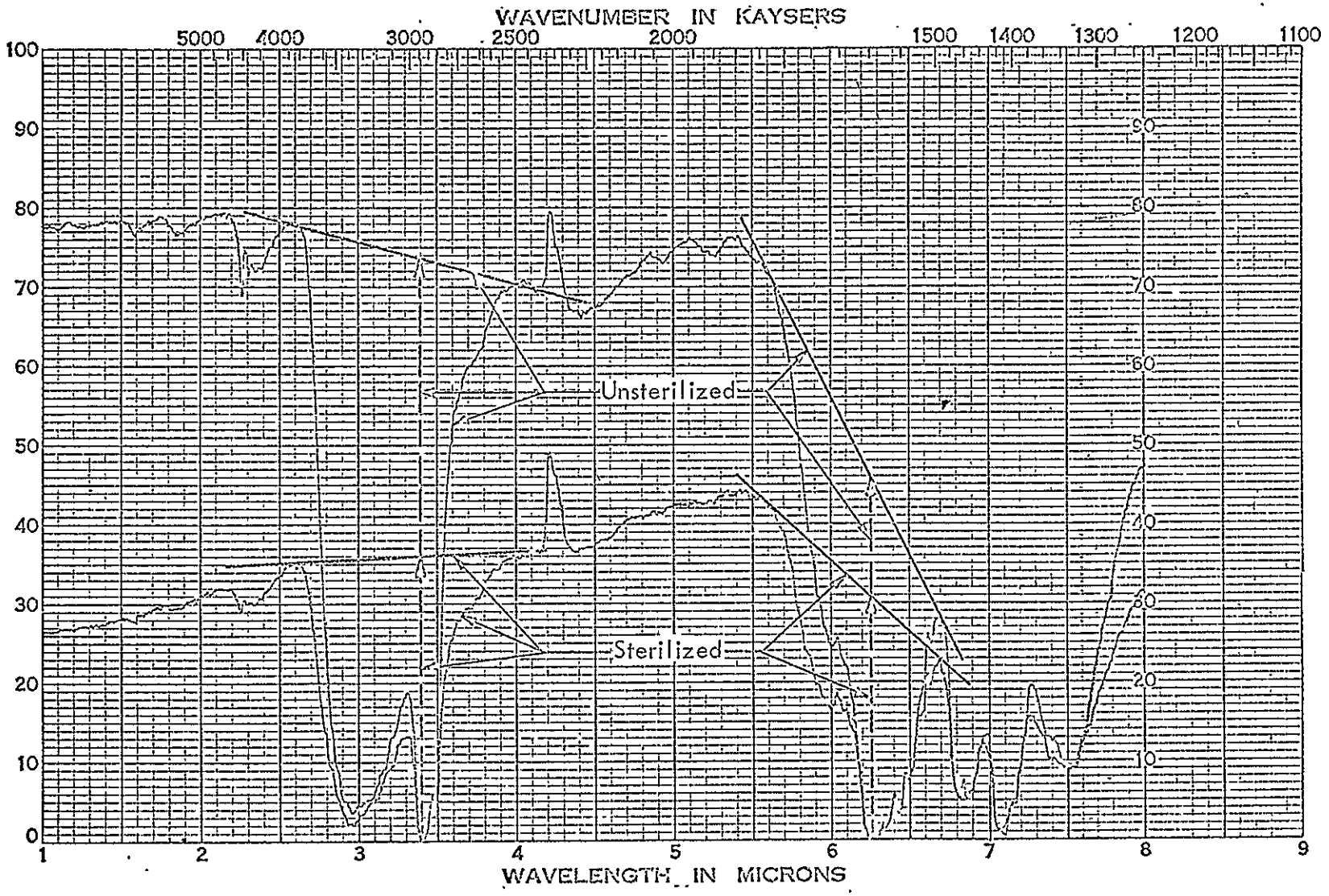

FIGURE 9 COMPARISON OF SPECTROGRAMS OF STERILIZED AND UNSTERILIZED MATERIAL

measurements. The electrolyte was returned to the reservoir and a test specimen was mounted in the cell so that the area measured was randomly selected with respect to length of the specimen (specimens were cut perpendicular to the length of the processed film) and was at least 0.125 inch from any specimen edge, the cell was closed, the wing nuts tightened to finger tight, and the cell filled with electrolyte to at least 0.250 inch above the electrodes. The bridge was then balanced for both capacitance and resistance to produce a null indication on both the phase angle difference and impedance indicators. When the cell resistance drift was observed to be less than 10 milliohms in a 15 second period the decade resistor value was recorded as the total cell and sample resistance. Electrical resistances were measured at 25° ± 0.3°C. The temperature of the electrolyte in the cell was recorded to the nearest 0.1°C at the time of this measurement. Empty cell resistance was determined by repositioning the specimen so that a 0.313 hole in the specimen was alligned with the 0.250 hole in the cell face and measuring the resistance as before. The resistance of the specimen was computed as follows:

$$\rho = \frac{(R_1 - R_2)\, A}{t \times 1{,}000}$$

where:

ρ = Resistance of sample ($\Omega/in^2/mil$)
R_1 = Resistance of sample and cell (Ω)
R_2 = Resistance of empty cell (Ω)
A = Area (in^2)
t = Thickness of sample (in)

Tensile strength measurements were made on a portion of the sample used for resistance measurements. Tensile specimens were nominally 1-inch wide and were tested over a 2-inch uniform width gage length. A load cell was used which provided for a 5 pound load over the full scale of the recorder. A number of specimen failures occurred at or within the grips. Although some failures of this type were presumably due to the uniform cross section between the grips, premature failures were to a great extent due to solution evaporation on the grips depositing minute crystals of potassium hydroxide which tended to cut the film and initiate failure. After this phenomenon was observed the grips were washed between tests thereby reducing the number of premature failures at the grips.

The specimens subjected to sterilization were coded for identification and placed in appropriate stainless steel sterilization containers according to the number of sterilization cycles to which they were to be subjected. The containers, which incorporated screens that ensured that the specimens were completely submerged during sterilization, were filled with 40 percent potassium hydroxide solution, sealed and placed in the oven for sterilization.

Twelve specimens from each of samples Ex-10, Ex-11, and 1B were subjected to sterilization as follows:

4 specimens	1 cycle	36 hours
4 specimens	2 cycles	72 hours
4 specimens	3 cycles	108 hours

Each sterilization cycle included 1 hour warm up time, 36 hours at 145 ± 2°C, and 2 hours cool down.

3.3 Test Results

The battery separator material furnished under Purchase Order CA 384807 was processed in 6 sample lengths designated Ex-10, Ex-11, 1A, 2B, 2C, and 3B. A complete set of 16 specimens were cut from near one end of each of samples Ex-10, Ex-11, and 2B. Four specimens from each set were tested after the following treatments:

- Unsterilized
- Sterilized for 36 hours in 40 percent KOH at 145°C.
- Sterilized for 72 hours in 40 percent KOH at 145°C.
- Sterilized for 108 hours in 40 percent KOH at 145°C.

On the unsterilized specimens wet width, wet thickness, and tensile strength were measured. On the sterilized specimens weight, length, width, and thickness were measured dry and wet before sterilization and wet after sterilization; infrared spectra and electrical resistance were measured both before and after sterilization; and tensile strength was measured after sterilization. The data are presented in Tables 1 through 6.

Electrical resistances of all specimens were well below the specified maximum. Tensile strengths of all specimens that did not fail in the grips were above the desired minimum; of those specimens that did fail in the grip only six failed at loads below the minimum. Tensile strength values obtained from such specimens do not represent the actual strength of the material since this type failure is usually initiated by damage to the specimen by the grip.

Results of these tests indicated that the material was uniform, that sterilization did not significantly change electrical resistance or reduce tensile strength below the acceptable minimum, and that material exhibiting certain carboxylate salt and methylene peaks also exhibited the desired electrical resistance characteristics. Comparison of infrared spectra and electrical resistance of the material initially processed which failed to meet electrical resistance requirements, as discussed in Section 2.2, further indicated the correlation between infrared spectra and electrical resistance properties. Therefore, the uniformity of

TABLE 1 - SAMPLE EX-10, WEIGHT AND DIMENSIONS

SPECIMEN		WEIGHT (GMS) STERILIZATION			LENGTH (IN.) STERILIZATION			WIDTH (IN.) STERILIZATION			THICKNESS (IN.) STERILIZATION								
											PRE - DRY			PRE - WET			POST - WET		
NO.	STERIL-IZATION	PRE DRY	PRE WET	POST WET	PRE DRY	PRE WET	POST WET	PRE DRY	PRE WET	POST WET	TOP	MIDDLE	BOTTOM	TOP	MIDDLE	BOTTOM	TOP	MIDDLE	BOTTOM
80	72 hrs.	0.206	0.567	0.645	9.72	10.96	11.42	0.97	1.08	1.05	0.0014	0.0013	0.0013	0.0012	0.0013	0.0012	0.0015	0.0014	0.0013
81	72 hrs.	0.202	0.577	0.687	9.77	10.80	11.26	0.99	1.08	1.05	0.0014	0.0013	0.0013	0.0012	0.0012	0.0011	0.0014	0.0014	0.0013
82	72 hrs.	0.206	0.600	0.686	9.75	10.86	11.31	0.98	1.08	1.05	0.0013	0.0013	0.0014	0.0012	0.0011	0.0011	0.0014	0.0013	0.0014
83	72 hrs.	0.198	0.674	0.697	9.77	10.85	11.30	0.98	1.08	1.05	0.0012	0.0013	0.0013	0.0011	0.0012	0.0011	0.0014	0.0013	0.0013
84	108 hrs.	0.201	0.550	0.696	9.68	10.87	11.32	0.98	1.07	1.06	0.0014	0.0013	0.0013	0.0013	0.0012	0.0011	0.0014	0.0013	0.0014
85	108 hrs.	0.205	0.651	0.729	9.88	10.95	11.30	0.99	1.08	1.07	0.0012	0.0013	0.0013	0.0011	0.0012	0.0012	0.0013	0.0014	0.0014
86	108 hrs.	0.211	0.638	0.665	9.93	10.93	11.35	1.00	1.08	1.05	0.0012	0.0013	0.0013	0.0012	0.0012	0.0012	0.0015	0.0015	0.0014
87	108 hrs.	0.200	0.566	0.651	9.78	10.79	11.15	0.99	1.09	1.03	0.0013	0.0013	0.0013	0.0012	0.0012	0.0011	0.0014	0.0015	0.0014
88	36 hrs.	0.203	0.622	0.690	9.79	10.58	10.77	1.00	1.07	1.05	0.0011	0.0012	0.0011	0.0013	0.0013	0.0012	0.0014	0.0013	0.0013
89	36 hrs.	0.188	0.534	0.619	9.77	10.50	10.66	1.00	1.07	1.03	0.0011	0.0012	0.0011	0.0013	0.0013	0.0013	0.0013	0.0014	0.0013
90	36 hrs.	0.203	0.622	0.599	9.83	10.59	10.75	1.00	1.08	1.05	0.0012	0.0012	0.0011	0.0012	0.0013	0.0012	0.0014	0.0014	0.0013
91	36 hrs.	0.194	0.511	0.640	9.74	10.42	10.57	1.00	1.09	1.05	0.0012	0.0011	0.0011	0.0011	0.0012	0.0013	0.0013	0.0013	0.0013
96	0 hrs.								1.03					0.0013	0.0014	0.0013			
97	0 hrs.								1.05					0.0014	0.0014	0.0014			
98	0 hrs.								1.05					0.0013	0.0011	0.0012			
99	0 hrs.								1.05					0.0013	0.0013	0.0014			

TABLE 2 - SAMPLE EX-10 PROPERTIES

SPECIMEN IDENTIFICATION	CARBOXYLATE / METHYLENE RATIO STERILIZATION (HRS.)				RESISTIVITY (Ω/IN²/MIL.) STERILIZATION (HRS.)				TENSILE STRENGTH (PSI) STERILIZATION (HRS.)			
	0	36	72	108	0	36	72	108	0	36	72	108
Outside End												
Top				0.99								
Middle	0.60			1.26								
Bottom				1.09								
Inside End												
Top												
Middle												
Bottom	0.59											
80	0.57		1.12		0.017		0.007				1136	
81	0.60		1.29		0.019		0.011				1099	
82	0.60		0.99		0.014		0.007				986	
83	0.72		1.27		0.015		0.009				813*	
84	0.60			1.11	0.015			0.007				889
85	0.63			1.32	0.014			0.004				1255*
86	0.58			1.21	0.014			0.004				912
87	0.64			1.65	0.016			0.006				860
88	0.60	1.33			0.022	0.010				623*		
89	0.65	1.81			0.018	0.009				665*		
90	0.59	1.21			0.018	0.008				733*		
91	0.66	1.73			0.018	0.008				872*		
96									1098*			
97									1048*			
98									1262*			
99									1116*			

*Failed in grip.

TABLE 3 - SAMPLE EX-J1, WEIGHT AND DIMENSIONS

SPECIMEN		WEIGHT (GMS) STERILIZATION			LENGTH (IN.) STERILIZATION			WIDTH (IN.) STERILIZATION			THICKNESS (IN.) STERILIZATION								
											PRE - DRY			PRE - WET			POST - WET		
NO.	STERIL-IZATION	PRE DRY	PRE WET	POST WET	PRE DRY	PRE WET	POST WET	PRE DRY	PRE WET	POST WET	TOP	MIDDLE	BOTTOM	TOP	MIDDLE	BOTTOM	TOP	MIDDLE	BOTTOM
67	72 hrs.	0.234	0.507	0.660	9.60	10.40	10.75	1.00	1.10	1.07	0.0013	0.0012	0.0012	0.0012	0.0012	0.0013	0.0015	0.0015	0.0014
68	72 hrs.	0.232	0.613	0.698	9.45	10.47	10.43	1.00	1.08	1.05	0.0012	0.0012	0.0012	0.0012	0.0012	0.0013	0.0016	0.0016	0.0016
69	72 hrs.	0.309	0.765	1.136	9.50	10.30	10.65	0.99	1.05	1.02	0.0012	0.0012	0.0013	0.0012	0.0013	0.0013	0.0016	0.0016	0.0017
70	72 hrs.	0.238	0.620	0.619	9.75	10.45	10.95	1.00	1.06	1.02	0.0011	0.0012	0.0012	0.0012	0.0012	0.0013	0.0015	0.0017	0.0016
71	36 hrs.	0.233	0.597	0.683	9.50	10.35	10.77	1.00	1.07	1.05	0.0010	0.0011	0.0011	0.0012	0.0013	0.0013	0.0014	0.0015	0.0015
72	36 hrs.	0.235	0.588	0.743	9.85	10.57	11.07	1.00	1.05	1.03	0.0012	0.0012	0.0012	0.0013	0.0013	0.0012	0.0015	0.0015	0.0015
73	36 hrs.	0.234	0.612	0.689	9.74	10.51	10.95	1.00	1.07	1.00	0.0012	0.0011	0.0011	0.0013	0.0013	0.0013	0.0015	0.0015	0.0015
74	36 hrs.	0.230	0.596	0.718	9.80	10.57	10.97	1.00	1.06	1.01	0.0013	0.0012	0.0012	0.0013	0.0012	0.0012	0.0015	0.0015	0.0015
75	108 hrs.	0.226	0.581	0.737	9.47	10.50	10.90	0.98	1.05	1.01	0.0012	0.0012	0.0012	0.0012	0.0013	0.0012	0.0015	0.0016	0.0016
76	108 hrs.	0.225	0.650	0.842	9.63	10.41	10.85	1.00	1.10	1.03	0.0012	0.0012	0.0012	0.0013	0.0014	0.0013	0.0017	0.0016	0.0016
77	108 hrs.	0.232	0.592	0.744	9.80	10.55	11.15	0.99	1.07	1.04	0.0012	0.0012	0.0012	0.0013	0.0012	0.0013	0.0016	0.0016	0.0016
78	108 hrs.	0.225	0.640	0.679	9.66	10.35	10.80	1.00	1.08	1.00	0.0013	0.0012	0.0013	0.0012	0.0013	0.0014	0.0016	0.0017	0.0017
92	0 hrs.								1.05					0.0013	0.0013	0.0013			
93	0 hrs.								1.06					0.0014	0.0014	0.0014			
94	0 hrs.								1.06					0.0014	0.0014	0.0014			
95	0 hrs.								1.06					0.0014	0.0014	0.0013			

TABLE 4 - SAMPLE EX-11, PROPERTIES

SPECIMEN IDENTIFICATION	CARBOXYLATE / METHYLENE RATIO STERILIZATION (HRS.)				RESISTIVITY (Ω/IN²/MIL) STERILIZATION (HRS.)				TENSILE STRENGTH (PSI) STERILIZATION (HRS.)			
	0	36	72	108	0	36	72	108	0	36	72	108
Outside End												
Top	0.53											
Middle	0.59											
Bottom	0.57											
Inside End												
Top	0.60											
Middle	0.67											
Bottom	0.66											
67	0.56		0.67		0.014		0.010				454*	
68	0.54		1.20		0.012		0.004				673*	
69	0.54		1.27		0.013		0.006				519*	
70	0.57		1.06		0.012		0.003				460*	
71	0.58	0.98			0.015	0.005				1016*		
72	0.55	0.91			0.016	0.007				803*		
73	0.53	0.88			0.021	0.007				907*		
74	0.55	0.76			0.025	0.005				738*		
75	0.57			1.24	0.016			0.006				743
76	0.55			1.65	0.015			0.007				758*
77	0.54			0.94	0.009			0.010				829
78	0.56			1.09	0.011			0.010				806*
92									1114*			
93									910			
94									1031*			
95									1061*			

*Failed in grip.

TABLE 5 - SAMPLE 2B, WEIGHT AND DIMENSIONS

SPECIMEN		WEIGHT (GMS)			LENGTH (IN.)			WIDTH (IN.)			THICKNESS (IN.)								
		STERILIZATION			STERILIZATION			STERILIZATION			STERILIZATION								
NO.	STERIL-IZATION	PRE DRY	PRE WET	POST WET	PRE DRY	PRE WET	POST WET	PRE DRY	PRE WET	POST WET	PRE-DRY			PRE-WET			POST-WET		
100	36 hrs.	0.220	0.608	0.740	9.60	10.52	10.91	0.97	1.05	1.02	0.0012	0.0011	0.0012	0.0012	0.0012	0.0014	0.0016	0.0016	0.0017
101	36 hrs.	0.217	0.711	0.744	9.60	10.45	10.95	0.98	1.05	1.05	0.0011	0.0012	0.0012	0.0012	0.0013	0.0013	0.0017	0.0017	0.0017
102	36 hrs.	0.232	0.771	0.756	9.84	10.73	11.32	0.97	1.06	1.03	0.0013	0.0013	0.0013	0.0012	0.0013	0.0013	0.0015	0.0016	0.0016
103	36 hrs.	0.222	0.702	0.773	9.77	10.67	11.30	0.98	1.05	1.02	0.0011	0.0012	0.0012	0.0012	0.0013	0.0014	0.0015	0.0017	0.0017
104	72 hrs.	0.230	0.725	0.807	9.76	10.77	11.25	0.98	1.06	1.05	0.0012	0.0013	0.0013	0.0012	0.0013	0.0014	0.0016	0.0017	0.0016
105	72 hrs.	0.216	0.662	0.757	9.85	10.65	11.12	0.99	1.05	1.00	0.0011	0.0012	0.0012	0.0012	0.0012	0.0013	0.0016	0.0017	0.0016
106	72 hrs.	0.221	0.723	0.804	9.89	10.77	11.22	0.99	1.05	1.03	0.0012	0.0012	0.0013	0.0012	0.0013	0.0015	0.0016	0.0016	0.0018
107	72 hrs.	0.230	0.815	0.975	10.00	10.85	11.30	1.00	1.06	1.03	0.0012	0.0012	0.0012	0.0012	0.0013	0.0013	0.0016	0.0017	0.0016
108	108 hrs.	0.214	0.817	0.579	9.97	10.83	11.47	0.98	1.05	1.01	0.0011	0.0011	0.0012	0.0011	0.0012	0.0011	0.0015	0.0015	0.0015
109	108 hrs.	0.222	0.759	0.657	9.89	10.80	11.20	0.99	1.08	1.01	0.0011	0.0013	0.0012	0.0012	0.0015	0.0012	0.0017	0.0018	0.0015
110	108 hrs.	0.217	0.803	0.573	9.86	10.72	11.30	0.98	1.05	1.01	0.0011	0.0012	0.0012	0.0011	0.0013	0.0012	0.0016	0.0016	0.0016
111	108 hrs.	0.222	0.651	0.592	9.83	10.78	11.18	0.98	1.05	1.01	0.0011	0.0013	0.0013	0.0013	0.0013	0.0013	0.0019	0.0019	0.0019
112	0 hrs.								1.07					0.0013	0.0014	0.0013			
113	0 hrs.								1.05					0.0012	0.0013	0.0012			
114	0 hrs.								1.06					0.0013	0.0014	0.0014			
115	0 hrs.								1.02					0.0012	0.0012	0.0013			

TABLE 6 - SAMPLE 2B, PROPERTIES

SPECIMEN IDENTIFICATION	CARBOXYLATE / METHYLENE RATIO STERILIZATION (HRS.)				RESISTIVITY (Ω/IN²/MIL) STERILIZATION (HRS.)				TENSILE STRENGTH (PSI) STERILIZATION (HRS.)			
	0	36	72	108	0	36	72	108	0	36	72	108
Outside End												
Top	0.69											
Middle	0.88											
Bottom	0.57											
100	0.66	1.92			0.015	0.008				930		
101	0.68	1.52			0.019	0.009				1031		
102	0.62	1.80			0.006	0.006				1068*		
103	0.65	1.57			0.007	0.006				957		
104	0.63		1.81		0.007		0.007				1054	
105	0.66		1.58		0.016		0.007				1199	
106	0.65		1.58		0.013		0.005				755*	
107	0.65		1.89		0.016		0.006				1032	
108	0.68			1.34	0.024			0.007				944*
109	0.68			0.83	0.016			0.005				1155
110	0.68			0.97	0.016			0.006				866*
111	0.68			0.78	0.015			0.006				703*
112	0.63								1510			
113	0.62								1286*			
114	0.59								1260			
115	0.64								1410			

*Failed in grip.

samples 1A, 2C, and 3B was assessed on the basis of infrared spectrograms made at random locations across specimens cut from the ends of the samples as processed. The spectrograms of these samples were essentially identical to all other spectrograms of unsterilized material from samples Ex-10, Ex-11, and 2B. Ratios of carboxylate to methylene peaks were essentially the same as shown in Table 7.

TABLE 7 - CARBOXYLATE/METHYLENE RATIOS FOR 1A, 2C, AND 3B

SPECIMEN IDENTIFICATION	END OF ROLL	
	OUTSIDE	INSIDE
SAMPLE 1A		
TOP	0.56	
MIDDLE	0.55	
BOTTOM	0.54	
SAMPLE 2C		
TOP	0.66	
MIDDLE	0.65	
BOTTOM	0.60	0.74
SAMPLE 3B		
TOP	0.57	0.58
MIDDLE	0.53	0.71
BOTTOM	0.52	0.64

29 JUN 66 54084

2

08