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Thermal and Arc Resistance of Polyester Resin Glass Reinforced Laminates

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Thermal and Arc Resistance of Polyester Resin Glass Reinforced Laminates.

A thesis presented to the Department of Chemistry of Union College in partial fulfillment of the requirements for the Degree of Master of Science in Chemistry.

B
By Frederick Budnik

Approved By H E Sluffer

Date

5/20/55

This work is dedicated to my wife--

Rita

I wish to express my thanks to Dr. Howard Sheffer for his help and guidance.

I also wish to thank the Schenectady Varnish Company for supplying the equipment and raw materials used for preparing the resins and also for supplying a few of the resins investigated.

Thanks are also expressed to the Mica Insulator Company for supplying the fiberglass reinforcing cloth and for the use of the laminating press and equipment for flexural and arc resistance tests.

ABSTRACT

The heat resistance of several types of polyester resins are studied by measuring the flexural strength of glass reinforced laminates before and after conditioning at 200°C. Arc resistance readings are also taken. The type of saturated acid, glycol and monomer is varied. The ratio of saturated to unsaturated acid and the ratio of monomer to base resin is also studied.

Chlorendic acid improves the heat resistance over phthalic and isophthalic acids but it lowers the arc resistance. Ethylene glycol yields resins with the highest flexural strengths. Styrene monomer content has little effect on heat resistance up to 40%. In the case of phthalic and isophthalic acid, increasing the proportion of unsaturated acid increases the heat resistance. However, the chlorendic-maleic acid combination shows optimum properties at a molar ratio of three to five.

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Introduction

Unsaturated polyesters are a natural outgrowth of the alkyd or polyester chemistry which developed in the early part of the twentieth century. They vary considerably in type and composition, although they are generally solutions of unsaturated alkyds made from difunctional glycols and acids in a vinyl monomer such as styrene. A free radical catalyst such as benzoyl peroxide will catalyze the cure by copolymerization of the vinyl monomer with the unsaturated groups in the polyester to yield a cross-linked or three-dimensional structure.

The properties of these polyester resins can be varied through a great range by means of the following variables:

1. Type of dihydric alcohol used.
2. Type of dibasic acid used.
3. Ratio of unsaturated to saturated dibasic acid.
4. Type and quantity of vinyl monomer used.

Each of these variables is studied in this paper.

Three different dihydric alcohols are used, namely ethylene glycol, diethylene glycol and propylene glycol.

A number of dibasic acids are used although most of the work involves phthalic anhydride, chlorendic acid and isophthalic acid with maleic acid as the unsaturated acid in all cases.

The ratio of unsaturated to saturated dibasic acid is varied over a significant range with some interesting results.

All of the resins are made with either styrene or diallyl phthalate. A short inquiry is made into the effect of styrene concentration. It is well known that the monomer triallyl cyanurate produces very heat resistant polyester resins but the cost of this material is prohibitive in commercial practice so there is considerable interest in reasonably priced heat resistant resins.

The heat resistance of resin laminates is of ever increasing significance as modern equipment is operated at higher and higher temperatures to increase efficiency and to save space or weight. Arc resistance is of great importance for switch gear and other electrical devices where easily formed conducting paths would cause failure of the equipment.

Perhaps the most important property of polyester resins is the fact that complete cure takes place without any by-products being formed. Thus, there is no solvent removal to hinder application of the resin, and the absence of any by-products permits low pressure molding and laminating to be carried out during resin cure.

History

The history of polyester resins is a relatively short one; however, the basic reactions involved in these and other synthetic resins were investigated as far back as 1847 when J. J. Berzelius reacted tartaric acid with glycerol to obtain a resinous mass. The primary interest of the early chemists was to prepare pure compounds and the resinous materials often produced were probably not mentioned in many cases, or just mentioned superficially without further investigation.

During the period between 1860 and 1863, Lourenço (7) synthesized polyethylene glycols by condensing ethylene glycol in the presence of ethylene dihalide. He separated individual members of the polymeric series up to $n = 6$. Their increasing viscosity led him to reason that the undistillable, highly viscous products also obtained must be of increased complexity, i.e., n greater than 6. He also noted that as n approaches infinity, the chemical composition approaches that of ethylene oxide. In 1894, D. Vorländer (19) prepared the maleates and fumarates of ethylene glycol. Watson H. Smith repeated Berzelius' work in 1901 and replaced the tartaric acid with phthalic anhydride.

The polymerization of vinyl monomers such as styrene, vinyl chloride and butadiene dates back to 1839 when Simon (20) reported the conversion of styrene to a gelatinous mass and Berthelot (21) applied the term, polymerization, to the process in 1866. These addition polymers were generally considered to be cyclic compounds until Staudinger proposed the chain formulas (22).

In 1929 Carothers (6) embarked on a series of brilliant investigations which established the molecular viewpoint expressed by Staudinger and cleared up the field of mystery surrounding resinous compounds. He introduced the distinction between condensation and addition polymers.

Voss and Dickhäuser obtained a German Patent in 1930 describing the preparation of hard, homogenous masses by heating a mixture of 120 parts of maleic anhydride and 105 parts of styrene on a water bath.

Carothers (24) did a great deal of work on alkyd and polyester type resins with the objective of preparing polymeric molecules of definitive structures through the use of established reactions of organic chemistry and to investigate how the properties of these substances depend on constitution.

Theory

The formation of a polyester resin involves two distinct reactions. The first involves the formation of an ester between a dicarboxylic acid and a glycol. Either the acid or the glycol must be suitably unsaturated so that addition polymerization, which is the second reaction, can be carried out. The resulting mass is a thermoset, chemically resistant resin with many excellent properties for a variety of applications. When combined with a reinforcing material such as fiberglass, these resins are capable of very high physical properties, therefore, they are put to many structural applications where weight is an important factor.

The final properties of a polyester resin are dependent to a large extent on the acid, glycol and monomer used and the relative proportions of each.

The esterification reaction is generally conceded to occur by breaking the carbon oxygen bond of the acid (8, 9).

Thus, $R\text{-}O\text{-}H + H\text{-}O\text{-}CO\text{-}R' \rightleftharpoons R\text{-}O\text{-}CO\text{-}R' + H_2O$

Evidence for this is based on the reaction between a mercaptan and a carboxylic acid.

$$C_2H_5SH + C_6H_5COOH \rightleftharpoons C_6H_5COSC_2H_5 + H_2O$$

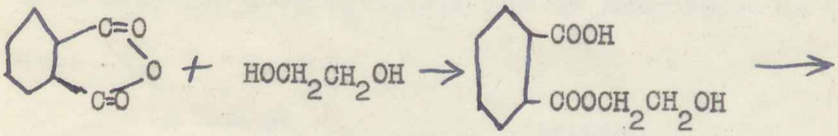
Similarly, reaction between an alcohol and a thioacid yields a sulfur free ester and hydrogen sulfide.

$$CH_3OH + C_6H_5COSH \rightarrow C_6H_5CO_2CH_3 + H_2S$$

These arguments are inconclusive, however, because the sulfur-containing analogs may not act in the same manner as the oxygen-containing compounds.

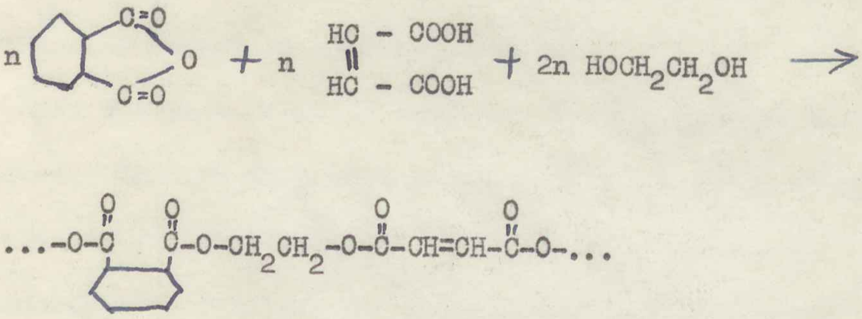
This ambiguity has been overcome by the use of isotopic oxygen following the same manner of reasoning and the same conclusions have been reached (10).

Reaction of the difunctional acid and alcohol yields a flexible chain polymer which is not heat convertible as there is no chance for cross linking.

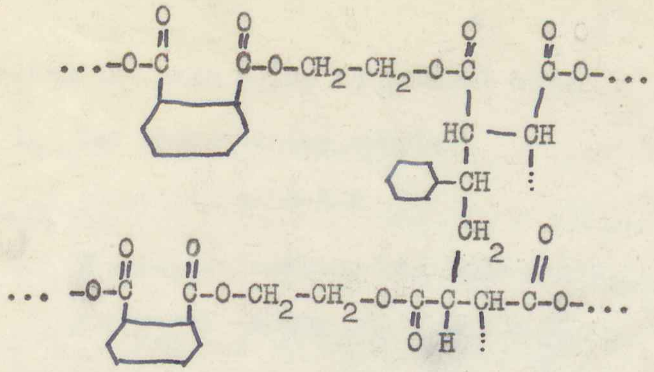


$HOCH_2CH_2O(COC_6H_4COOCH_2CH_2O)_nCOC_6H_4COOH$

Incorporating a suitably unsaturated component such as maleic acid allows cross linking to be developed. A copolymer is thus created by adding maleic anhydride to the above reaction.



This copolymer is still a linear chain and is termed an unsaturated alkyd. It is dissolved in an unsaturated vinyl type monomer such as styrene, diallyl phthalate, etc. which acts as both a solvent to reduce the viscosity of the alkyd and as a cross linking agent by addition polymerization.



This addition polymerization can be considered as taking place in three steps (4).

$M \rightarrow M^{*}$............Initiation

$M^{*} \xrightarrow{M} M_2^{*}$

$M_n^{*} \xrightarrow{M} M_{n+1}^{*}$..........Propagation

$M_n^{*} \rightarrow M_n$ ....Cessation

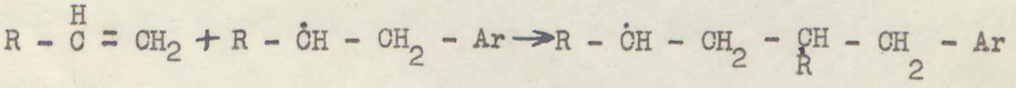
Tobolsky's report (11) of an equilibrium viscosity reached by polystyrene at high temperatures suggests that the polymerization propagation step may become reversible. This may account for part of the thermal degradation of resins at high temperatures.

Most evidence seems to support a free radical mechanism for polymerization. This is supported by the catalytic action of many compounds which are believed to decompose by way of active free radical intermediates. The commonly used catalyst for polyesters is benzoyl peroxide. This is decomposed by heat to acyl radicals.

$(ArCOO)_2 \rightleftharpoons 2\ ArCOO\cdot \xrightarrow{\Delta} Ar\cdot + CO_2$

These radicals initiate the polymerization:

$R - CH = CH_2 + Ar\cdot \rightarrow R - \dot{C}H - CH_2 - Ar$



$R - \overset{H}{C} = CH_2 + R - \dot{C}H - CH_2 - Ar \rightarrow R - \dot{C}H - CH_2 - \underset{R}{C}H - CH_2 - Ar$

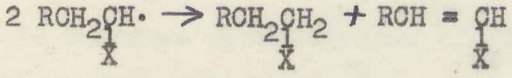
etc.

Cessation can take place in several ways.

1. Two radicals can couple.

$$2\,R\cdot \rightarrow R{-}R$$

2. Disproportionation can take place.



$$2\,RCH_2\underset{X}{C}H\cdot \rightarrow RCH_2\underset{X}{C}H_2 + RCH = \underset{X}{C}H$$

3. Chain transfer can occur.

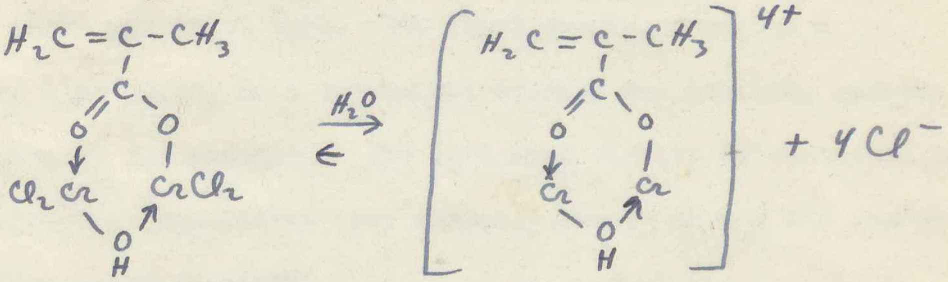
$$R{-}CH_2{-}CH_2\cdot + CCl_4 \rightarrow R{-}CH_2{-}CH_2Cl + CCl_3\cdot$$

Thus, the activity is transferred to another material in the mixture.

In order to retard the polymerization of the polyester resins during room temperature storage, an inhibitor must be added (17). Quinones and polynitroaromatic compounds rapidly react with any active free radicals to form inactive free radicals. Inactive here refers to the inability to add monomer molecules to propagate the chain. Obviously only a small quantity of inhibitor is desired because a sufficient excess of catalyst must be added to overcome it when polymerization is desired.

The properties of glass reinforced laminates prepared with polyester resins are greatly affected by the type of fiberglass cloth used and the finish on the fibers. The type of cloth determines the physical properties gained by various types of weave and will not be considered here. The finish applied to the fibers determines the bond attainable between the resin and the glass (18). The theory behind the various finishes is to provide a surface layer which will act as a bridge with a glass bonding end and a resin bonding end to the molecule. Type 136 finish is a vinyl silane, thus, the silicone end of the molecule has a strong affinity for the glass while the vinyl group can enter into the resin polymerization.

A great deal of work has been done on these glass finishes because of their importance in lowering moisture absorption with its resulting effect on electrical properties. The same fiberglass and finish are used throughout this work, these are #181 glass with 114 or Volan A finish. The Volan A finish is a Werner type compound (12).



Methacrylic acid is coordinated with chromium to form a highly reactive complex. The chromium complex is strongly held to siliceous materials such as glass fibers and the reactive double bond can combine with a wide variety of unsaturated resins.

Ebers et al (16) claim that aging characteristics of the laminates are unchanged by different glass finishes while Cummings & Botwick (13) claim better aging results with the 136 finish than with the 114 finish. To eliminate this possible variable, all laminates are made with the same 114 finish (Volan A).

Feuer et al (16) prove that copolymerization of maleic diesters with styrene does not take place directly. The maleic ester isomerizes to the fumaric ester during preparation of the base resin which readily reacts with styrene. Thus, no resins are prepared with fumaric acid for this paper since they should be essentially the same as the maleic resins.

EXPERIMENTAL

Equipment

The preparation of the polyester resin is carried out in a three liter, four necked flask. The center opening is used for an agitator which is sealed by a packed connector. The right hand opening is adapted to an inert gas inlet tube. The front opening contains a thermometer and also serves as a convenient opening for removing samples during the course of the reaction. The left hand opening is connected to a condenser through a Dean-Starke trap assembly except during the course of an ester interchange reaction.

The agitator consists of a glass rod with a stainless steel paddle. Nitrogen is used as an inert gas and is allowed to bubble through a glycol trap before entering the flask. This bubbler allows the rate to be controlled more easily.

Heat is applied by means of a Glas-Col heating mantle and is regulated with a Variac.

The laminates are cured in an 8 x 12 inch electrically heated Preco Press between chromium plated pressing steels. The steels are closed to 1/8 inch stops to maintain the desired thickness.

The glass reinforced laminates are cut into the required specimen sizes by means of a Felker Di-Met (diamond) circular saw. This saw is operated under water to prevent dispersing the hazardous glass dust into the atmosphere.

The flexural strength tests are made on a Baldwin-Southwark Universal Testing machine.

Arc resistance tests are performed on standard equipment as described in A.S.T.M. D-495. This test involves a high voltage low current type of arc and the values obtained are not applicable to other types such as high current arcs. The tungsten electrodes are spaced 0.250 inches apart and are pressed against the laminate surface with a force of 50 ± 5 grams. The sequence of arcs is applied as shown in Table I.

Table I - Sequence of One-Minute Current Steps

Step	Current Ma.	Time Cycle	Rate of Heat Generation in Watts	Total Time Seconds
1/8-10	10	1/4 sec. on, 1 3/4 sec. off	3	60
1/4-10	10	1/4 sec. on, 3/4 sec. off	6	120
1/2-10	10	1/4 sec. on, 1/4 sec. off	12	180
10	10	Continuous	24	240
20	20	Continuous	34	300

Procedure - Resin Preparation

The same general procedure is followed on all of the resins with an additional step of ester interchange for the resins involving dimethyl isophthalate and dimethyl terephthalate.

The glycols are charged to the flask and heated to 100°C. while the air is being flushed out with nitrogen. The acids and/or acid anhydrides are added with agitation. In most cases, these dissolve in a short time as the temperature is raised to the reacting level. The temperature is maintained at 160°C. for the HET resins. The other types are reacted at slowly increasing temperatures to as high as 225°C.

Test samples are removed with a glass thief at suitable intervals to maintain a control over the degree of esterification and polymerization. The sample is cut to 80% by weight with a solvent (equal parts of isopropyl alcohol and toluene) using heat if necessary. Part of this solution is placed in a Gardner-Holt Viscosimeter tube and checked for viscosity at 20°C. The remaining sample is cut with more solvent to reduce the viscosity and titrated with standardized 0.I N potassium hydroxide solution using phenolphthalein indicator. The result is reported as acid number by converting into mg. of KOH required per gram of sample. The acid number is an indication of the number of unreacted carboxy groups still present in the resin.

Most of the resins are cooked about 20 hours. About 10% excess glycol is added to lower the acid number before the polymerization proceeds too far.

When the esterification and polymerization have proceeded far enough, the resin batch is cooled below 200°F. and poured. The monomeric styrene or diallyl phthalate is added at or below 170°F. Good stirring is usually necessary to dissolve the resin.

0.02% p-benzoquinone is added as an inhibitor.

Laminate Preparation

Fiberglass cloth #181 with 114 finish is used for all laminates. This is 0. 0085" thick with a count of 57 x 54 and imparts high physical strength to the laminate.

Eleven pieces of the 181-114 glass cloth are cut to 7 x 12 inches, The long dimension must always be cut in the same direction so that the laminate will have consistent directionality of glass weave. The glass is weighed and the proper amount of resin is determined. A 60-40 ratio of glass to resin is used with a slight excess of resin to allow for squeeze out, and losses in the container, etc.

The weighed resin is catalyzed with 1% of Luperco ATC. This is a paste consisting of 50% benzoyl peroxide in tricresyl phosphate. It is more easily mixed with the resin than benzoyl peroxide alone. If the resin is very viscous and air bubbles are not readily removed, the catalyzed resin is carefully warmed to reduce the viscosity and allow the air bubbles to escape more rapidly. The lower viscosity of the warm resin also facilitates the coating operation.

The first ply of glass cloth is laid out on an oversized sheet of cellophane (non-waterproof) and a portion of the resin spread over the surface of the cloth with a spatula or other suitable instrument. Brushing is not feasible because of the high viscosity and tack of the resin. This coated ply is then turned resin side down on the cellophane which will act as a release agent during the pressing operation. The uncoated surface is then spread with more resin and the next ply laid with interlocking weave. This ply and each subsequent one is coated only on the top surface. The last ply is covered with a second sheet of cellophane.

The "stacked" laminate is then sandwiched between two 8x12 inch pressing steels and put into the hot press. Stops are placed to yield a 1/8 inch laminate and the press is closed to contact pressure. This is maintained for three to five minutes and then raised to 50 psi. After one hour at this pressure and 105°C., the press is opened, without cooling, and the laminate is removed. It is allowed to cool slowly between the pressing steels to minimize warping. If the cellophane release sheets do not come free, they are moistened with water and stripped off.

The laminates are trimmed and cut into 1 x 4 inch specimens. As the glass cloth reinforcing has directionality of weave, the specimens must be cut lengthwise and crosswise. The size of the laminate and the saw cut width allow fifteen specimens to be cut, i.e., eight in one direction, and seven in the other. Half of the specimens are tested after Condition A (no special preconditioning) according to the procedure of A.S.T.M. D 790-49. The 1/8 inch thick samples are tested as a simple beam (i.e., the load is applied at the center and the beam is supported at each end of the span). The span is two inches, the radius of supports and loading nose is 1/8 inch, and the rate of loading is 0.053 inches per minute.

The remaining half of the specimens are flexural tested at 200°C. after Condition E-18/200 (oven conditioning for 18 hr. at 200°C). The specimens are conditioned in the testing oven so that they are not allowed any opportunity to cool. The jig for the hot flexural test oven is equivalent to that used at room temperature and the loading conditions are the same.

The width and thickness are measured at the center of each specimen. The specimens do not break completely under test so the first sharp drop in load is taken as the breaking point.

The flexural strength is computed by means of the following equation:

$$S = \frac{3\ PL}{2\ bt^2}$$

where P - breaking load in pounds

L - distance between supports in inches

b - width in inches

t - thickness in inches.

The calculation for Resin II is typical. The following values are obtained from the Appendix:

P - 184.2 lbs.

L - 2 inches for all specimens

b - 1.005 inches

t - 0.119 inches

Inserting these in the above equation:

$$S = \frac{3(184.2)(2)}{2(1.005)(0.119)^2}$$

$$S = 38{,}800 \text{ PSI}$$

Arc resistance measurements are made on the same specimens that have been flexure tested with care taken to stay at least 1/4 inch away from the edges and the ruptured area. Breakdown occurs when carbonization forms a conductive path between the electrodes.

Results

An initial laminate was prepared with a commercial heat resistant polyester resin, namely No. BRSQ-142 made by the Bakelite Company (3). This provided a tentative goal indicative of the range of values to be expected under the laminating and testing conditions used throughout this work. The test results are shown in Table II. Table II also shows the results obtained with the use of HET.* Table III deals with resins made up with maleic and phthalic anhydride while Table IV contains data on terephthalic and isophthalic resin combinations. The individual results for all of the laminates are given in the Appendix.

The initial work was done with HET* also known as chlorendic acid. Robitschek and Bean (2) claim that this acid yields resins with higher strength at elevated temperatures with less susceptibility to heat degradation than the comparable phthalic resins. This is discussed later in greater detail.

Resin I

124 g.	2.0 moles (25%)	Ethylene Glycol
212 g.	2.0 moles (25%)	Diethylene Glycol
244 g.	2.5 moles (31%)	Maleic Anhydride
584 g.	1.5 moles (19%	HET Acid

Temperature maintained at 180°C. for four hours, acid number apparently 170.

The viscosity of Resin I was found to be too high to handle. It also was not compatible with styrene.

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*HET is a registered trade-mark of the Hooker Electrochemical Co., Niagara Falls, New York.

The acid number was very erratic; this was later found to be due to the Cellosolve used for dilution. Succeeding runs therefore were cut with a solvent mixture containing equal volumes of toluene and isopropanol. No laminate was prepared from Resin I.

Resin II

47.8 g.	0.77 mole (23%)	Ethylene Glycol
106.0 g.	1.00 mole (30%)	Diethylene Glycol
75.5 g.	0.77 mole (23%)	Maleic Anhydride
300.0 g.	0.77 mole (23%)	HET acid

Reaction was carried out at 180°C. for four hours. Half-hour samples were taken with a final acid number of 63 and a viscosity of V-W 23% styrene and 0.02% p-benzoquinone were mixed with the alkyd to yield a polyester resin with viscosity W and acid No. 38. As the p-benzoquinone inhibitor is added to all of the resins, it will not be mentioned specifically hereafter. The same procedure was carried out in preparing each laminate, in this case, 130 g. of resin (containing 1.3 g. Luperco ATC catalyst) and about 170 g. of glass cloth were combined as described previously (see under Experimental Procedure).

Cold Flexural Strength 38,800 and 35,800 PSI av.

Hot Flexural Strength 5,150 and 4,800 PSI av.

Arc Resistance ranged from 3 to 127 seconds.

Resin III

62 g. 1 mole (25%) Ethylene Glycol
106 g. 1 mole (25%) Diethylene Glycol
122 g. 1.25 mole (31%) Maleic Anhydride
292 g. 0.75 mole (19%) HET Acid

Esterification was carried out at 160°C. for a total of 20 hours to an acid number of 24 and a viscosity less than 16. 23% styrene was used.

Cold Flexural Strength 47,900 41,500 PSI av.
Hot Flexural Strength 20,900 13,700 PSI av.
Arc Resistance ranged from 4 to 19 seconds.

During the 200° conditioning, the color of the laminate changed to a tan.

Resin IV

106 g. 1 mole (25%) Diethylene Glycol
62 g. 1 mole (25%) Ethylene Glycol
147 g. 1.5 mole (37.5%) Maleic Anhydride
195 g. 0.5 mole (12.5%) HET acid

Esterification was carried out at 160°C. for 20 hours to a viscosity of V-W and an acid number of 54 with 23% styrene.

Cold Flexural Strength 45,000 41,900 PSI av.
Hot Flexural Strength 10,000 8,900 PSI av.
Arc Resistance ranged from 4.0 to 183.0 seconds

Arc resistance measurements were also made after Condition E-18/200 and showed slight improvement, although the range of values was similar from 6.5 to 182 seconds.

Resin V

124.1 g.	2 moles (50%)	Ethylene Glycol
145.1 g.	1.25 moles (31%)	Maleic Anhydride
291.6 g.	0.75 mole (19%)	HET Acid

Esterification was carried out at 160°C. for 16.5 hours to a viscosity of W and an acid number of 61 with 23% styrene. It was necessary to add a total of 32 grams of ethylene glycol during the reaction period to bring the acid number down.

Cold Flexural Strength	35,800	30,200 PSI av.
Hot Flexural Strength	10,460	8,700 PSI av.

Arc Resistance ranged from 3.2 to 127.6 seconds.

Two laminates were prepared with Resin V, the first was tested in the normal way while the second was given a post cure consisting of one hour in a 105°C. oven immediately after the pressing.

Cold Flexural Strength	47,100	43,100 PSI av.
Hot Flexural Strength	15,100	11,770 PSI av.

Arc Resistance ranged from 5.2 to 10.0 seconds.

Resin XIII

3.3 moles	(52%)	Propylene Glycol
2.0 moles	(32%)	Maleic Anhydride
1.0 moles	(16%)	Phthalic Anhydride

This resin was prepared by the Schenectady Varnish Co. and was esterified for 5.75 hours at 200°C. to a viscosity of Z-2 and an acid number 48 (in solvent). It was combined with 50% diallyl phthalate monomer. The reason for the "out of sequence" number designation is that the series numbers were not originally assigned to the resins made up by Schenectady Varnish, however, for this paper, the Company designations will be omitted.

Cold Flexural Strength	47,100	44,600 PSI av.
Hot Flexural Strength	11,000	10,500 PSI av.

Arc resistance ranged from 142 to 165 seconds.

Resin XIV

This was the same resin as XIII except it was combined with 33% styrene to yield a viscosity < U.

Cold Flexural Strength	43,400	41,300 PSI av.
Hot Flexural Strength	10,900	10,140 PSI av.

Arc Resistance ranged from 137 to 156 seconds.

Resin VI

248.3 g.	4 moles (50%) Ethylene Glycol
222.0 g.	1.5 moles (19%) Phthalic Anhydride
290.0 g.	2.5 moles (31%) Maleic Anhydride

Resin VI was esterified at 160°C. for 16.5 hours followed by 3.5 hours at 170°C. A total of 4.8 moles of ethylene glycol, i.e., 20% excess, only lowered the acid number to 78 when dissolved in 23% diallyl phthalate. A second portion was dissolved in the same ratio of styrene yielding resins VI-D and VI-S respectively.

Both resins were made into laminates and neither would cure during the normal pressing operation. A subsequent oven bake for 12 hours at 105° C. still left the laminates soft and tacky. No tests could be made.

Resin VII

136.6 g.	2.2 moles (52%)	Ethylene Glycol
97.1 g.	0.5 mole (12%)	Dimethyl Terephthalate
0.5 g.	--	Litharge

These materials were charged to the reaction flask and the temperature was slowly raised to 210°C. during four hours. The litharge acts as a catalyst in this ester interchange reaction. The methanol was removed as it formed. The theoretical quantity of methanol was not collected probably due to leaks in the various corks. The temperature would not go higher than 210°C. and the color darkened slightly. When the reaction vessel was cooled to room temperature, the reaction mass solidified.

After reheating to 100°C. the following anhydrides were added:

83.1 g.	0.5 mole (12%)	Phthalic Anhydride
116.1 g.	1.0 mole (24%)	Maleic Anhydride

The regular esterification reaction was then carried out at 160°C. for five hours, 160° to 175° for three hours, 180° to 210° for three hours and held at 210° for two hours. The solvent solubility was becoming very bad so the reaction was stopped at a viscosity X. The alkyd was insoluble in styrene but soluble in 23% diallyl phthalate with heating to yield an acid number of 56.

The high viscosity of this resin did not allow the normal glass ply coating procedure so a portion was spread in the center of each ply in the hope that the heat and pressure during lamination would cause the resin to flow. Some flow occurred but a complete filling of the laminate was lacking, therefore, only a few test specimens were possible. The test results did not warrant further work to produce a better laminate.

Cold Flexural Strength	45,700	61,500 PSI av.
Hot Flexural Strength	8,190	8,070 PSI av.

Arc Resistance ranged from 83 to 147 seconds.

A second laminate was prepared from this resin by diluting to a 72% solids solution in methyl ethyl ketone. The glass plies were easily coated with this solution and were oven dried for five minutes at 66°C. in a circulating air oven. The hot press was held at contact pressure for five minutes after inserting the laminate to allow any remaining solvent to breathe out.

Cold Flexural Strength	23,800	17,050 PSI av.
Hot Flexural Strength	1,023	9,880 PSI av.

Arc Resistance ranged from 126.8 to 139.6 seconds

Resin VIII

127.1 g.	0.5 moles (10.6%)	Pyromellitic Acid
147.1 g.	1.5 moles (32.0%)	Maleic Anhydride
105.5 g.	2.7 moles (57.4%)	Ethylene Glycol

Temperature rose from 155°C. to 184°C. in one hour, apparently due to an exothermic reaction. A check sample was insoluble in the usual solvents. Temperature rose to 200°C. in the next two hours. The reaction mass suddenly became very viscous and assumed a gelatinous state which became glassy when cooled. No laminates could be prepared.

Resin IX

273.1 g.	4.4 moles (52.4%)	Ethylene Glycol
194.1 g.	1.0 mole (11.9%)	Dimethyl Isophthalate
1.0 g.		Litharge

The ester interchange was carried out during three hours at a maximum temperature of 210°C. This mixture remained a clear, golden red liquid at room temperature.

232.1 g.	2.0 moles (23.8%)	Maleic Anhydride
166.1 g.	1.0 mole (11.9%)	Phthalic Anhydride

The total reaction time was 19 hours at temperatures from 160°C. to a maximum of 225°C. and a viscosity of Y. When dissolved in 30% D.A.P., the acid number was 48.

Cold Flexural Strength	46,800	50,200 PSI av.
Hot Flexural Strength	6,620	6,210 PSI av.

Arc resistance ranged from 136 to 154 seconds.

Resin X

116.4 g.	0.6 mole (14.3%)	Dimethyl Isophthalate
168.0 g.	2.2 moles (52.4%)	Propylene Glycol
0.5 g.		Litharge

The same procedure as IX was followed to remove methanol. 1.4 moles (33.3%) maleic anhydride, fifteen hours of reaction time between 180°C. and 215°C. yielded a resin with a Z-2 viscosity and acid number of 39 (80% solution). It was dissolved in 30% DAP with heat.

Cold Flexural Strength	43,100	49,400 PSI av.
Hot Flexural Strength	11,100	13,400 PSI av.

Arc Resistance ranged from 134 to 165 seconds

Resin XI

76.1 g. 2.2 moles (52.4%) Propylene Glycol

122.0 g. 1.25 moles (29.8%) Maleic Anhydride

292.0 g. 0.75 moles (17.8%) HET

Esterification was carried out at 160°C. for 28 hours to a viscosity of Z-5 and an acid number 50 (80%). The alkyd was dissolved in 30% styrene.

Cold Flexural Strength	38,300	41,200 PSI av.
Hot Flexural Strength	8,650	9,400 PSI av.

Arc resistance ranged from 3 to 7 seconds.

Resin XII

(Prepared by Schenectady Varnish Co.)

0.75 (17.8%) mole HET

1.25 (29.8%) mole Maleic Anhydride

2.2 (52.4%) moles Ethylene Glycol

Esterified 20 hours at 160°C. and dissolved in 20% styrene to yield an acid number 29 and viscosity Z-5.

Cold Flexural Strength	48,500	49,600 PSI av.
Hot Flexural Strength	13,160	13,500 PSI av.

Arc resistance ranged from 3 to 9 seconds.

Resin XIII

(Prepared by Schenectady Varnish Co.)

2.0 moles (31.7%) Maleic Anhydride

1.0 mole (15.9%) Phthalic Anhydride

3.3 moles (52.4%) Propylene Glycol

Esterified 5.75 hours at 200°C. and diluted with 50% D.A.P., viscosity Z-2 and acid number 30.

Cold Flexural Strength	47,100	44,600 PSI av.
Hot Flexural Strength	11,000	10,500 PSI av.

Arc Resistance ranged from 142 to 165 seconds.

Resin XIV

This resin was the same as XIII except it was dissolved in 33% styrene.

Cold Flexural Strength	43,400	41,300 PSI av.
Hot Flexural Strength	10,900	10,140 PSI av.

Arc Resistance ranged from 137 to 148 seconds.

Resins XV through XVIII

(Prepared by Schenectady Varnish Co.)

This series of resins was prepared under similar conditions with varying component ratios. The following quantities are given in moles:

Resin	Maleic	Phthalic	Prop. Glycol	Time	Temp.	Viscosity	Acid No.
XV	1.0	1.0	2.2	9½ hrs.	200°	Z-2 to Z-3	49
XVI	2.0	1.0	3.3	9 hrs.	200°	Z-2 to Z-3	48
XVII	3.0	1.0	4.4	8½ hrs.	200°	Z-2	53
XVIII	1.0	2.0	3.3	8¼ hrs.	200°	Z-1 to Z-2	46

The acid numbers as given are for the unsaturated alkyd. Each resin was dissolved in 25% styrene.

Resin	Cold Flexural Strength	Hot Flexural Strength	Arc Resistance
XV	47,700 and 54,200 PSI av.	6,430 & 7,510 PSI av.	134 to 182 sec.
XVI	49,300 and 49,400 PSI av.	9,670 & 9,920 PSI av.	133 to 170 "
XVII	55,000 and 54,800 PSI av.	9,950 & 11,780 PSI av.	142 to 181 "
XVIII	52,500 and 61,200 PSI av.	4,040 & 4,350 PSI av.	128 to 152 "

Resins XIX through XXI

These resins are all prepared from the same unsaturated alkyd (made by Schenectady Varnish Co.) with varying amounts of styrene.

1 mole (23.8%) Dimethyl Isophthalate

1 mole (23.8%) Maleic Anhydride

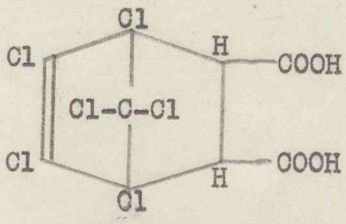
2.2 moles (52.4%) Propylene Glycol

The methanol was removed during seven hours with increasing temperature to a maximum of 193°C. Esterification occurred over 8.75 hours at 200°C. to an acid number 12 and viscosity W.

Resin	% Styrene	Cold Flex. Strength	Hot Flex. Strength	Arc Resistance
XIX	20	52,500 & 55,400 PSI	6,200 & 7,360 PSI	135 to 182 sec.
XX	30	52,800 & 53,700 "	8,420 & 8,730 "	30 to 168 "
XXI	40	51,400 & 55,200 "	6,150 & 8,150 "	27 to 174 "

Discussion of Results

The initial work was carried out with HET acid using maleic anhydride as the cross linking agent. HET is the abbreviated term for the cis isomer of hexachloroendomethylene tetrahydrophthalic acid I. The double bond is not reactive under polymerizing conditions and the



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chlorine atoms are firmly held and not effective as chain transfer agents. These halogens are also not readily susceptible to thermal dehydrohalogenation. This last reaction is one cause for poor heat resistance (13) and immediately excluded such acids as tetrachlorophthalic from this study.

Resins II, III and IV were prepared with varying ratios of maleic to HET acid (Table II). A maximum retention of flexural strength at 200°C. occurred with a ratio of 5 moles of maleic anhydride to 3 moles of HET acid (Fig.1.).

The same ratio of maleic to HET acid was kept while the diols were changed from equal parts of ethylene and diethylene glycols to all ethylene and all propylene glycol. The lowest values of flexural strength were obtained with propylene glycol. The highest values were expected with ethylene glycol. This was verified with the cold flexural strength, but the hot strength favored the mixture of ethylene and diethylene glycols.

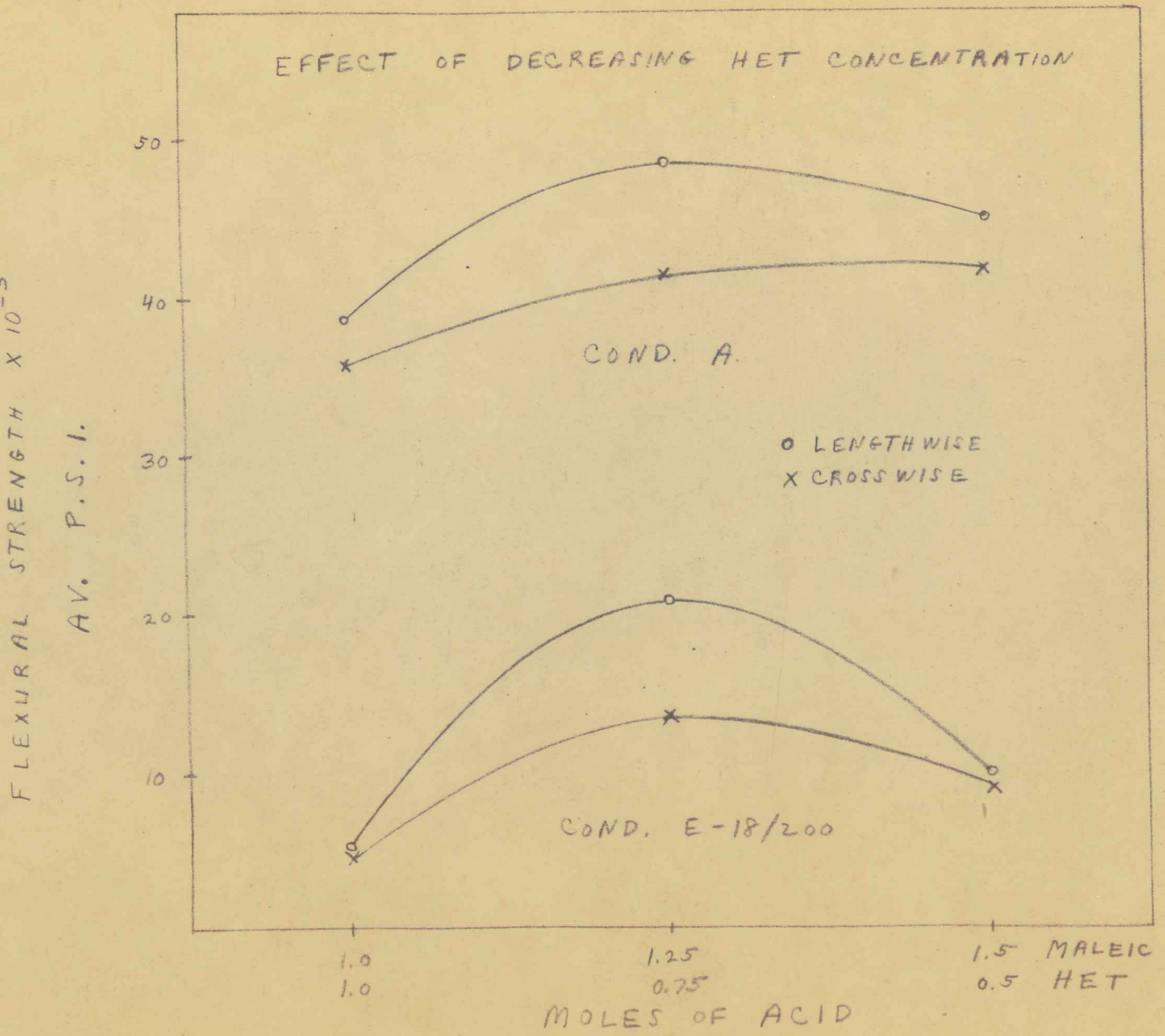
EFFECT OF DECREASING HET CONCENTRATION
FLEXURAL STRENGTH X 10^{-3}
AV. P.S.I.
50
40
30
20
10
COND. A.
o LENGTHWISE
X CROSSWISE
COND. E-18/200
1.0
1.0
1.25
0.75
1.5 MALEIC
0.5 HET
MOLES OF ACID

FIG. 1

Resin V has the same proportions as XII but the esterification was not as complete as indicated by a higher acid number. A second laminate as listed under V-a (Table II) was given a postcure which improved its properties to approximate those of XII.

Arc resistance is very low on all of these HET polymers. Apparently the flame resistance imparted to the resin by the chlorine atoms does not help to prevent tracking. To the contrary, it probably prevents combustion of the breakdown products formed under the arc, thus allowing rapid build-up of a conductive carbon path.

TABLE II

Resin	BRSQ-142	II	III	IV	V	V-a	XI	XII
Acid	Unknown	Maleic	Maleic	Maleic	Maleic	Maleic	Maleic	Maleic
Moles	--	1.0	1.25	1.5	1.25	1.25	1.25	1.25
Acid	--	HET	HET	HET	HET	HET	HET	HET
Moles	--	1.0	0.75	0.5	0.75	0.75	0.75	0.75
Glycol	Unknown	Ethylene	Ethylene	Ethylene	Ethylene	Ethylene	Propylene	Ethylene
Moles	--	1.0	1.0	1.0	2.5	2.5	2.2	2.2
Glycol	--	Diethylene	Diethylene	Diethylene	--	--	--	--
Moles	--	1.3	1.0	1. 0	--	--	--	--
Monomer	Styrene	Styrene 23%	Styrene 23%	Styrene 23%	Styrene 23%	Styrene 23%	Styrene 30%	Styrene 20%
Flex. Str.								
Cond. A (Av. PSI) L. W.	49,200	38,800	47,900	45,000	35,800	47,100	38,300	48,500
C. W.	47,200	35,800	41,500	41,900	30,200	43,100	41,200	49,600
E-18/200 (Av. PSI) L.W.	31,400	5,150	20,900	10,000	10,460	15,100	8,650	13,160
C.W.	22,800	4,800	13,700	8,900	8,700	11,770	9,400	13,500
Arc Resistance								
Cond. A (sec)	161-175	3-27	4-19	4-183	3-128	5-10	5	5
Percent Flex. Str. Retained L.W.	63.8	13.3	43.0	22.2	29.2	32.1	22.6	27.1
C.W.	48.3	13.4	33.2	21.2	28.8	27.3	22.8	27.2

A series of resins prepared with maleic and phthalic anhydrides is shown in Table III. Increasing the maleic to phthalic ratio, i.e., increasing the number of potential cross-linkages, showed no definite effect on the Condition A flexural strength but the high temperature measurements were improved. This is more clearly shown by Fig. 2. Thus, increased cross-linking density improves the heat resistance of the polyester.

Comparing Resin XIV and XVI (Table III), the increased amount of styrene reduced the cold flexural strength slightly but the hot flexural strength after aging is essentially the same. The effect of the styrene proportion is indicated in further detail in Table IV. Changing from styrene to diallyl phthalate monomer slightly improved the initial strength which ultimately dropped to the same value as the styrene resin after heat aging. The scope of this work did not include the effect of monomer variation and no further work was performed toward this end.

Arc resistance is good on all of the maleic phthalic combinations.

Resin VIII was prepared with the tetrafunctional pyromellitic acid. This was unsuccessful because cross linking occurred during the esterification reaction and the reaction mass gelled.

The maleic-phthalic resin combinations showed less promise for heat resistance than HET based resins so another series of resins was prepared using terephthalic and isophthalic acids.

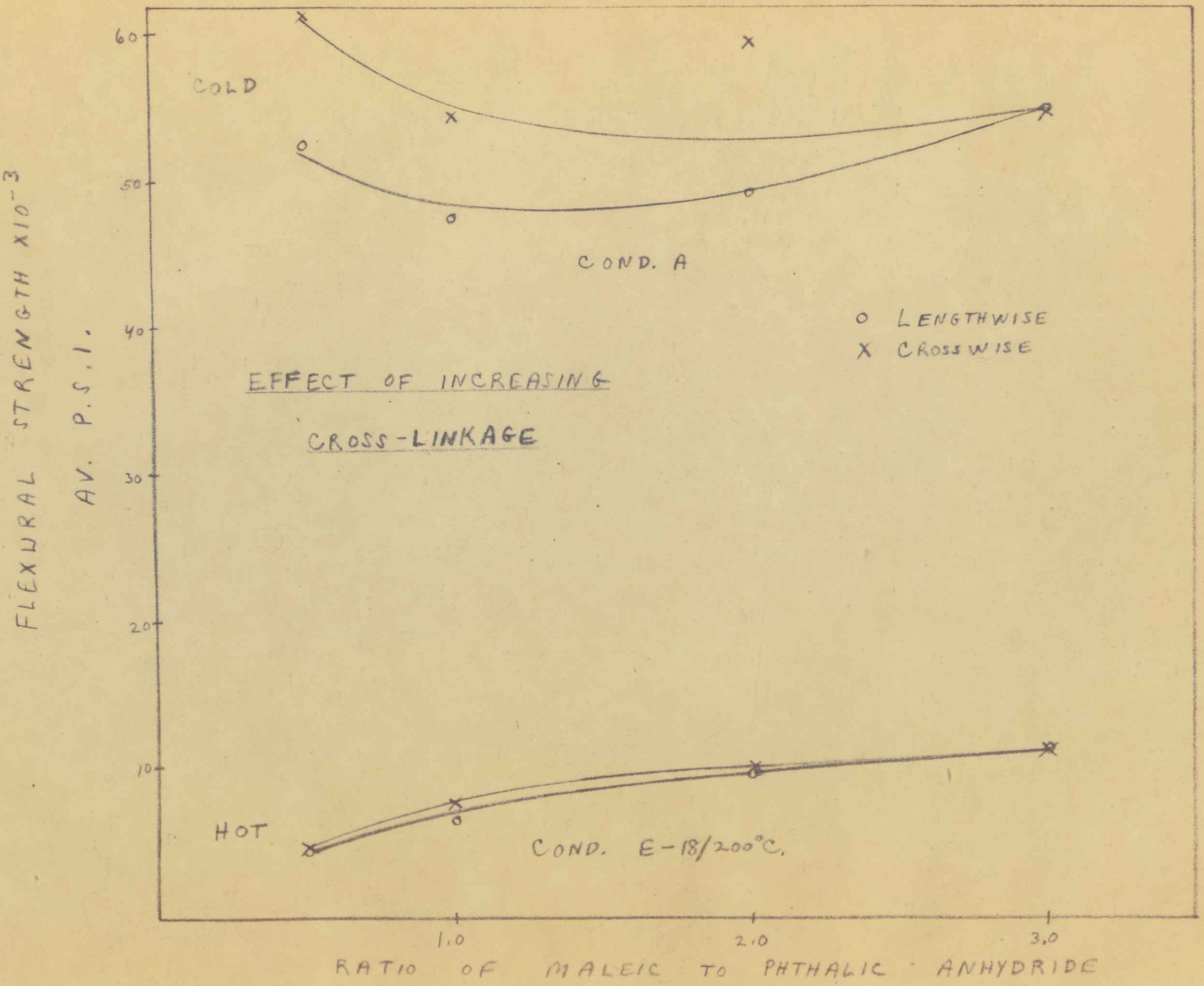
FLEXURAL STRENGTH X10⁻³
AV. P.S.I.
60
50
40
30
20
10
COLD
COND. A
o LENGTHWISE
X CROSSWISE
EFFECT OF INCREASING
CROSS-LINKAGE
HOT
COND. E-18/200°C.
1.0
2.0
3.0
RATIO OF MALEIC TO PHTHALIC ANHYDRIDE

FIG. 2

TABLE III

Resin	XIII	XIV	VI-S	VI-D	XV	XVI	XVII	XVIII
Acid	Maleic	Maleic	Maleic	Maleic	Maleic	Maleic	Maleic	Maleic
Moles	2.0	2.0	2.5	2.5	1.0	2.0	3.0	1.0
Acid	Phthalic	Phthalic	Phthalic	Phthalic	Phthalic	Phthalic	Phthalic	Phthalic
Moles	1.0	1.0	1.5	1.5	1.0	1.0	1.0	2.0
Glycol	Propylene	Propylene	Propylene	Ethylene	Propylene	Propylene	Propylene	Propylene
Moles	3.3	3.3	4.8	4.8	2.2	3.3	4.4	3.3
Monomer	DAP 50%	Styrene 33%	Styrene 23%	DAP 23%	Styrene 25%	Styrene 25%	Styrene 25%	Styrene 25%
Flex. Str.								
Cond. A (Av. PSI)								
L.W.	47,100	43,400	--	--	47,700	49,300	55,000	52,500
C.W.	44,600	41,300	--	--	54,200	59,400	54,800	61,200
E-18/200 (Av. PSI)								
L.W.	11,000	10,900	--	--	6,430	9,670	9,950	4,040
C.W.	10,500	10,140	--	--	7,510	9,920	11,780	4,350
Arc Resistance								
Cond. A (sec)	142-165	137-156	--	--	134-182	133-170	142-181	128-152
Percent Flex. Str. Retained								
L.W.	23.4	25.1	--	--	13.5	19.6	18.1	7.7
C.W.	23.5	24.6	--	--	13.9	16.7	11.7	7.1

The terephthalic and isophthalic acids were available as the dimethyl esters. Therefore, an ester interchange was performed with the glycol replacing the methanol. The terephthalate resin was insoluble in styrene, therefore, it was partially dissolved in DAP monomer to give a cloudy solution (VII-D). The extremely high viscosity did not allow a good laminate to be prepared although tests were made on the best portion of the laminate. The viscosity was lowered by preparing a solvent solution of the resin and making a laminate by drying out the solvent after coating (VII-D solvent). A very low, cold flexural strength was realized due either to incomplete solvent removal or insufficient cure, although the strength after aging was in the same order of magnitude as the better maleic-phthalic values.

Diallyl phthalate monomer appears to be a better solvent than monostyrene for most of the polyester resins. Ethylene glycol resins are usually more difficult to dissolve than their diethylene or propylene glycol counterparts. In a like manner, terephthalic polyester resins are less soluble than the corresponding isophthalic and phthalic acid resins. These are qualitative observations and are not based on any actual solubility determinations.

Additional resins were prepared with the isophthalic rather than the terephthalic acid. Resin IX is somewhat equivalent to Resin IV in Table III except that half of the phthalic was replaced by isophthalic acid. The test results were very similar, showing no significent improvement with the addition of isophthalic acid.

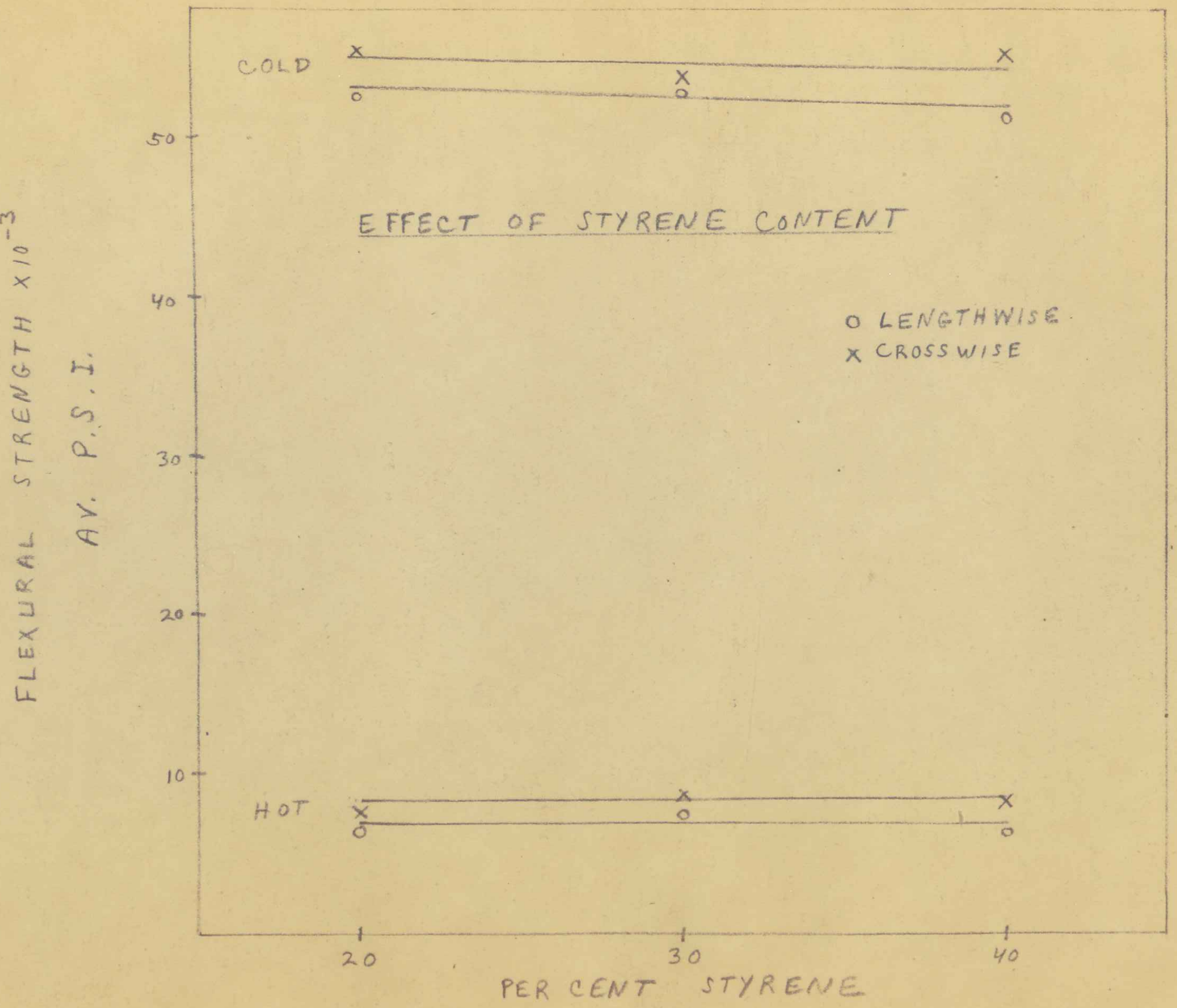
EFFECT OF STYRENE CONTENT
o LENGTHWISE
x CROSSWISE
COLD
HOT
FLEXURAL STRENGTH x10^-3
AV. P.S.I.
50
40
30
20
10
20
30
40
PER CENT STYRENE

FIG. 3

TABLE IV

Resin	VII-D	VII-D (Solvent)	VIII	IX	XIX	XX	XXI	X
Acid	Terephthalic	--	PMA*	Isophthalic	Isophthalic	Isophthalic	Isophthalic	Isophthalic
Moles	0.5	--	0.5	1.0	1.0	1.0	1.0	0.6
Acid	Phthalic	--	Maleic	Phthalic	Maleic	Maleic	Maleic	Maleic
Moles	0.5	--	1.5	1.0	1.0	1.0	1.0	1.4
Acid	Maleic	--	--	Maleic	--	--	--	--
Moles	1.0	--	--	2.0	--	--	--	--
Glycol	Ethylene	--	Ethylene	Ethylene	Propylene	Propylene	Propylene	Propylene
Moles	2.2	--	2.7	4.4	2.2	2.2	2.2	2.2
Monomer	DAP 23%	--	--	DAP 30%	Styrene 20%	Styrene 30%	Styrene 40%	DAP 30%
Flex. Str.								
Cond. A (Av. PSI) L.W.	45,700	23,800	--	46,800	52,500	52,800	51,400	43,100
C.W.	61,500	17,050	--	50,200	55,400	53,700	55,200	49,400
E-18/200 (Av. PSI) L.W.	8,190	1,023	--	6,620	6,200	7,420	6,150	11,100
C.W.	8,070	9,880	--	6,210	7,360	8,730	8,150	13,400
Arc Resistance								
Cond. A (sec)	83-147	127-140	--	136-154	135-182	30-168	27-174	134-165
Percent Flex. Str. Retained L.W.	17.9	43.0	--	14.1	11.8	14.1	12.0	15.8
C.W.	13.1	57.8	--	12.4	13.3	16.2	14.8	17.1

*PMA is abbreviation for pyromellitic acid.

Summary

The heat resistance of phthalic-maleic polyester resins is essentially the same as isophthalic-maleic polyesters. A terephthalic-maleic acid resin also showed similar properties. All of these types are somewhat inferior to the chlorendic (HET)-maleic polyesters.

An optimum resistance to heat is attained with a ratio of five moles of maleic anhydride to three moles of chlorendic acid. The phthalic resins above show a slowly increasing resistance to heat without a maximum as the amount of maleic anhydride is increased. This is explainable as being due to increased density of cross-linkages. The maximum obtained with the chlorendic acid, however, is not as easily explained and is probably due to a steric effect causing greater stability of polymeric structure.

The highest flexural strength is obtained with resins made with the shortest glycol, namely, ethylene glycol, or combination of it with diethylene glycol. Ethylene glycol resins maintain their relatively higher flexural strength after heat aging, but the relative drop in strength is the same as for the higher glycols. Thus, there is no apparent effect on heat resistance by varying the glycols investigated, although a stronger resin results before and after heating by using ethylene glycol.

The styrene content, between 20% and 40% by weight, seems to have little effect on the systems studied. The small amount of work done comparing diallyl phthalate monomer with styrene indicated a higher initial cold flexural strength and a larger drop in strength during heat aging than occurred with styrene.

The arc resistance of ordinary polyester resin combinations using phthalic and maleic anhydride with any of the ordinary glycols is very good, ranging from 130 to 180 seconds. Increasing the styrene percentage tends to lower the arc resistance, while introduction of a chlorinated acid destroys the arc resistant properties.

APPENDIX

All of the test results as given below refer to the initial column headings in order to conserve space. The first set of readings is the Condition A (no special conditioning) room temperature flexural strength; the second set of readings if the flexural strength at 200°C. after condition E-18/200 (18 hrs. at 200°C.). Details of these and the arc resistance test are given under Procedure.

Resin II

Condition A - Flexural Strength at Room Temperature

	Lengthwise			Crosswise		
	Width	Thickness	Load	Width	Thickness	Load
	1.005"	0.119"	160.0 lbs.	1.004"	0.118"	167.5 lbs.
	1.005"	0.119"	188.5 lbs.	1.001"	0.119"	184.0 lbs.
	1.005"	0.118"	204.0 lbs.	1.002"	0.119"	167.0 lbs.
	--	--	--	1.003"	0.118"	159.0 lbs.
Av.	1.005"	0.119"	184.2 lbs.	1.003"	0.119"	169.4 lbs.
Av. PSI			38,800			35,800
Min. PSI			33,700			33,600

Condition E-18/200 - Flexural Strength at 200°C.

	Lengthwise			Crosswise		
	Width	Thickness	Load	Width	Thickness	Load
	1.003"	0.118"	25.5 lbs.	1.002"	0.117"	23.5 lbs.
	1.004"	0.117"	24.5 lbs.	1.003"	0.117"	21.5 lbs.
	1.004"	0.118"	22.0 lbs.	1.003"	0.117"	21.5 lbs.
	--	--	--	1.004"	0.117"	21.5 lbs.
Av.	1.004"	0.118"	24.0 lbs.	1.003"	0.117"	22.0 lbs.
Av. PSI			5,150			4,800
Min. PSI			4,720			4,690

Arc Resistance - Condition A - 127, 5, 10, 3 seconds

Resin III

Condition A

	0.973"	0.119"	213.0 lbs.	0.958"	0.120"	163.0 lbs.
	1.033"	0.117"	237.0 lbs.	0.968"	0.122"	220.5 lbs.
	0.981"	0.118"	192.0 lbs.	1.002"	0.121"	210.0 lbs.
	1.029"	0.117"	251.5 lbs.	---	---	---
Av.	1.004"	0.118"	223.4 lbs.	0.976"	0.121"	197.8 lbs.
Av. PSI			47,900			41,500
Min. PSI			42,100			35,500

Condition E-18/200

				1.005"	0.123"	53.5 lbs.
	1.006"	0.119"	68.5 lbs.	1.004"	0.121"	93.5 lbs.
	1.006"	0.121"	109.5 lbs.	1.006"	0.122"	91.0 lbs.
	1.006"	0.125"	130.0 lbs.	1.006"	0.124"	40.0 lbs.
Av.	1.006"	0.121"	102.7 lbs.	1.005"	0.123"	69.5 lbs.
Av. PSI			20,900			13,700
Min. PSI			14,400			7,800

Arc Resistance - 19, 5, 415, 4 seconds

Resin IV

Condition A

	1.002"	0.116"	213.5 lbs.	1.005"	0.119"	210.0 lbs.
	1.002"	0.117"	193.0 lbs.	1.005"	0.118"	197.0 lbs.
	1.005"	0.119"	222.5 lbs.	1.005"	0.118"	190.5 lbs.
	1.005"	0.119"	210.5 lbs.	1.006"	0.117"	184.0 lbs.
Av.	1.004"	0.118"	209.9 lbs.	1.005"	0.118"	195.4 lbs.
Av. PSI			45,000			41,900
Min. PSI			42,300			39,500

Condition E-18/200

	1.004"	0.116"	43.5 lbs.	1.001"	0.115"	41.0 lbs.
	1.005"	0.117"	44.5 lbs.	1.003"	0.116"	36.5 lbs.
	1.004"	0.117"	44.0 lbs.	1.004"	0.117"	38.0 lbs.
	1.004"	0.117"	51.5 lbs.	1.004"	0.115"	45.0 lbs.
Av.	1.004"	0.117"	45.9 lbs.	1.003"	0.116"	40.1 lbs.
Av. PSI		10,000			8,900	
Min. PSI		9,610			8,100	

Arc Resistance - Condition A - 9, 183, 23, 4, 10 seconds

Condition E-18/200 - 177, 120, 182, 7, 6, 5, 18 seconds

Resin V

Condition A

				0.980"	0.120"	150.0 lbs.
	0.976"	0.118"	167.0 lbs.	0.981"	0.121"	140.0 lbs.
	0.979"	0.116"	157.5 lbs.	0.980"	0.120"	141.5 lbs.
	0.979"	0.116"	154.0 lbs.	0.981"	0.120"	135.5 lbs.
Av.	0.978"	0.117"	159.5 lbs.	0.981"	0.120"	141.8 lbs.
Av. PSI		35,800			30,200	
Min. PSI		34,600			28,800	

Condition E-18/200

	0.978"	0.118"	51.5 lbs.	0.979"	0.121"	36.5 lbs.
	0.979"	0.117"	43.0 lbs.	0.977"	0.120"	40.5 lbs.
	0.979"	0.117"	45.5 lbs.	0.979"	0.120"	43.5 lbs.
	--	--	--	0.977"	0.119"	42.5 lbs.
Av.	0.979"	0.117"	46.7 lbs.	0.978"	0.120"	40.8 lbs.
Av. PSI		10,460			8,700	
Min. PSI		9,630			7,630	

Arc Resistance - 127.6, 120.0, 82.5, 42.0, 3.2 seconds

Resin V

Laminate Postcured 1 Hour at 105°C.

	0.975"	0.119"	214.0 lbs.	0.976"	0.118"	189.5 lbs.
	0.979"	0.115"	215.5 lbs.	0.977"	0.119"	203.5 lbs.
	0.977"	0.119"	209.0 lbs.	0.976"	0.118"	188.0 lbs.
	--	--	--	0.976"	0.118"	188.0 lbs.
Av.	0.977"	0.118"	212.8 lbs.	0.977"	0.118"	194.4 lbs.
Av. PSI			47,100			43,100
Min. PSI			46,200			41,700

Condition E-18/200

	0.976"	0.118"	63.0 lbs.	0.977"	0.117"	49.0 lbs.
	0.975"	0.117"	64.5 lbs.	0.976"	0.117"	50.0 lbs.
	0.978"	0.117"	74.0 lbs.	0.977"	0.118"	53.0 lbs.
	--	--	--	0.976"	0.117"	57.5 lbs.
Av.	0.976"	0.117"	67.2 lbs.	0.977"	0.117"	52.4 lbs.
Av. PSI			15,100			11,770
Min. PSI			13,900			11,000

Arc Resistance - 6.3, 10.0, 5.2, 7.4, 6.2 seconds.

Resin VII

(Condition A)

	1.007"	0.145"	321.0 lbs.	1.010"	0.139"	406.0 lbs.
	--	--	--	1.007"	0.144"	425.5 lbs.
Av.	1.007"	0.145"	321.0 lbs.	1.008"	0.142"	415.8 lbs.
Av. PSI			45,700			61,500

Condition E-18/200

	1.018"	0.138"	54.5 lbs.	1.005"	0.141"	63.0 lbs.
	1.003"	0.140"	58.0 lbs.	1.003"	0.141"	44.5 lbs.
	1.005"	0.139"	47.0 lbs.	--	--	--
Av.	1.009"	0.139"	53.2 lbs.	1.004"	0.141"	53.7 lbs.
Av. PSI			8,190			8,070
Min. PSI			7,260			6,690

Arc Resistance - 142.0, 141.0, 83.2, 147.2, 141.8 seconds

Resin VII

Solvent Solution

Condition A

	1.005"	0.121"	117.5 lbs.	1.004"	0.124"	81.5 lbs.
	1.002"	0.122"	115.5 lbs.	1.004"	0.120"	91.5 lbs.
	1.004"	0.121"	116.0 lbs.	1.002"	0.122"	80.0 lbs.
	--	--	--	1.003"	0.124"	92.0 lbs.
Av.	1.004"	0.121"	116.3 lbs.	1.004"	0.123"	86.3 lbs.
Av. PSI			23,800			17,050
Min. PSI			23,200			16,340

Condition E-18/200

	1.003"	0.116"	39.5 lbs.	1.003"	0.119"	44.0 lbs.
	1.003"	0.116"	43.0 lbs.	1.006"	0.119"	49.5 lbs.
	1.005"	0.117"	55.0 lbs.	1.003"	0.118"	47.0 lbs.
	1.007"	0.117"	50.0 lbs.	--	--	--
Av.	1.006"	0.117"	46.9 lbs.	1.004"	0.119"	46.8 lbs.
Av. PSI			1,023			9,880
Min. PSI			8,770			9,300

Arc Resistance - 126.8, 138.2, 130.5, 139.6, 134.0 seconds

Resin IX

Condition A

	0.997"	0.148"	380.5 lbs.	1.000"	0.155"	378.5 lbs.
	1.005"	0.151"	289.5 lbs.	1.007"	0.150"	409.5 lbs.
	1.003"	0.147"	356.0 lbs.	1.000"	0.152"	376.0 lbs.
Av.	1.001"	0.148"	342.0 lbs.	1.002"	0.152"	388.0 lbs.
Av. PSI			46,800			50,200
Min. PSI			37,800			48,900

Condition E-18/200

	1.001"	0.145"	45.5 lbs.	1.000"	0.150"	47.0 lbs.
	1.005"	0.146"	46.5 lbs.	1.003"	0.154"	47.0 lbs.
	1.001"	0.146"	49.5 lbs.	1.003"	0.153"	49.5 lbs.
Av.	1.002"	0.146"	47.2 lbs.	1.002"	0.152"	47.8 lbs.
Av. PSI			6,620			6,210
Min. PSI			6,490			5,920

Arc Resistance - 148.4, 136.0, 154.2, 148.2, 145.4 seconds

Resin X

Condition A

	1.006"	0.129"	223.5 lbs.	1.008"	0.121"	251.5 lbs.
	1.006"	0.123"	229.5 lbs.	1.009"	0.121"	235.5 lbs.
	1.006"	0.123"	229.0 lbs.	--	--	--
Av.	1.006"	0.125"	225.9 lbs.	1.008"	0.121"	242.8 lbs.
Av. PSI			43,100			49,400
Min. PSI			40,100			47,900

Condition E-18/200

	1.007"	0.124"	56.5 lbs.	1.002"	0.116"	70.0 lbs.
	1.008"	0.127"	56.0 lbs.	1.006"	0.119"	66.0 lbs.
	1.003"	0.127"	55.0 lbs.	1.004"	0.120"	62.0 lbs.
	1.005"	0.127"	67.5 lbs.	1.004"	0.121"	56.5 lbs.
Av.	1.006"	0.126"	58.8 lbs.	1.004"	0.119"	63.6 lbs.
Av. PSI			11,100			13,400
Min. PSI			10,200			11,500

Arc Resistance - 133.8, 151.4, 150.1, 140.0, 165.2 seconds

Resin XI

Condition A

	1.004"	0.125"	200.5 lbs.	1.002"	0.125"	217.5 lbs.
	1.003"	0.124"	197.0 lbs.	1.003"	0.122"	204.0 lbs.
	1.004"	0.126"	190.5 lbs.	1.002"	0.123"	202.5 lbs.
	0.997"	0.120"	199.0 lbs.	--	--	--
Av.	1.002"	0.124"	196.8 lbs.	1.002"	0.123"	208.0 lbs.
Av. PSI			38,300			41,200
Min. PSI			35,800			40,000

Condition E-18/200

	1.003"	0.127"	43.0 lbs.	1.002"	0.123"	51.5 lbs.
	1.003"	0.121"	42.0 lbs.	1.002"	0.122"	46.5 lbs.
	1.003"	0.123"	51.0 lbs.	1.002"	0.122"	46.0 lbs.
	1.003"	0.126"	42.0 lbs.	1.003"	0.120"	43.0 lbs.
Av.	1.003"	0.124"	44.5 lbs.	1.002"	0.122"	46.8 lbs.
Av. PSI			8,650			9,400
Min. PSI			8,570			8,630

Arc Resistance - 7, 6, 5, 5, 3 seconds

Resin XII

Condition A

	1.003"	0.127"	259.5 lbs.	1.003"	0.124"	251.5 lbs.
	1.003"	0.116"	226.0 lbs.	1.002"	0.125"	276.0 lbs.
	1.002"	0.125"	263.0 lbs.	1.002"	0.126"	250.0 lbs.
	1.001"	0.114"	201.0 lbs.	--	--	--
Av.	1.002"	0.121"	237.4 lbs.	1.002"	0.125"	259.2 lbs.
Av. PSI			48,500			49,600
Min. PSI			46,300			48,800

Condition E-18/200

	1.002"	0.122"	63.0 lbs.	0.999"	0.126"	61.5 lbs.
	0.994"	0.127"	65.5 lbs.	1.002"	0.124"	73.0 lbs.
	1.002"	0.122"	64.5 lbs.	1.000"	0.123"	75.0 lbs.
	1.003"	0.120"	72.5 lbs.	1.002"	0.123"	68.0 lbs.
Av.	1.000"	0.123"	66.4 lbs.	1.001"	0.124"	69.4 lbs.
Av. PSI			13,160			13,500
Min. PSI			12,260			11,620

Arc Resistance - 9, 6, 4, 3, 4 seconds

Resin XIII

Condition A

	1.004"	0.117"	212.0 lbs.	1.007"	0.116"	200.0 lbs.
	1.005"	0.117"	216.0 lbs.	1.007"	0.117"	223.0 lbs.
	1.004"	0.114"	208.0 lbs.	1.007"	0.116"	201.0 lbs.
	--	--	--	--	--	--
Av.	1.004"	0.116"	212.0 lbs.	1.007"	0.117"	205.8 lbs.
Av. PSI			47,100			44,600
Min. PSI			46,200			43,400

Condition E-18/200

	1.004"	0.116"	51.0 lbs.	1.007"	0.118"	54.0 lbs.
	1.003"	0.116"	50.0 lbs.	1.005"	0.117"	48.5 lbs.
	1.002"	0.116"	47.5 lbs.	1.006"	0.117"	45.0 lbs.
	--	--	--	1.006"	0.117"	45.0 lbs.
Av.	1.003"	0.116"	49.5 lbs.	1.006"	0.117"	48.1 lbs.
Av. PSI			11,000			10,500
Min. PSI			10,540			9,800

Arc Resistance - 142, 161, 165, 160 seconds

Resin XIV

Condition A

	1.006"	0.117"	204.0 lbs.	1.003"	0.117"	204.0 lbs.
	1.006"	0.116"	202.5 lbs.	1.003"	0.117"	185.5 lbs.
	1.006"	0.117"	191.0 lbs.	1.004"	0.116"	176.0 lbs.
	--	--	--	1.005"	0.118"	191.5 lbs.
Av.	1.006"	0.117"	199.2 lbs.	1.004"	0.117"	189.3 lbs.
Av. PSI			43,400			41,300
Min. PSI			41,600			39,100

Condition E-18/200

	1.004"	0.115"	48.5 lbs.	1.004"	0.116"	48.0 lbs.
	1.005"	0.114"	48.0 lbs.	1.004"	0.116"	47.0 lbs.
	1.006"	0.115"	48.5 lbs.	1.003"	0.117"	46.0 lbs.
	--	--	--	1.003"	0.116"	42.0 lbs.
Av.	1.005"	0.115"	48.3 lbs.	1.004"	0.116"	45.8 lbs.
Av. PSI			10,900			10,140
Min. PSI			10,900			9,320

Arc Resistance - 145, 147.5, 156, 137 seconds

Resin XV

Condition A

	0.990"	0.111"	225.5 lbs.	1.006"	0.117"	262.5 lbs.
	0.989"	0.118"	221.5 lbs.	1.004"	0.116"	243.5 lbs.
	1.002"	0.120"	228.0 lbs.	1.003"	0.116"	250.0 lbs.
	1.002"	0.122"	222.0 lbs.	1.004"	0.118"	256.0 lbs.
Av.	0.996"	0.118"	224.2 lbs.	1.002"	0.117"	253.0 lbs.
Av. PSI			47,700			54,200
Min. PSI			44,500			53,900

Condition E-18/200

	1.002"	0.115"	27.5 lbs.	1.005"	0.116"	36.0 lbs.
	1.002"	0.120"	29.5 lbs.	1.002"	0.119"	32.0 lbs.
	1.002"	0.119"	31.5 lbs.	1.004"	0.120"	37.0 lbs.
	1.002"	0.115"	29.5 lbs.	1.004"	0.119"	36.0 lbs.
Av.	1.002"	0.117"	29.5 lbs.	1.004"	0.118"	35.0 lbs.
Av. PSI			6,430			7,510
Min. PSI			6,210			6,740

Resin XVI

Condition A

	1.008"	0.117"	240.0 lbs.	1.005"	0.119"	295.0 lbs.
	1.004"	0.118"	208.0 lbs.	1.006"	0.122"	278.5 lbs.
	1.006"	0.113"	217.5 lbs.	1.006"	0.119"	288.5 lbs.
	1.007"	0.112"	207.5 lbs.	--	--	--
Av.	1.006"	0.115"	218.2 lbs.	1.006"	0.120"	287.0 lbs.
Av. PSI			49,300			59,400
Min. PSI			44,600			53,800

Arc Resistance - 136.0, 135.8, 148.6, 169.7, 133.4 seconds

Resin XVII

Condition A

	1.005"	0.122"	296.0 lbs.	1.004"	0.126"	288.5 lbs.
	1.004"	0.124"	277.0 lbs.	1.005"	0.120"	243.0 lbs.
	1.005"	0.122"	276.0 lbs.	1.004"	0.121"	291.5 lbs.
	1.005"	0.122"	265.5 lbs.	--	--	--
Av.	1.005"	0.123"	278.6 lbs.	1.004"	0.122"	273.3 lbs.
Av. PSI			55,000			54,800
Min. PSI			53,200			50,300

Condition E-18/200

	0.999"	0.127"	55.0 lbs.	1.008"	0.122"	59.0 lbs.
	1.002"	0.124"	52.0 lbs.	0.996"	0.117"	53.5 lbs.
	1.002"	0.126"	53.0 lbs.	1.005"	0.119"	55.0 lbs.
	1.003"	0.129"	54.5 lbs.	--	--	--
Av.	1.002"	0.127"	53.6 lbs.	1.003"	0.119"	55.8 lbs.
Av. PSI			9,950			11,780
Min. PSI			9.810			11,760

Arc Resistance - 176.2, 180.5, 155.2, 179.0, 142.0 seconds

Resin XVIII

Condition A

	1.001"	0.125"	274.5 lbs.	1.007"	0.132"	367.0 lbs.
	1.006"	0.127"	272.5 lbs.	1.004"	0.129"	332.0 lbs.
	1.007"	0.127"	278.5 lbs.	1.004"	0.127"	324.5 lbs.
	1.006"	0.127"	309.0 lbs.	--	--	--
Av.	1.005"	0.127"	283.6 lbs.	1.005"	0.129"	341.1 lbs.
Av. PSI			52,500			61,200
Min. PSI			50,400			60,000

Condition E-18/200

	1.008"	0.126"	22.0 lbs.	1.006"	0.125"	23.5 lbs.
	1.006"	0.126"	21.5 lbs.	1.002"	0.125"	21.0 lbs.
	1.006"	0.128"	21.0 lbs.	1.003"	0.130"	26.0 lbs.
	1.008"	0.123"	21.5 lbs.	--	--	--
Av.	1.007"	0.126"	21.5 lbs.	1.004"	0.127"	23.5 lbs.
Av. PSI			4,040			4,350
Min. PSI			3,820			4,010

Arc Resistance - 152.2, 132.2, 150.0, 128.8, 128.4 seconds

Resin XIX

Condition A

	1.007"	0.125"	293.5 lbs.	1.008"	0.118"	266.0 lbs.
	1.004"	0.126"	255.5 lbs.	1.006"	0.113"	247.5 lbs.
	1.007"	0.125"	290.0 lbs.	1.006"	0.116"	235.0 lbs.
	1.005"	0.125"	260.0 lbs.	--	--	--
Av.	1.006"	0.125"	274.8 lbs.	1.007"	0.116"	249.5 lbs.
Av. PSI			52,500			55,400
Min. PSI			48,100			52,100

Condition E-18/200

	1.006"	0.125"	33.5 lbs.	1.008"	0.120"	36.5 lbs.
	1.007"	0.121"	32.5 lbs.	1.007"	0.120"	34.5 lbs.
	1.005"	0.121"	30.0 lbs.	1.006"	0.119"	36.5 lbs.
	1.004"	0.125"	29.5 lbs.	1.004"	0.118"	32.0 lbs.
Av.	1.006"	0.123"	31.4 lbs.	1.006"	0.119"	34.9 lbs.
Av. PSI			6,200			7,360
Min. PSI			5,640			6,860

Arc Resistance - 180.6, 135.0, 182.0, 144.2, 138.4 seconds

Resin XX

Condition A

	1.007"	0.119"	237.5 lbs.	1.010"	0.122"	273.0 lbs.
	1.010"	0.119"	266.0 lbs.	1.007"	0.124"	274.5 lbs.
	1.006"	0.119"	227.0 lbs.	1.009"	0.120"	257.5 lbs.
	1.007"	0.118"	272.0 lbs.	--	--	--
Av.	1.008"	0.119"	250.6 lbs.	1.009"	0.122"	268.3 lbs.
Av. PSI			52,800			53,700
Min. PSI			47,700			53,200

Condition E-18/200

	1.004"	0.117"	32.0 lbs.	1.007"	0.116"	37.0 lbs.
	1.003"	0.116"	33.5 lbs.	1.009"	0.124"	43.5 lbs.
	1.004"	0.116"	34.5 lbs.	1.006"	0.114"	42.0 lbs.
	1.004"	0.116"	33.5 lbs.	1.006"	0.115"	37.5 lbs.
Av.	1.004"	0.116"	33.4 lbs.	1.007"	0.117"	40.0 lbs.
Av. PSI			7,420			8,730
Min. PSI			6,970			8,210

Arc Resistance - 147.2, 167.8, 143.2, 30.2, 164.0 seconds

Resin XXI

Condition A

	1.006"	0.118"	224.5 lbs.	1.008"	0.123"	254.5 lbs.
	1.006"	0.120"	257.5 lbs.	1.006"	0.125"	297.5 lbs.
	1.010"	0.120"	252.0 lbs.	1.008"	0.122"	290.0 lbs.
	1.008"	0.121"	261.5 lbs.	--	--	--
Av.	1.008"	0.120"	248.9 lbs.	1.007"	0.123"	280.7 lbs.
Av. PSI			51,400			55,200
Min. PSI			46,400			50,100

Condition E-18/200

	1.006"	0.127"	34.0 lbs.	1.005"	0.119"	41.0 lbs.
	1.003"	0.122"	30.5 lbs.	1.005"	0.121"	41.0 lbs.
	1.001"	0.120"	28.0 lbs.	1.008"	0.123"	49.5 lbs.
	1.006"	0.123"	32.0 lbs.	1.007"	0.125"	31.0 lbs.
Av.	1.004"	0.123"	31.1 lbs.	1.006"	0.122"	40.6 lbs.
Av. PSI			6,150			8,150
Min. PSI			5,830			5,920

Arc Resistance - 174.4, 26.8, 109.6, 142.4, 134.2 seconds

Resin - Bakelite BRSQ-142

Condition A

	0.961"	0.123"	232.5 lbs.	1.008"	0.119"	231.0 lbs.
	0.967"	0.123"	243.0 lbs.	0.986"	0.119"	218.5 lbs.
	1.011"	0.124"	253.0 lbs.	0.976"	0.120"	211.5 lbs.
Av.	0.980"	0.123"	242.8 lbs.	0.990"	0.119"	220.3 lbs.
Av. PSI			49,200			47,200
Min. PSI			48,200			45,200

Condition E-18/200

	1.007"	0.119"	92.5 lbs.	1.003"	0.121"	92.5 lbs.
	1.005"	0.119"	181.0 lbs.	1.004"	0.121"	118.0 lbs.
	1.006"	0.120"	169.5 lbs.	1.001"	0.120"	125.0 lbs.
	1.007"	0.121"	163.5 lbs.	--	--	--
Av.	1.006"	0.120"	151.6 lbs.	1.003"	0.121"	111.8 lbs.
Av. PSI			31,400			22,800
Min. PSI			19,500			18,900

Arc Resistance - 165.0, 169.6, 161.4, 175.0

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