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Second
Report on

Replacement of Tung Oil in Resins to be used in Navy Aeronautical Finishing Materials

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ABSTRACT

This report describes the progress made to date on the elimination of tung oil from Navy Aeronautical Specifications and is a continuation of the investigation described in NRL Report No. P-2141. It deals most specifically with the preparation of alkyd resins from dehydrated castor oil and other commercial oils to be used as substitutes for the tung oil containing alkyd, Rezyl 113, in P-27-b zinc chromate primer and AN-TT-P-656A zinc chromate primer.

In addition to this, it describes the preparation of larger quantities of several of the tung oil free dispersion resins described in NRL Report No. P-2141 to be used as substitutes for BK-3962.

Also included is the detailed preparation of the various tung oil free alkyds and data on the comparison of these with Rezyl 113 as a clear vehicle and of the final primers which were prepared from them as substitutes for Rezyl 113.

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INTRODUCTION

A. Authorization

1. This study was authorized by Bureau of Aeronautics letter dated 20 April 1942, Aer-E-2574 MVS1 F38(6-7); NP14, F38-2(12), and continued by a subsequent letter dated 9 July 1943, Aer-E-2577-MB/8-15-43 F38 (6-7).

B. Statement of the Problem

2. The objects of this study were (1) to investigate commercial substitutes of tung oil; (2) to synthesize oils in the laboratory which might be used for the same purpose; and, (3) to prepare from these oils alkyd and dispersion resins similar to Rezyl 113 and Bakelite BK-3962, respectively, for use in formulating AN-TT-P-656A zinc chromate primer eliminating tung oil from its composition.

3. This report is a supplement to NRL Report No. P-2141 of 6 August 1943 and mainly describes the preparation of alkyd and dispersion resins from various commercial oils and fatty acids and from several others prepared in this Laboratory.

C. Known Facts Bearing on the Problem

4. Tung oil is generally accepted to be the most nearly perfect drying oil attributable to the fact that it contains approximately 85% eleostearic acid which has three double bonds triply conjugated. No other oil found in nature contains such a high degree of conjugated unsaturation. Oiticica oil contains approximately 75% licanic acid which is similar to eleostearic in that it has three triply conjugated double bonds, but it also contains a ketone group which greatly detracts from its alkali and water resistance. In addition, oiticica oil is not sufficiently available at the present time to warrant its study here.

5. Dehydrated castor oil is probably the next best substitute for tung oil since in its composition it has from 20-30% of a conjugated diene 9, 11 linoleic acid. This structure makes for a shorter drying time and an increase in water and alkali resistance most nearly approximating tung oil although it does not equal it by any degree. Dehydrated castor oil is readily available in sufficient quantities to be considered satisfactory from the procurement aspect.

6. In addition to dehydrated castor oil there are on the market various other chemically treated vegetable oils which are claimed to possess faster drying time and improved water and alkali resistance. These oils are generally prepared from linseed, soya bean and other

readily available natural vegatable oils. They are given consideration along with dehydrated castor oil as possible substitutes for tung oil.

7. An extensive analysis is being carried out on all commercial oils available and results will be submitted in another report. It was on the basis of this analysis that the oils were chosen for this investigation.

METHOD

A. Plan of Investigation

8. The investigation put forth in this report deals with the preparation of alkyd resins which are tung oil free and in which an attempt is made to equal or surpass the alkyds containing tung oil which have been used in P-27-b zinc chromate primer, based on final performance of the finished product.

9. It must be remembered that these oils alone do not equal the desirable characteristics of tung oil, thus changes must be made in the other ingredients of the alkyd and also in the processing of the alkyd to compensate for the deficiency of the substituted oils.

10. In the preparation of an alkyd resin there are four variables that may be considered. They are, briefly:

a. Ratio of the phthalic anhydride to amount of fatty acid used.

b. Type of polyhydric alcohol employed.

c. Addition of carboxyl containing compounds other than fatty acids and phthalic anhydride.

d. Time, temperature and method of cooking.

11. It is known generally that esters made with pentaerythritol as the polyhydric alcohol which is itself insoluble in water, have better water resistance than those made with glycerine. Since all the hydroxyl groups on a molecule of pentaerythritol are in the primary position, the esterification reaction goes more regularly and easier than in the case of glycerine which contains one secondary hydroxyl. In addition to this fact, there are four hydroxyl groups attacked to the same structure; therefore, an alkyd resin or oil synthesized with pentaerythritol as the polyhydric alcohol would possess a larger and more complex molecular structure than those synthesized from glycerine. For these reasons pentaerythritol was used in comparison to glycerine in preparation of the alkyds in this discussion.

12. The ratio of fatty acids to phthalic anhydride was varied over a fairly wide range to decide at what combination the various constituents produced the best product. It has been found in general that the less reactive an oil from the standpoint of polymerization, the higher the percentage of phthalic anhydride that can be used per unit of oil and still carry the reaction to completion.

13. It is known from the patent literature that furylacrylic acid esterified on glycerine along with fatty acids gives a much harder and faster drying product than the oil itself. For this reason furylacrylic acid was used in this investigation to modify the various alkyds prepared.

14. The time and temperature of the cooks were varied with the different combinations of material used to obtain the most desirable finished resin. It was found in general that those oils and fatty acids containing a higher percentage of conjugated unsaturation could not be cooked as long or at as high a temperature as those containing less conjugated unsaturation.

15. In order to obtain a more complete esterification of the acids and get as low an acid value as possible, an excess of the theoretical molecular quantity of the polyhydric alcohol must be used. The reason for this has not been thoroughly determined but it is probably due to the mass action factor of the reaction; or to the fact that there is possibly some inter-reaction between the OH groups of the polyhydric alcohol forming an ether linkage and thereby blocking its reaction with a carboxyl group. In the case of glycerine, an excess over a range of 5-15% was tried and it was found that about 10% excess gave the best results. In the case of pentaerythritol, an excess over a range of from 10-25% was tried and it was found that about 15% excess gave the best results.

16. If too great an excess of the polyhydric alcohol is used, it decreases the water resistance of the final resin.

17. Each of the alkyd resins after preparation was substituted into the P-27-b zinc chromate primer formulation in place of Rezyl 113 and prepared in the usual manner on the ball mill or roller mill. After preparation of the primers, the specification tests were run on them using regular AN-TT-P-656A zinc chromate primer as a control.

18. In the case of the dispersion resins, large batches were made of NRL Dispersion Resin Nos. 3, 6 and 7, described in NRL Report No. P-2141 dated 6 August 1943, Appendix A. As it was shown in that study, these dispersion resins seemed to show the most promise as possible tung oil free substitutes for BK-3962. The larger batches were prepared in order to obtain enough material for further grinding and testing of these

dispersion resins in place of BK-3962 in the P-27-b formulation.

B. Materials

19. The materials used in preparation of the alkyd resins are shown in Tables I, II and III. The Castung special fatty acids were prepared as described in paragraph 18 of NRL Report No. P-2141, and the furylacrylic acid was prepared as described in paragraph 20 of that same reference. All the other materials were obtained commercially.

20. The materials used in the preparation of the dispersion resins are shown in Table IV of this report.

C. Methods of Preparation of Materials

21. As has been explained previously, the work described herein is largely a continuation of that described in NRL Report No. P-2141. In that report paragraph 15 describes a typical reaction chart of one of the alkyd resins prepared. The description of the other alkyd resins made up to that time are given in Appendix B of the same report.

22. There are two generally known methods of preparing an alkyd resin. They are:

a. Solvent process method.
b. Inert gas method.

The solvent method consists of cooking the material in a 3-neck round bottom flask equipped with a seal stirrer, a reflux condenser connected with a water trap, and a sampling device. From 5-15% xylene or similar solvent is added to the material in the beginning and filled into the water trap. As refluxing begins and water starts coming off, it refluxes with the xylene and is caught in the water trap displacing the xylene therein. The xylene goes back into the flask and maintains a constant reflux. This makes it possible to maintain a constant boiling temperature and at the same time collect the water given off from the reaction. Any temperature desired can be obtained by drawing off or adding xylene to the trap. Its advantages are:

a. Ease of control of temperature,
b. Ease of collecting water given off.
c. No loss of phthalic anhydride.
d. Ease of thinning operation of final product.

A picture of the setup used for cooking an alkyd resin by the solvent process is shown in Plate I.

23. The inert gas method consists of cooking the material in an open container equipped with an apparatus for passing an inert gas, such as CO_2 or N_2, through the materials during the entire cooking process. This serves as a means of removing the water produced by the reaction and also produces a blanketing action which prevents oxidation of the material. Its advantages are:

a. Simplicity of cooking. Can be cooked in most any type of open container since no reflux condenser is required.

b. Somewhat lower acid values obtained on final product due to the removing of the excess acid by the blowing process.

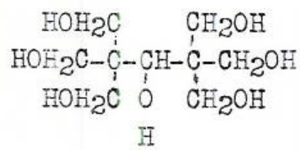
Both of these methods were employed in this investigation.

24. When an alkyd resin is prepared from the oil directly and glycerine is used as the polyhydric alcohol, the oil must be alcoholized to a monoglyceride before the phthalic anhydride is added. This is accomplished by cooking the oil with an equivalent amount of glycerine (i.e., 1 mol oil to 2 mols of glycerine) using litharge as an alcoholysis catalyst. About 0.2% to 0.5% of litharge based on the weight of the oil is used. The cook is carried out at about 200°C - 210°C and alcoholysis is considered complete when one part of the product is soluble in two and one-half parts of methyl alcohol. After the monoglyceride is prepared, the phthalic anhydride is added and the cook is carried out as described in paragraph 22 above.

25. When using pentaerythritol as the polyhydric alcohol, fatty acids rather than oil must be used and all the ingredients are added together in the beginning. The cooking procedure is carried out from the start as described in paragraph 22.

26. The system of designation used in identifying the resins prepared is as follows. The first letters designate the type of product, i.e., (AR indicates alkyd resin); the first number indicates the formulation used; the second number indicates variation of that formulation, and the third number indicates the batch number.

27. The first alkyd resin to be reported on in this discussion would correspond to Alkyd #6 in the series as reported in NRL Report No. P-2141 or AR6-0-1 by the new system of identification now used in this Laboratory. The purpose in cooking this alkyd was to determine whether anhydroenoheptite,



(prepared by Cannizzaro reaction from acetone and formaldehyde) could be used as the polyhydric alcohol in place of glycerine. The formulation and cooking log are given in Appendix A. It was found that anhydroenoheptite did not esterify well and seemed to decompose within itself. The final product was unsatisfactory.

28. Alkyd resins AR8-0-1, AR9-0-1 and AR10-0-1 were prepared mainly to determine the highest percentage of phthalic anhydride that could be used in relation to dehydrated castor fatty acids with pentaerythritol as the polyhydric alcohol and also to determine the effect of a small percentage of furylacrylic acid. The linseed fatty acids were used to slow down the polymerization rate enough so that the reaction could be taken to completion. AR8-0-1 and AR9-0-1 were found to body very fast making the final product very viscous when thinned. They also had rather high acid values. In AR10-0-1 the temperature range was increased to determine the effect produced. This resulted in the product jelling before the reaction was complete.

29. Alkyds AR11-0-1, AR12-0-1, AR13-0-1, AR14-0-1 and AR15-0-1 were prepared by the inert gas method to determine any effect on the final product. In addition, the linseed fatty acids content was varied over a wide range in this series to determine the quantity yielding the best results. AR11-0-1 jelled before the reaction was complete, probably because it contained no linseed fatty acids to retard the reaction. AR12-0-1, AR13-0-1 and AR14-0-1 all cooked satisfactorily, but the one containing the most linseed oil required the longest time to react and possessed the lowest viscosity. From this series it was also deduced that the inert gas system of cooking, although it produced materials with somewhat lower acid values, was not as satisfactory as the solvent method because much phthalic anhydride sublimed off during the operation and greater difficulty was encountered in controlling the temperature. From here on in the investigation the solvent method was used exclusively.

30. In Alkyds AR16-0-1 and AR17-0-1 the same percentages of phthalic anhydride, dehydrated castor fatty acids, linseed fatty acids, and furylacylic acid were used, the only variable being that pentaerythritol was employed as the polyhydric alcohol in the former and glycerine in the latter. This was to determine whether there was any advantage in pentaerythritol over the glycerine, all other things being equal.

31. Alkyds AR2-0-2 and AR2-0-3 were large batches of the original Alkyd #2 as described in NRL Report No. P-2141. They were cooked under exactly the same conditions as the original for the purposes of determining control and of producing enough material for grinding into primers for testing purposes. In a like manner, AR3-0-2 and AR3-0-3 were large batches of Alkyd #3 formulation in NRL Report No. P-2141 and AR4-0-2 a large batch of the original Alkyd #4 formulation also described in that report.

32. Alkyds AR8-0-2, AR9-0-2, AR16-0-2 and AR17-0-2 are large batches of AR8-0-1, AR9-0-1, AR16-0-1 and AR17-0-1, respectively, prepared for the same purpose as described above.

33. Alkyd AR2-0-4 is the same formulation as the original Alkyd #2 in NRL Report No. P-2141, the difference being that the reaction reaction temperature was somewhat increased. Due to this change its final acid value was somewhat lower. AR2-0-5 is a repetition of this cook for purposes of determining control.

34. On examining the formulation of the original Alkyd #3 formulation as described in Appendix B of NRL Report No. P-2141 and AR3-0-2 and AR3-0-3 in Appendix A of this report, it is found that this alkyd contains only phthalic anhydride, dehydrated castor acids and pentaerythritol. The purpose of the #3 series was to determine the relative results using only dehydrated castor fatty acids and pentaerythritol as against some of the other alkyds prepared in this investigation containing linseed oil and furylacrylic acid. Alkyd AR3-1-1 and AR3-2-1 are variations of the original Alkyd #3, AR3-0-2 and AR3-0-3. In this case the percentage of phthalic anhydride was decreased and the percentage of dehydrated fatty acid increased. AR3-1-1 jelled before the reaction was complete. AR3-2-1 was cooked at a much lower temperature and was quite satisfactory, having a much lower acid value and not so high a viscosity. Later AR3-3-1 was prepared having a phthalic anhydride percentage composition and dehydrated castor percentage composition which came in between the original Alkyd #3 formulation and AR3-1-1 and AR3-2-1. This was also satisfactory but still considerably lower than the original Alkyd #3.

35. Resins AR18-0-1, AR19-0-1 and AR19-0-2, AR20-0-1 and AR20-0-2, AR21-0-1 and AR21-0-2, AR22-0-1, AR23-0-1 and AR29-0-1 were all cooked from commercial drying oils, Dryinol, RD-43-26, RD-43-27, Falkowood, GF Oil, T-H Oil, Castung Special and K-Oil, respectively. The commercial concerns from which these oils were obtained are shown in Table I. Since these materials were in the form of oils, they had to be alcoholized to the mono-glyceride before being cooked into the alkyd as described in paragraph 24. In all cases the same percentage of phthalic anhydride was used. This was done so that a direct comparison could be made between the final products. Glycerine had to be used in all cases since the oils were, in most cases, glycerides. These oils were chosen upon the investigation of around 34 commercial oils because they had the most suitable physical and chemical constants to be used for the manufacture of alkyds.

36. AR18-0-1 of this series jelled before completion of the reaction. This was probably due to the fact that the original viscosity of Dryinol was so high that it did not allow time for further polymerization. AR19-0-1 was found to be very heavy and to possess a high acid value on

completion. This same formulation was cooked a second time as AR19-0-2 with a change in time and temperature but still found to be unsatisfactory due to high acid value. Falkowood oil was thus eliminated as a possibility. AR22-0-1, AR-23-0-1 and AR29-0-1 were all prepared with satisfactory results. AR19-0-3, AR22-0-2 and AR23-1-1 are large batches, respectively, of AR19-0-1, AR22-0-1 and AR23-0-1 prepared to obtain enough material for grinding and testing purposes. AR23-1-1 in this case had a slightly larger excess of glycerine than AR23-0-1.

37. AR31-0-1 is a straight glycerol alkyd containing only Castung special dehydrated castor oil, phthalic anhydride and glycerine. This resin was made for the purpose of comparing a glycerol alkyd made in the usual manner by the alcoholysis of the dehydrated castor oil and then cooked into an alkyd with the alkyds made from dehydrated castor acids with pentaerythritol as the polyhydric alcohol.

38. Resins AR12-1-1, AR14-1-1 and AR15-1-1 were large batches, respectively, of AR12-0-1, AR14-0-1 and AR15-0-1. They differed from the originals in the respect that they were prepared by the solvent method rather than the inert gas method.

39. Alkyd AR21-1-1 was prepared using the same formulation as the original Alkyd #2 described in Appendix B of NRL Report No. P-2141 except that isoline dehydrated castor fatty acids were used instead of those derived from Castung dehydrated castor oil. It was found that this product could be cooked much longer under the same conditions than those using Castung special dehydrated castor fatty acids.

40. Alkyd AR8-1-1 is a variation of AR8-0-1 in that the percentage of phthalic anhydride was slightly reduced and the percentage of fatty acid increased. This seemed to give a better, more workable product with a lower acid value.

41. All of the alkyds prepared are shown in Table V giving the percentage of raw material used, the temperature range and the constants on the final product. The cooking log of each resin is given in detail in Appendix A of this report.

42. In Table VI is shown a comparison of the water resistance and drying times of the clear alkyds with that of Rezyl 113. This test was made for screening purposes to eliminate those materials which did not compare favorably with the control.

43. DISPERSION RESINS. DR8-0-2 is a large batch of NRL Dispersion Resin #3 described in NRL Report No. P-2141, Appendix A. DR10-0-2 and DR10-0-3 are large batches of NRL Dispersion Resin #6 described in NRL Report No. P-2141, Appendix A. DR11-0-2 and DR11-0-3 are large batches of NRL Dispersion Resin #7 as described in NRL Report No. P-2141,

Appendix A. These dispersions were cooked in a 2-gallon stainless steel kettle under the exact conditions of time and temperature as the originals. The final appearance of the products was the same as for the original batches. The preparation of these dispersion resins is described in detail in Appendix B of this report.

44. The control formula used in the specification test is the regular AN-TT-P-656A formula based on a 1-gallon batch.

Zinc Chromate	2.95 lbs.
Asbestine	.52
Maleic anhydride	.105
50% 501 Kopol	.52
Rezyl 113	2.85
Bakelite BK-3962	1.80
Xylene	2.04
Lead Nuodex 24%	.025
Cobalt Nuodex 6%	.01
Total	10.820 lbs.

The method of grinding on the ball mill was carried out in the following manner. All of the asbestine (.52 lbs.), .405 lbs. of Rezyl 113 and .23 lbs. of xylene were ground together for 120 hours on the ball mill. All of the zinc chromate (2.95 lbs.) maleic anhydride (.105 lbs.) Kopol 501 (.52 lbs.) and one-third of the total Rezyl 113 (.95 lbs.) with enough xylene to facilitate proper grinding were then ground together for 24 hours. These two batches were then mixed together and to this mix was added all of the BK-3962 (1.8 lbs.), the remainder of the alkyd (1.49 lbs.), the rest of the xylene and driers. This final mix was then ground for 4 hours to produce the finished primer. On the roller mill grind, all the asbestine (.52 lbs.) and an equal part by weight of Rezyl 113 (.52 lbs.) were run through the mill one time; all the rest of the material except the BK-3962, xylene and drier was mixed with the above grind and the whole mix run through the mill two times. The BK-3962 was then cold cut with the xylene and the drier added. This was then mixed thoroughly with the above grind to produce the final primer.

45. In the case of the experimental resins, the grinding was carried out in the same manner as was the control, the alkyd resin being substituted in each case for Rezyl 113 and the dispersion resins being substituted for BK-3962. Table VII indicates which resins were ground on the ball mill and which on the roller mill, and gives the results of the specification test as compared to the control.

DISCUSSION

46. In the foregoing investigation of possible substitutes for tung oil in the alkyd which is used in P-27-b or AN-TT-P-656A, it has been shown that a great deal depends on the manner in which the alkyd is prepared, the polyhydric alcohol used, and the ratio of substituted oils to the phthalic anhydride and other materials which go into the composition of the alkyd.

47. It has been shown that by using dehydrated castor acids alone and in combination with furylacrylic acid with pentaerythritol as the polyhydric alcohol, an alkyd can be prepared that has superior water resistance and improved drying time to the tung oil containing alkyd Rezyl 113 which is specified in P-27-b. This is shown in the case of AR3-3-1, AR3-0-2, AR2-0-2 and AR8-0-2. If pentaerythritol were used it would necessitate using fatty acids as a raw product rather than oil.

48. Of the glycerol alkyds using oils as the starting product, AR19-0-2 made from RD-43-26 and AR23-0-2 made from T-H oil, appeared the best in the final primers but did not nearly equal in performance the alkyds made from dehydrated castor fatty acids and pentaerythritol.

CONCLUSIONS AND RECOMMENDATIONS

49. From the final data in Table VII, it is found that AR8-0-2 and AR8-1-1, AR3-0-3 and AR3-3-1, AR2-0-5 and AR4-5-1 showed the best results when ground into a primer as a substitute for Rezyl 113. All of these alkyd resins seemed to equal the control formula in most respects. However, since the control formula failed the test in water resistance and, likewise, all the substituted primers failed this same test, it was decided that further work should be done on grinding to find the best method for both the control and the substituted resins. As soon as this work has been completed with the above resins, a sample of each primer will be sent to the Naval Air Experimental Station at Philadelphia for testing; and another report will be made describing the results.

50. In like manner, the dispersion resin DR7-0-2, made from Esskol oil, gave the best results when substituted for BK-3962 in the AN-TT-P-656A formulation.

51. From the above data the following conclusions have been drawn:

(a) It is possible to make a tung oil free alkyd resin from dehydrated castor fatty acids with pentaerythritol as the polyhydric alcohol which will exceed Rezyl 113 in fast drying and water resistance.

(b) A small amount of furylacrylic acid in the formulation increases hardness of the film and gives a faster dry.

(c) It is possible to introduce up to 13% linseed fatty acids along with the dehydrated castor fatty acids with pentaerythritol as the polyhydric alcohol without slowing up the drying time too much.

(d) Pentaerythritol alkyds are definitely better in water resistance and drying time than glycerol alkyds which have the same composition other than the polyhydric alcohol.

(e) Substitution of a treated linseed oil for tung oil in dispersion resins shows promise and more work should be done in this direction.

52. It is recommended that further development be done on alkyd resins using pentaerythritol as the polyhydric alcohol with dehydrated castor fatty acids until the best condition possible for producing them is found. It is necessary that this be done since this is a new type of alkyd resin which should be explored thoroughly before it is attempted to put it into production. It is also possible that they may be applicable to Navy specifications other than zinc chromate primer. Also, further work should be carried out on grinding the materials to determine the best method possible.

TABLE I

OILS USED IN PREPARATION OF ALKYD RESINS

NAME & MANUFACTURER	TYPE OF OIL	PHYSICAL AND CHEMICAL CONSTANTS							
		Visc.	Spec. Gravity	Refractive Index	Acid Value	Iodine Value	Saponification Value	Acetyl Value	Gelling Time in Minutes
Castung Special- Baker Castor Oil Company	Dehydrated Castor Oil	H-I	0.9339	1.4820	3.7	137.4	185	28.5	229
GF Oil - DuPont	Modified Linseed Oil	X-Y	1.0389	1.5901	12.4	187.1	255	39.6	17
RD-43-26 - Bakelite Corp.	Modified Linseed Oil	K	0.9586	1.4863	8.3	160.7	216.5	28.9	185
RD-43-27 - Bakelite Corp.	Modified Linseed Oil	T	0.9720	1.4896	2.67	152	228.2	36.6	107
Falkwood - Falk & Co.	Modified Linseed Oil	W	0.9705	1.4885	3.41	142.7	210	20.6	93
T-H Oil - Armour & Co.	Unknown	F	0.9335	1.4830	-	150.5	144.5	56.8	-
"K" Oil - Atlas Powder Co.	Modified Linseed Oil	I	0.9484	1.4839	17.87	-	-	-	-

TABLE II

FATTY ACIDS USED IN PREPARATION OF ALKYD RESINS

Name	Manufacturer	Type of Acid	Acid Value	Mol Weight	M. P.
Castung Special Fatty Acids	Prepared in NRL Lab. from Castung Special Oil of Baker Castor Oil Co.	Dehydrated Castor Fatty Acids	195	280	
Isoline Fatty Acids	Woburn Degreasing Co.	Dehydrated Castor Fatty Acids	199	280	
Linseed Fatty Acids	Woburn Degreasing Co.	Fatty acids from ordinary alkali refined linseed oil.	198	280	
Furyl Acrylic Acid	Prepared in NRL Lab.	A derivative of Furfuraldehyde	405	138	136°C-139°C

TABLE III

POLYHYDRIC ALCOHOLS USED IN PREPARATION OF ALKYD RESINS

Name	Manufacturer	Grade
Glycerine		C. P.
Pentaerythritol	Hercules Powder Co.	C. P.
Anhydroenoheptite	Prepared in NRL Laboratory	Purity not known
Phthalic Anhydride	American Cyanamid Co.	C. P.

TABLE IV

MATERIALS USED IN PREPARATION OF DISPERSION RESINS

NAME & MANUFACTURER	TYPE OF OIL	PHYSICAL AND CHEMICAL CONSTANTS							
		Visc.	Spec. Gravity	Refractive Index	Acid Value	Iodine Value	Saponification Value	Acetyl Value	Gelling Time in Minutes
Oils									
GF Oil - DuPont	Modified Linseed Oil	X-Y	1.0389	1.5901	12.4	187.1	255	39.6	17
ESSKOL Spencer Kellog	Maleic Treated Modified Linseed Oil	Z	0.9771	1.4881	21.8	134.3	219.4	65	52
Resin									
BR-254 - Bakelite	Paraphenyl Phenol								
Zn Oxide	AZO Type								

TABLE V

COMPOSITION AND PHYSICAL & CHEMICAL PROPERTIES OF PREPARED RESINS

Raw Material (%) Used In Preparation of Resins

Alkyd Number	Percent Phthalic Anhydride	% Dehydrated Castor Fatty Acid	% Linseed Fatty Acids	% Furyl Acrylic Acid	% Penta-erythritol	Percent Glycerin	Percent Oil	Degrees C. Temperature Range of Cooking	Reaction Time from Start of Reaction (Min.)	Acid Value Thinned	% Solids Thinned	Viscosity Gardner-Holt	Remarks
AR6-0-1	19.2	48.3		11.9	20.7*			150 -208	193 Min.				
AR-7-0-1	36.9	30.7		7.6	24.8			170°-195°	250 "				No good. Jelled before reaction comrlete.
AR,8-0-1	35.8	26.4	3	7.4	27.4			160°-205°	135 "	35	50	Z6	Very heavy. Hard to handle.
AR-9-0-1	35.6	23.6	5.9	7.4	27.5			175°-220°	90 "	22.6	50	Z6	Very heavy. Hard to handle.
AR-10-0-1	35.6	23.6	5.9	7.4	27.5			190°-205°	270 "	49	50		Acid value too high. Eliminated.
AR-11-0-1	33.8	42.3			23.9								Jelled before reaction complete.
AR-12-0-1	17.2	32.3	32.3		18.2			170°-220°	330 "	13	60	C	
AR-13-0-1	24.4	36.8	18.3		20.5			175°-220°	240 "	17	60	U-V	
AR-14-0-1	28.7	39	9.9		22.4			175°-220°	210 "	14	60	Z6	
AR-15-0-1	26.7	37.6	12.6		23.1			175°-210°	390 "	13.8	50	U-V	
AR-16-0-1	29	27.5	13.7	6.8	23			165°-220°	180 "	21.5	60	U-V	
AR-17-0-1	29.8	28.2	14.2	7		20.8		177°-210°	120 "	16.5	60	H	
AR-2-0-2	19.3	48.4		12	20.3			160°-204°	390 "	15.9	60	R	
AR-2-0-3	19.3	48.4		12	20.3			168°-222°	250 "	15.9	60	O	
AR-3-0-2	33.4	42.6			24			150°-220°	220 "	20.8	60	Z6	Very heavy. Hard to handle.
AR-3-1-1	27	51			22			200°-230°	150 "				Jelled before reaction complete. Eliminated.
AR-3-2-1	27	51			22			160°-215°	200 "	12.6	60	Z3	
AR-2-0-4	19.3	48.4		12	20.3			180°-215°	300 "	12.2	60	O	
AR18-0-1	38.1					16.6	Dryinol 45.3	180°-218°	60 "				Jelled before reaction complete. Eliminated.
AR19-0-1	38.2					16.6	Rd-43-26 45.2	180°-225°	75 "	23.8	60	Z6	Very heavy. Almost jelled. Eliminated.
AR19-0-2	38.2					16.6	Rd-43-26 45.2	180°-190°	90 "	10.8	60	L	
AR20-0-1	38.2					16.6	Rd-43-27 45.2	160°-190°	165 "	34.5	60	Z6	Acid value too high. Eliminated.
AR21-0-1	38.2					16.6	Falkwood 45.2	180°-200°	115 "				Jelled before reaction complete. Eliminated.
AR21-0-2	38.2					16.6	Falkwood 45.2	180°-210°	105 "	40.5	60	Z6	Acid value too high. Eliminated.
AR22-0-1	38.2					16.6	G.F. oil 45.2	195°-198°	125 "	15	60	Z	
AR23-0-1	38.2					16.6	T-H oil 45	170°-210°	150 "	9.2	60	T	
AR20-0-2	38.2					16.6	RD-43-27 45.2	175°-180°	115 "	28.5	60	V	
AR28-0-1	39.6				31.2		Castung Special 39.2	182°-240°	240 "				Reaction not complete. Kicked out of solution.
AR29-0-1	33.8					15.5	"K"oil Atlas 50.5	180°-218°	95 "	15.8	60	U-V	
AR3-0-3	33.5	42.5			24			170°-205°	255 "	27	60	Z6	
AR-12-1-1	17.2	32.3	32.3		18.2			180°-250°	305 "	5	60	S	
AR-4-0-2	25.2	34.1	13.7		27			175°-238°	335 "	3.2	60	Z3	
AR-8-0-2	35.8	26.4	3	7.4	27.4			158°-195°	175 "	31	60	Z6	
AR-9-0-2	35.6	23.6	5.9	7.4	27.5			162°-210°	245 "	26.5	60	Z6	Very heavy. Difficult to handle.
AR-14-1-1	28.8	39.3	9.8		22.1			166°-204°	290 "	16.3	60	Z3	
AR-17-0-2	29.8	28.2	14.2	7		20.8		168°-210°	290 "	13.8	60	F-G	
AR-19-0-3	38.2					16.5	RD-43-26 oil 45.3	178°-210°	125 "	24	60	R	
AR-22-0-2	38.2					16.6	GF-oil 45.2	168°-220°	140 "	23	60	T-U	
AR-23-1-1	37.8					17.2	T-H oil 45	162°-210°	200 "	13	60	U-V	
AR-31-0-1	33.6					16.8	Castung Special 49.8	178°-187°	170 "	14.2	60	G-H	
AR-15-1-1	26.7	37.6	12.6		23.1			170°-215°	315 "	14.5	60	W	
AR-16-0-2	29	27.5	13.7	6.8	23			170°-205°	230 "	24	60	X-Y	
AR4-5-1	26.2	35.2	14.2		24.4			160°-210°	340 "	7.9	60	Z3	
AR2-1-1	19.2	48.5		12	20.3			166°-214°	515 "	15.6	60	G-H	
AR-3-3-1	31.6	45.3			23.1			167°-203°	345 "	17.9	60	Z1	
AR-2-0-5	19.3	48.4		12	20.3			190°-223°	287 "	12.5	60	Y	
AR-8-1-1	33.5	30.2	3.4	8.5	24.4			165°-210°	300 "	29.9	60	Z6	

* Anhydroenoheptie used as polyhydric alcohol.
Decomposition took place. Reaction incomplete. Discarded.

TABLE VI

WATER RESISTANCE OF EXPERIMENTAL ALKYD RESINS AFTER 46 HOURS AIR DRY

Alkyd	Elapsed Time Before Observation					Remarks
	3 hrs.	24 hrs.	48 hrs.	72 hrs.	10 days	
AR2-0-5	4	5	4	3	2	Soft at 3 hrs. but film still fairly good after 10 days.
AR3-0-3	1	2	2	3	7	Good at 72 hrs. but blistered and soft after 10 days.
AR4-5-1	10	10	10	10	10	Worst of all from beginning.
AR8-1-1	2	2	1	1	1	Best of all after 10 days film still good.
AR9-1-1	3	7	5	6	10	Complete failure after 10 days.
AR15-1-2	4	6	8	5	9	Soft and cloudy at 24 hrs., blistered at 48 hrs., complete failure at 10 days.
AR16-0-2	2	3	7	7	9	Blistered at 24 hrs., soft at 48 hrs., complete failure at 10 days.
AR17-0-2	4	4	7	6	8	Soft at 3 hrs., cloudy and blistered at 24 hrs., complete failure at 10 days.
AR22-C-2	4	1	5	5	8	Good at 24 hrs., blistered and soft at 48 hrs., complete failure at 10 days.
AR23-0-2	3	4	5	4	7	Softer at 24 hrs., blistered at 48 hrs., complete fail-ure at 10 days.
AR31-0-2	4	7	9	9	10	Cloudy and softer at 3 hrs., blistered at 48 hrs., complete failure at 10 days.
AR19-0-2	3	4	4	4	3	Softer at 3 hrs., slight cloudy but fairly good at 10 days.

TABLE VI (Continued)

WATER RESISTANCE OF EXPERIMENTAL ALKYD RESINS AFTER 46 HOURS AIR DRY

Alkyd	Elapsed Time Before Observation					Remarks
	3 hrs.	24 hrs.	48 hrs.	72 hrs.	10 days	
CONTROL REZYL-113	8	8	9	9	10	Soft at 3 hrs., cloudy & blistered at 24 hrs., complete failure at 72 hrs.
AR12-1-1	2	7	10	10	10	Soft and blistered at 24 hrs., complete failure at 72 hrs.
AR14-1-1	1	5	10	10	10	Soft and blistered at 24 hrs., complete failure at 72 hrs.

NOTE: The numbers in the above represent a scale of comparison with 1 as the best and 10 as the poorest.

TABLE VII

SPECIFICATION TEST ON FINAL PRIMERS CONTAINING EXPERIMENTAL RESINS GROUND ON PEBBLE MILL

Resin Used in Primer	Appearance	Wt. per Gallon	Viscosity (Stormer)	Skinning	Stability	Wk'g. Prop.	Drying Time	Surface Appearance	Color	Flexibility	Metal Anchorage	Coating Anchorage	Lacquer Resistance	Water Resistance	Gasoline Resistance	Settling	Non-Volatile
Control	Good but Skinned	10.63 lb.	10.6 sec.	Skinned	Good	OK	Air dry to handle in 6 hrs. To touch in 4 min.	Good	OK	OK	Good	OK	Good at 10 min.	Complete Failure	Very good	Did not disperse on shaking (8 cc)	64.5%
AR-3-0-3	Very good	9.56 lb.	7.2 sec.	No skin after 48 hrs.	Good	OK	"	Slight silking	OK	OK	Good	OK	"	"	" "	Best	50.0%
AR-12-1-1	Good	10.42 lb.	7.5 sec.	"	Poor	OK	"	Much silking	OK	Complete Failure	Poor	OK	No good after 4 hours	"	" "	As good as Control (9 cc)	61.0%
DR-10-0-3	Very good	10.67 lb.	7.8 sec.	"	Poor	OK	"	Slight silking	OK	OK	Fair	OK	Good at 4 hrs.	"	" "	As good as Control (7 cc)	61.0%
DR-7-0-2	Very good	10.69 lb.	11.0 sec.	"	Fair	OK	"	Much silking	OK	OK	Fair	OK	Good at 10 min.	"	" "	"	64.0%
DR-11-0-4	Very good	10.55 lb.	9.0 sec.	"	Poor	OK	"	Slight silking	OK	OK	Good	OK	Good at 4 hrs.	"	" "	"	60.7%
AR-4-0-2	Good needed straining	10.52 lb.	9.6 sec.	No skin	Fair	OK	"	Silking	OK	OK	Poor	OK	Good at 10 min.	"	" "	As good as Control	63.4%
AR-8-0-2	Good needed straining	10.13 lb.	8.4 sec.	Slight skin	Fair	OK	"	Silking	OK	OK	Good	OK	"	"	" "	Disperses after 2 min. shaking	57.0%
AR-2-1-1	Good some settlement	10.83 lb.	8.2 sec.	No skin	Good	OK	"	Some silking	OK	OK	Fair	OK	"	"	" "	As good as Control (7 cc)	62.3%
AR-9-0-2	Very good	9.84 lb.	7.9 sec.	No skin	Good	OK	"	"	OK	OK	Fair	OK	"	"	" "	As good as Control (9 cc)	62.2%
AR-23-1-1	Very good	10.68 lb.	8.3 sec.	No skin	Fair	OK	"	Silking	OK	OK	Poor	OK	"	"	" "	" (8 cc)	62.2%
AR-15-1-1	Very good	11.2 lb.	8.9 sec.	No skin	Good	OK	"	Silking	OK	OK	Good	OK	"	"	" "	Disperses after 2 min.	67.7%
AR-31-0-1	Very good	11.44 lb.	7.7 sec.	No skin	Fair	OK	"	Much silking	OK	OK	Good	OK	"	"	" "	As good as Control (8 cc)	67.8%
AR-16-0-2	Very good	11.20 lb.	8.5 sec.	No skin	Good	OK	"	Slight silking	OK	OK	Good	OK	Good at 10 min.	Complete Failure	Very good	Disperses after 2 min.	67.9%

SPECIFICATION TEST ON FINAL PRIMERS CONTAINING EXPERIMENTAL RESINS GROUND ON ROLLER MILL

Resin Used in Primer	Appearance	Wt. per Gallon	Viscosity (Stormer)	Skinning	Stability	Wk'g. Prop.	Drying Time	Surface Appearance	Color	Flexibility	Metal Anchorage	Coating Anchorage	Lacquer Resistance	Water Resistance	Gasoline Resistance	Settling	Non-Volatile
AR-3-3-1	Good	10.66 lb.	7.3 sec.	No skin	OK Vehicle separated	OK	OK	Slight silking	OK	OK	Good	Good	Good at 1 hour	"	" "	Very good	60.5%
AR-19-0-3	Good	10.56 lb.	7.35 sec.	No skin	OK	OK	OK	Some silking	OK	OK	Good	Good	Not good at 1 hr.	"	" "	" "	61.8%
AR-2-0-5	Very viscous. Good, some skin.	10.64 lb.	8.40 sec.	Skinning	OK Skinning	OK Skinning	OK	Very good	OK	OK	Good	Good	Good at 10 min.	"	" "	" "	62.7%
AR-4-5-1	Good	10.71 lb.	8.65 sec.	No skin	OK	OK	OK	Slight silking	OK	OK	Good	Good	Good at 1 hour	"	" "	" "	61.7%
AR-8-1-1	Good	10.26 lb.	7.50 sec.	No skin	OK	OK	OK	Very good	OK	OK	Good	Good	Good at 10 min.	"	" "	" "	61.8%

APPENDIX A

AR6-0-1

Raw Materials:

48.0g	Anhydroenoheptite	.2 mols + 10% excess
44.4g	Phthalic anhydride	.3 "
112.0g	Dehydrated castor fatty acids	.4 "
27.6g	Furyl acrylic acid	.2
232.0	Total	

The material was placed in a reacting setup as described in paragraph 23, cooked by solvent method.

Cooking Log:

Time	Temp.	H_2O
10:40	Start	-
10:44	125° C	1st drop
10:47	150° C	3.0 cc
11:00	180° C	13.5 "
11:15	190° C	14.0 "
11:45	189° C	16.0 "
12:15	200° C	17.1 "
12:45	200° C	17.5 "
2:00	208° C	20.0 "

The color was becoming dark and it looked as if there was some sort of decomposition taking place within the reaction.

Acid Value:

56.81
56.52
.29 6.8 cc = A.V. - 129.0

This acid value showed that the esterification was not taking place properly.

Added 8 g more anhydroenoheptite

Time	Temp.	H_2O
3:00	208° C	22 cc
4:00	208° C	23 "

The color was becoming darker and the reaction did not seem to be going right. It was concluded that the anhydroenoheptite was decomposing within itself.

Product Discarded

AR7-0-1

Raw Materials:

74.8 g	Pentaerythritol	.5	mol + 8% excess
111.0 g	Phthalic anhydride	.75	"
92.4 g	Dehydrated castor fatty acids (Castung Special)	.35	"
23.0 g	Furyl acrylic acid	.16	"
301.2 g	Total		

The material placed in a reacting setup as described in paragraphs 22 and 23. Cooked by solvent method.

Cooking Log:	Time	Temp	H_2O
	10:00	Start	-
	10:20	170° C	1st drop
	10:30	170° C	4.0 cc
	10:45	170° C	6.5 "
	11:00	170° C	8.0 "
	11:15	170° C	9.0 "
	11:45	170° C	11.0 "
	12:15	172° C	12.0 "
	12:45	172° C	13.0 "
	1:15	190° C	14.0 "
	1:45	195° C	16.0 "
	2:15	195° C	17.0 "
A.V. - 58.6 sample	2:30		

Product jelled was discarded

AR8-0-1

Raw Materials:

74.8 g	Pentaerythritol	.5 mols + 8% excess
111.6 g	Phthalic anhydride	.75 "
83.2 g	Dehydrated castor fatty acids (Castung Special)	.30 "
9.2 g	Linseed fatty acids	.03 "
23.0 g	Furyl acrylic acid	.16 "
301.8 g	Total	

The material was placed in a reacting setup as described in paragraphs 22 and 23. Cooked by solvent method.

Cooking Log:	Time	Temp.	H_2O
	9:35	Start heating	-
	9:42	140° C	1st drop
	10:00	160° C	3.0 cc
	10:15	160° C	6.5 "
	10:30	160° C	8.0 "
	10:45	160° C	9.2 "
	11:00	160° C	11.0 "
	11:15	200° C	13.0 "
	11:30	203° C	15.2 "
	11:45	205° C	17.2 "
	12:00	205° C	18.2 "
Very heavy at this time	12:15	205° C	19.2 "

Thinned with 250 g xylene - solids 53%
Final acid value (thinned) - 35
Driers added: - 0.3% Pb
.03% Co.

Viscosity - Very heavy Z_6+

AR9-0-1

Raw Materials:

74.8 g	Pentaerythritol	.5 mol + 8% excess
111.6 g	Phthalic anhydride	.75 "
73.9 g	Dehydrated castor fatty acids (Castung Special)	.26 "
18.5 g	Linseed fatty acids	.07 "
23.0 g	Furyl acrylic acids	.16 "
301.8 g	Total	

The material placed in a reacting setup as described in paragraphs 22 and 23. Cooked by solvent process.

Cooking Log:

	Time	Temp.	H_2O
	2:30	Start	-
	2:40	175° C	1st drop
	2:45	212° C	4.0 cc
	3:00	205° C	9.0 "
	3:15	220° C	14.5 "
	3:30	215° C	18.5 "
	3:45	215° C	19.7 "
Becoming very heavy	4:00	220° C	21.0 "

Thinned with 279.8 g xylene to give 50% solids.

Final acid value (thinned) - 22.6

Driers added; .3% Pb
.03% Co

Viscosity - very heavy Z_6+

AR10-0-1

Raw Materials:

74.8 g	Pentaerythritol	.5 mol + 8% excess
111.6 g	Phthalic anhydride	.75 "
73.9 g	Dehydrated castor fatty acids (Castung Special)	.26 "
18.5 g	Linseed fatty acids	.07 "
23.0 g	Furyl acrylic acid	.16 "
301.8 g	Total	

All ingredients except the phthalic anhydride were placed in the reacting setup as described in paragraphs 22 and 23. Cooked by the solvent process.

Cooking Log of initial esterification:

	Time	Temp.	H_2O
	10:30	Start	-
	10:45	200° C	3.0 cc
	11:15	200° C	6.0 "
	12:35	190° C	7.9 "
Acid Val. 5-8	1:00	210° C	9.0 "
Sample	2:15 added the phthalic anhydride		
	2:30	170° C	10.0 "
	2:45	190° C	11.5 "
	3:15	195° C	13.7 "
	3:30	195° C	15.0 "
Acid Val. 49.	4:15	205° C	18.5 "
Sample			

Acid value was too high. It was concluded that this method was unsatisfactory.

Product Discarded

AR11-0-1

Raw Materials

100.0 g	Dehydrated Castor acids (Castung Special)	.36 mols.
79.9 g	Phthalic anhydride	.54 "
56.3 g	Pentaerythritol	.36 "
236.2 g	Total	

These materials were placed in a reaction setup as described in paragraphs 22 and 23. The inert gas method was employed using CO_2 as gas.

Cooking Log:

	Time	Temp.
	10:00	Start
	10:30	160° C
	11:00	190° C
	11:30	190° C
	12:00	200° C
	12:30	205° C
	1:00	205° C
Acid value 74 sample	1:30	205° C
	2:30	205° C

JELLED

Product Discarded

AR12-0-1

Raw Materials:

100.0 g	Dehydrated castor acids (Castung Special)	.36 mols	
100.0 g	Linseed fatty acids	.36 "	
53.3 g	Phthalic anhydride	.36 "	
56.3 g	Pentaerythritol	.36 "	+ 15% excess
309.6 g	Total		

The material was placed in a reacting setup as described in paragraphs 22 and 23. The inert gas method was employed using CO_2 as gas.

Cooking Log:

	Time	Temp.
	8:20	Start
	8:30	170° C
	8:45	180° C
Acid value 72.2 sample	9:30	180° C
	9:45	185° C
Acid value 52.2 sample	10:30	200° C
Acid value 31 sample	11:30	220° C
Acid value 13 sample	2:00	220° C

Thinned with 200 g xylene
60% solids

Final acid value (thinned) 6.8

Drier added: 0.3% Pb
.03% Co

Viscosity - C

AR13-0-1

Raw Materials:

100.0 g	Dehydrated Castor fatty acids (Castung Special)	.36 mols	
50.0 g	Linseed fatty acids	.18 "	
60.6 g	Phthalic anhydride	.45 "	
56.3 g	Pentaerythritol	.36 "	+ 15% excess
266.9 g	Total		

These materials were placed in a reacting setup as described in paragraphs 22 and 23. The inert gas method was employed with CO_2 as the gas.

Cooking Log:

	Time	Temp.
	9:20	Start
	9:28	175° C
	10:00	190° C
Acid value 65 sample	10:30	195° C
	11:00	210° C
Acid value 23 sample	1:15	220° C
Acid value 7 sample	2:30	220° C

Thinned to 60% solids with xylene

Final acid value (thinned) - 4.2

Viscosity - U-V

Drier added - .3% Pb
.03% Co.

AR14-0-1

Raw Materials:

100.0 g	Dehydrated Castor fatty acids (Bakers 403)	.36 mols.	
25.0 g	Linseed fatty acids	.09 "	
73.3 g	Phthalic anhydride	.49 "	
56.3 g	Pentaerythritol	.36 "	+ 15% excess
254.6 g	Total		

These materials were placed in a reacting setup as described in paragraphs 22 and 23. The inert gas method was employed with CO_2 as the gas.

Cooking Log:

	Time	Temp.
	9:05	Start
	9:30	150° C
	10:00	175° C
	10:30	180° C
	11:00	180° C
	11:30	200° C
	12:00	200° C
A.V. 58.4 sample	12:30	200° C
	1:00	220° C
Began to jell	1:30	220° C

Thinned to 50% solids with xylene

Final acid value (thinned) 14

Viscosity - very heavy Z_6+

Drier added - .3% Pb
.03% Co.

AR15-0-1

Raw Materials:

100.0 g	Dehydrated Castor Fatty acids (Bakers 403)	.36 mols
33.6 g	Linseed fatty acids	.12 "
71.0 g	Phthalic anhydride	.48 "
56.3 g	Pentaerythritol	.36 " + 15% excess
260.9 g	Total	

These materials were placed in a reacting setup as described in paragraphs 22 and 23. The inert gas method was employed with CO_2 as the gas.

Cooking Log:

	Time	Temp.
	9:00	Start
	9:30	155° C
	10:00	175° C
	10:30	175° C
	11:00	175° C
	11:30	175° C
	12:00	175° C
	12:30	180° C
	1:30	190° C
	2:30	205° C
	3:00	205° C
Acid value 22.9 sample	3:30	210° C

Thinned to 50% solid with xylene

Final acid value (thinned) 11.5

Viscosity - U-V

Drier added - .3% Pb
.03% Co.

AR16-0-1

Raw Materials:

67.2 g	Dehydrated Castor acids (Castung Special)	.24 mols.	
16.5 g	Furyl acrylic acid	.12 "	
33.6 g	Linseed fatty acids	.12 "	
71.0 g	Phthalic anhydride	.48 "	
56.4 g	Pentaerythritol	.36 "	+ 15% excess
244.7 g	Total		

The materials were placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O over
10:10	Start	-
10:30	143° C	1st drop
10:45	165° C	5.4 cc
11:00	184° C	8.0 "
11:15	185° C	12.0 "
11:30	190° C	14.0 "
11:45	196° C	16.0 "
12:00	205° C	17.0 "
12:15	200° C	17.5 "
12:30	206° C	18.2 "
12:45	206° C	19.2 "
1:00	212° C	19.2 "
1:30	220° C	20.0 "

Thinned to 60% solid with xylene

Final acid (thinned) - A.V. 21.5

Viscosity - U-V

Dried added: - .3% Pb
.03% Co.

AR17-0-1

Raw Materials:

67.2 g	Dehydrated castor fatty acids (Castung Special)	.24 mols	
16.5 g	Furyl acrylic acid	.12 "	
33.6 g	Linseed fatty acids	.12 "	
71.0 g	Phthalic anhydride	.48 "	
48.6 g	Glycerin	.48 "	+ 10% excess
236.9 g	Total		

The materials were placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O
9:35	Start	-
9:45	140° C	-
10:00	177° C	4.4 cc
10:15	186° C	10.0 "
10:30	194° C	14.0 "
10:45	195° C	16.5 "
11:00	197° C	17.5 "
11:15	201° C	18.7 "
11:30	204° C	19.4 "
12:00	201° C	20.0 "
12:30	210° C	21.0 "

Thinned to 60% solids

Final acid value (thinned) - 16.5

Viscosity - H

Drier added: - .3% Pb
.03% Co.

AR2-0-2

Raw Materials:

234.6 g	Pentaerythritol	1.5 mols + 15% excess
222.0 g	Phthalic anhydride	1.5 "
138.0 g	Furyl acrylic acid	1.0 "
560.0 g	Dehydrated castor fatty acids	2.0 "
1154.6 g	Total	

The materials were placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O over
8.40	Start	-
9.05	160° C	1st drop
9:15	162° C	7.0cc
9:30	166° C	16.0"
9:45	174° C	30.0"
10:00	180° C	37.0"
10:30	178° C	43.0"
11:00	185° C	50.0"
11:30	180° C	51.0"
12:00	184° C	55.0"
12:30	185° C	59.0"
1:00	195° C	61.0"
1:30	195° C	63.0"
2:00	200° C	64.5"
2:30	202° C	66.0"
3:00	204° C	68.6"
3:30	204° C	69.0"

Thinned to 60% solids with 726 g xylene

Final acid value (thinned) - 15.9

Viscosity - 0

Driers added; - .3% Pb
.03% Co.

AR2-0-3

Raw Materials:

234.6 g	Pentaerythritol	1.5 mols + 15% excess
222.0 g	Phthalic Anhydride	1.5 "
138.0 g	Furyl Acrylic acid	1.0 "
560.0 g	Dehydrated castor fatty acid (Castung Special)	2.0 "
1154.6 g	Total	

The material was placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O over
9:20	Start	--
9:40	168° C	1st drop
10:00	168° C	16.0 cc
10:15	168° C	26.0 "
10:30	170° C	32.8 "
10:45	176° C	38.5 "
11:00	180° C	43.0 "
11:15	188° C	47.5 "
11:45	190° C	52.0 "
12:15	198° C	58.0 "
12:45	212° C	63.5 "
1:15	216° C	67.0 "
1:45	222° C	69.0 "
1:55	222° C	71.0 "

Heat off.

Thinned to 60% solids with 726 g xylene

Final acid value (thinned) - 15.9

Viscosity - 0

Drier added: - .3% Pb
.03% Co.

AR3-0-2

Raw Materials:

158.7 g	Pentaerythritol	1 mol + 15% excess
222.0 g	Phthalic anhydride	1.5 "
280.0 g	Dehydrated castor fatty acids (Castung Special)	1.0 "
660.7 g	Total	

This material was placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O over
8:25	Start	-
8:50	190° C	1st drop
9:25	225° C	27 cc
9:55	220° C	35 "
10:00	Foamed badly shut down and ran acid value. Acid value 40. Introduced enough solvent to reduce to following temperaturs.	
10:40	150° C	35.2 cc
11:30	150° C	36.0 "
12:30	150° C	36.2 "

Heat off.

Thinned to 60% solids with xylene

Final acid value (thinned) - 20.8

Viscosity (very heavy) - Z6+

Drier added: - .3% Pb
.03% Co.

AR3-1-1

Raw Materials:

140.0 g	Dehydrated castor fatty acids (Castung Special)	.5 mol
74.0 g	Phthalic Anhydride	.5 "
60.9 g	Pentaerythritol	.37 " + 15% excess
274.9 g	Total	

This material was placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

	Time	Temp.	H_2O over
	10:00	Start	-
	10:20	200° C	4 cc
	10:40	230° C	14 "
	11:15	220° C	16 "
A.V. 46 sample	12:30		

Jelled

AR3-2-1

Raw Materials:

140.0 g	Dehydrated castor fatty acids	.5 mol
74.0 g	Phthalic anhydride	.5 "
60.9 g	Pentaerythritol	.37 " + 15% excess
274.9 g	Total	

This material was placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

	Time	Temp.	H_2O over
	1:10	Start	-
	1:25	190° C	1st drop
	1:35	210° C	12.0cc
	2:10	210° C	14.0 "
	3:00	215° C	16.5 "
Acid value 25.3 sample	3:15	215° C	16.7 "
	3:30	160° C	17.0 "
	3:45	160° C	17.0 "

Thinned to 60% solids with xylene

Viscosity - Z_3

Final acid value (thinned) - 12.6

Drier added: - .3% Pb
.03% Co.

AR2-0-4

Raw Materials:

117.0 g	Pentaerythritol	.75 mols + 15% excess
111.0 g	Phthalic anhydride	.75 "
69.0 g	Furyl acrylic acid	.5 "
280.0 g	Dehydrated castor fatty acid (Castung Special)	1.0 "
577.0 g	Total	

This material was placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

	Time	Temp.	H_2O
	10:20	Start	-
	11:00	180° C	1st drop
	11:30	195° C	19.0cc
	11:45	195° C	24.0 "
	12:00	195° C	26.5 "
	12:15	195° C	27.5 "
	12:30	195° C	28.5 "
	12:45	195° C	29.25"
	1:00	195° C	30.00"
	1:15	195° C	31.00"
	1:30	195° C	31.50"
	1:45	195° C	32.00"
	2:00	195° C	32.5 "
	2:15	195° C	32.75"
	2:30	195° C	33.25"
	2:45	195° C	33.75"
	3:00	195° C	34.00"
	3:15	195° C	34.5 "
	3:30	195° C	35.0 "
Acid value 24 sample	3:45	215° C	35.5 "
	4:00	220° C	36.0 "

Heat off

Thinned to 60% solids with xylene

Final acid value (thinned) - 12.2

Viscosity - 0

Driers added: - .3% Pb
.03% Co.

AR18-0-1

Raw Materials:

222.0 g Phthalic anhydride	1.5 mols	
96.6 g Glycerine	1.0 "	+ 5% excess
264.0 g Dryinol oil	.34 "	
0.5 g Litharge		
583.1 g Total		

of these materials,

264.0 g Dryinol oil
61.6 g Glycerine
0.5 g Litharge

were placed in a reacting setup as described in paragraphs 22 and 23 and alcoholized to the monoglyceride as described in paragraph 25.

Cooking Log: (Preparation of monoglyceride)

Time	Temp.
10:55	Start
11:20	220° C
12:20	220° C - Sample slightly cloudy with 2:1 MeOH
1:20	220° C - Sample clear with 2:1 MeOH

To the monoglyceride was added:

35 g Glycerine
222 g Phthalic anhydride

The cooking was then continued by solvent method as described in paragraphs 22 and 23.

Cooking Log:	Time	Temp.	H_2O
	1:15	160° C	-
	1:30	188° C	7 cc
	2:00	200° C	12 "
A.V. 74 Sample	2:07	218° C	16 "
	2:13	Jelled	

Discarded

AR19-0-1

Raw Materials:

222.0 g	Phthalic anhydride	1.5 mols
96.6 g	Glycerine	1.0 " + 5% excess
264.0 g	Bakelite RD-43-26 oil	.34 "
0.5 g	Litharge	
583.1 g	Total	

Of these materials,

264.0 g Bakelite RD-43-26 oil
61.6 g Glycerine
0.5 g Litharge

were placed in a reacting setup as described in paragraphs 22 and 23 and alcoholized to the monoglyceride as described in paragraph 25.

Cooking Log of Alcoholysis:

Time	Temp.	H_2O over
10:25	Start	-
10:45	220° C	
12:45	210° C -	Sample clear in 2:1 MeOH

To the monoglyceride was added:

222.0 g Phthalic anhydride
35.0 g Glycerine

The cooking was then continued by the solvent method as described in paragraphs 22 and 23.

	Time	Temp.	H_2O
	12:55	120° C	-
	1:05	180° C	1 cc
	1:30	218° C	14 "
	1:45	220° C	20 "
	2:00	225° C	22 "
Very heavy	2:10	225° C	23 "

Thinned to 60% solids with xylene

Final acid value (thinned) - 28.8

Viscosity - Z_6+

AR19-0-2

Raw Materials:

222.0 g	Phthalic anhydride	1.5 mols
96.6 g	Glycerine	1.5 " + 5% excess
264.0 g	Bakelite RD-43-26 oil	.34 "
0.5 g	Litharge	
583.1 g	Total	

Of these materials,

264.0 g Bakelite RD-43-26 oil
61.6 g Glycerine
0.5 g Litharge

were placed in a reacting setup as described in paragraph 22 and alcoholized to the monoglyceride as described in paragraph 25.

Cooking Log of Alcoholysis:

Time	Temp.
8:10	Start
8:45	200° C
10:00	200° C - Sample clear in 2:1 MeOH

To the monoglyceride was added:

222.0 g Phthalic anhydride
35.0 g Glyceride

The cooking was then continued by the solvent method as described in paragraphs 22 and 23.

Cooking Log:

Time	Temp.	H_2O
10:25	140° C	-
10:45	180° C	3 cc
11:00	190° C	9 "
11:15	190° C	15 "
11:30	190° C	17 "
11:45	190° C	19.5"
12:00	190° C	21 "
12:15	190° C	22 "
1:00	160° C	23 "

Heat off

Thinned to 60% solids with xylene		
Final acid value (thinned)	-	10.8
Viscosity	-	L
Drier added:	-	.3% Pb
		.03% Co.

AR20-0-1

Raw Materials:

222.0 g	Phthalic anhydride	1.5 mols
96.6 g	Glycerine	1.0 " + 15% excess
264.0 g	Bakelite RD-43-27 oil	
0.5 g	Litharge	
583.1 g	Total	

Of these materials,

264.0 g Bakelite RD-43-27 oil
61.6 g Glycerine

were placed in a reacting setup as described in paragraph 22 and alcoholized to the monoglyceride as described in paragraph 25.

Cooking Log of Alcoholysis:

Time	Temp.
9:20	Start
9:40	200° C
11:00	210° C - Sample not clear in 2:1 MeOH
11:30	210° C - Sample clear in 2:1 MeOH

To the monoglyceride was added:

222. g Phthalic anhydride
35. g Glycerine

The cooking was then continued by the solvent method as described in paragraphs 22 and 23.

Cooking Log:

Time	Temp.	H_2O
12:50	Start	-
1:10	180° C	2.5 cc
1:30	190° C	10.0 "
1:45	182° C	14.0 "
2:00	184° C	16.0 "
2:15	184° C	18.0 "
2:30	184° C	19.0 "
2:45	170° C	19.5 "
3:00	170° C	20.0 "
3:15	160° C	20.5 "
3:30	160° C	20.8 "
3:45	160° C	21.0 "
3:55	Heat off	

Thinned to 60% solids with xylene
Final acid value (thinned) - 34.5
Viscosity very heavy - Z_6+

Discarded because of high acid value

AR20-0-2

Raw Materials:

222.0 g	Phthalic anhydride	1.5 mols
96.6 g	Glycerine	1.0 " + 15% excess
246.0 g	Bakelite RD-43-27 oil	.34 "
0.5 g	Litharge	
565.1 g	Total	

Of these materials,

264.0 g Bakelite RD-43-27 oil
61.6 g Glycerine
0.5 g Litharge

were placed in a reacting setup as described in paragraph 22 and alcoholized to the monoglyceride as described in paragraph 25.

Cooking Log of alcoholysis:

Time	Temp.
8:05	Start
8:25	210° C
9:00	220° C
9:35	220° C - Sample clear with 2:1 MeOH

To the monoglyceride was added:

222.0 g Phthalic anhydride
35.0 g Glycerol

The cooking was then continued by the solvent method as described in paragraphs 22 and 23.

Cooking Log:

	Time	Temp.	H_2O
	9:55	-	-
	10:20	180° C	7.5 cc
	10:35	180° C	11.5 "
	10:50	180° C	14.0 "
	11:05	180° C	17.0 "
	11:20	180° C	18.0 "
	11:45	180° C	19.0 "
	12:00	180° C	20.0 "
Began to foam badly	12:15	180° C	21.0 "

Heat off

Thinned to 60% solids with xylene
Final acid value (thinned) - 28.5
Viscosity - L

Discarded because of high acid value

AR21-0-1

Materials:

222.0 g	Phthalic anhydride	1.5 mols
96.6 g	Glycerine	1.0 " + 5% excess
264.0 g	Falkwood oil	
0.5 g	Litharge	
583.1 g	Total	

Of these materials,

264.0 g Falkwood Oil
61.6 g Glycerine
0.5 g Litharge

were placed in a reacting setup as described in paragraph 22 and alcoholized to the monoglyceride as described in paragraph 25.

Cooking Log of alcoholysis:

Time	Temp.
9:05	240° C
10:05	220° C
10:45	220° C - Sample clear in 2:1 MeOH

To the monoglyceride was added:

222 g Phthalic anhydride
35 g Glycerine

The cooking was then continued by the solvent method as described in paragraphs 22 and 23.

Cooking Log:

Time	Temp.	H_2O
11:05	Start	-
11:20	180° C	35.0 cc
11:35	180° C	7.5 "
11:50	180° C	14.0 "
12:05	180° C	16.0 "
12:15	190° C	19.0 "
12:30	200° C	21.0 "
1:00	Jelled	

Discarded

AR21-0-2

Raw Materials:

222.0 g	Phthalic anhydride	1.5 mols
96.6 g	Glycerine	1.0 " + 5% excess
264.0 g	Falkwood oil	
0.5 g	Litharge	
583.1 g	Total	

Of these materials,

264.0 g Falkwood oil
61.6 g Glycerine
0.5 g Litharge

were placed in a reacting setup as described in paragraph 22 and alcoholized to the monoglyceride as described in paragraph 25.

Cooking Log of alcoholysis:

Time	Temp.
9:30	-
9:50	222° C
10:30	210° C
11:10	210° C - Sample clear in 2:1 MeOH

To the monoglyceride was added:

222.0 g Phthalic anhydride
35.0 g Glycerine

The cooking was then continued by the solvent method as described in paragraphs 22 and 23.

Cooking Log:

	Time	Temp.	H_2O over
	12:45	200° C	3 cc
	1:00	180° C	8 "
	1:15	180° C	12 "
	1:30	180° C	14 "
	1:45	180° C	16 "
	2:00	180° C	17 "
Acid value 67 sample	2:15	210° C	19 "
Very heavy, much foaming	2:30	180° C	21 "

Heat off

Thinned to 60% solids with xylene
Final acid value (thinned) - 40.5
Viscosity - Z_6+

Discarded

AR22-0-1

Raw Materials:

222.0 g	Phthalic Anhydride	1.5 mols
96.6 g	Glycerine	1.0 " + 5% excess
264.0 g	G.F. oil	.34 "
0.5 g	Litharge	
583.1 g	Total	

Of these materials:

264.0 g G.F. Oil
61.6 g Glycerine

were placed in a reacting setup as described in paragraph 22 and alcoholized to the monoglyceride as described in paragraph 25.

Cooking Log of alcoholysis:

Time	Temp.
9:25	Start
9:45	200° C
10:20	230° C
11:45	220° C - Sample cloudy in 2:1 MeOH
12:15	220° C - Sample clear in 2:1 MeOH

To the monoglyceride was added:

222.0 g Phthalic anhydride
35.0 g Glycerine

The cooking was then continued using the solvent method as described in paragraphs 22 and 23.

Cooking Log:

	Time	Temp.	H_2O
	12:35	Start	--
	12:45	195° C	5 cc
	1:00	195° C	14 "
	1:15	195° C	19 "
	1:30	195° C	22 "
	1:45	195° C	24 "
Sample -	2:00	195° C	25 "
	2:50	198° C	27 "

Thinned to 60% solids with xylene
Final acid value (thinned) - 15.0
Viscosity - Z+
Drier added: - 0.3% Pb
.03% Co

AR23-0-1

Raw Materials:

222.0 g Phthalic anhydride	1.5 mols	
96.6 g Glycerine	1.0 "	+ 5% excess
264.0 g Armours T-H oil	.34 "	
0.5 g Litharge		
583.1 g Total		

Of these materials,

264.0 g Armours T-H oil
61.6 g Glycerine

were placed in a reacting setup as described in paragraph 22 and alcoholized to a mono-glyceride as described in paragraph 25.

Cooking Log of the alcoholysis:

Time	Temp.
9:30	Start
9:40	220° C
10:25	215° C
11:00	215° C Sample cloudy with 2:1 MeOH
12:20	215° C Sample clear with 2:1 MeOH

To the monoglyceride was added:
222.0 g Phthalic anhydride
35.0 g Glycerine

The cooking was then continued using the solvent method as described in paragraphs 22 and 23.

Cooking Log:	Time	Temp.	H_2O
	12:35	Start	-
	12:50	170° C	3.cc
	1:15	180° C	12.5 "
	1:30	182° C	16.5 "
	1:50	182° C	18.0 "
	2:00	182° C	18.5 "
	2:15	210° C	20.5 "
	2:30	210° C	21.5 "
A.V. 21 - Sample	3:00	210° C	22.5 "
	3:20	210° C	24.5 "
		Heat Off	

Thinned to 60% solids with xylene
Final acid value (thinned) - 9.2
Viscosity - T+
Drier added: - 0.3% Pb
.03% Co.

AR28-0-1

An attempt to prepare a mixed pentaerythritol, glycerol alkyd, from dehydrated castor oil by alcoholizing to the monoester with pentaerythritol.

Raw Materials:

293.0 g	Dehydrated Castor Oil (Bakers Special)	.33 mol
234.6 g	Pentaerythritol	1.5 " + 15% excess
222.0 g	Phthalic anhydride	1.5 "
0.5 g	Litharge	
750.1 g	Total	

Of these materials:

293.0 g Dehydrated Castor Oil (Bakers Special)
90.6 g Pentaerythritol
0.5 g Litharge

were placed in a reacting setup as described in paragraph 22 and alcoholized to a monoester as described in paragraph 25.

Cooking Log of alcoholysis:

Time	Temp.
9:50	Start
10:05	198° C
10:15	218° C Sample clear in 2:1 MeOH

To the monoglyceride was added:

222 g Phthalic anhydride
144 g Pentaerythritol

The cooking was then continued using the solvent method described in paragraphs 22 and 23.

Cooking Log:

Time	Temp.	H_2O
11:05	Start	-
11:20	182° C	1st drop
11:30	185° C	4 cc
12:10	179° C	10.5 "
12:30	180° C	13 "
1:00	190° C	16 "
1:30	180° C	17.75"
2:00	180° C	19.5 "
2:30	180° C	22 "
3:00	185° C	23 "
3:15	185° C	23.7 "
3:50	235° C	24.5 "
4:00	240° C	27.0 "

Heat off. Thinned to 60% solids with 454 g xylene.

A solid kicked out when the alkyd was thinned and when filtered out amounted to about 200 g material. Evidently the pentaerythritol would not alcoholize properly along with the glycerine, but reacted separately with the phthalic anhydride and fatty acids. Since it was not linked in the chain with the rest of the glyceride, it kicked out of solution when thinned.

AR29-0-1

Preparation of a glycerol alkyd from Atlas Powder K Oil. This oil was known to be a sorbitol ester therefore a molecular weight had to be run to determine the amount of glycerine to use in order to carry out the alcoholysis. The experimentally determined molecular weight was 987 for "K" oil. On this basis the following formula was set up.

Raw Materials:

329.0 g	Atlas Powder "K" oil	.33 mol
101.0 g	Glycerine	1.0 " + 10% excess
222.0 g	Phthalic anhydride	1.5 "
0.5 g	Litharge	
652.5 g	Total	

Of these materials,

329.0 g "K" oil
61.3 g Glycerine
0.5 g Litharge

were placed in a reacting setup as described in paragraph 22 and alcoholized to a mixed mono-ester as described in paragraph 25.

Cooking Log of alcoholysis:

Time	Temp.	
9:10	Start	
9:30	210° C	
1:00	218° C	Sample clear in 2:1 MeOH

To the mixed mono-ester was added:

222.0 g Phthalic anhydride
39.9 g Glycerine

The cooking was then continued using the solvent method as described in paragraphs 22 and 23.

Cooking Log:

	Time	Temp.	H_2O over
	10:25	Start	-
	10:35	180° C	1st drop
	10:50	218° C	8.5 cc
	11:15	218° C	20.5 "
	11:45	218° C	24.5 "
Visc. H at 60% - Sample	12:00	218° C	25.5 "
	12:10	218° C	26.5 "

Thinned to 60% solids with 384 g xylene
Final acid value (thinned) - 15.8
Viscosity - U-V

Drier added - 0.3% Pb
.03% Co.

AR3-0-3

Raw Materials:

635.8 g	Pentaerythritol	4 mols + 15% excess
888.0 g	Phthalic anhydride	6.0 "
1120.0 g	Dehydrated castor fatty acids (Castung Special)	4.0 "
2643.8 g	Total	

This material was placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O over
10:00	Start	-
11:00	158° C	1st drop
11:25	165° C	10 cc
11:30	170° C	40 "
12:00	170° C	80 "
12:35	172° C	96 "
1:00	178° C	106 "
1:30	178° C	113 "
2:00	195° C	122 "
2:30	205° C	133 "
3:00	205° C	143 "
3:15	205° C	147.5"

Thinned to 60% solid with 1649 g xylene

Final acid value (thinned) - 27

Viscosity very heavy - Z_6+

Drier added: - 0.3% Pb
.03 Co.

AR12-1-1

Raw Materials:

700.0 g	Dehydrated castor fatty acids (Castung Special)	2.5 mols
700.0 g	Linseed fatty acids	2.5 "
373.0 g	Phthalic anhydride	2.5 "
395.0 g	Pentaerythritol	2.5 " + 15% excess
2168.0 g	Total	

This material was placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

	Time	Temp.	H_2O
	11:10	Start	--
	11:50	160° C	1st dropp
	12:10	180° C	30.0 cc
	12:35	192° C	66.0 "
	1:10	215° C	98.0 "
	1:30	205° C	105.0 "
	2:15	205° C	111.0 "
	2:45	208° C	117.0 "
	3:45	208° C	121.0 "
Visc. A at 60% solids - Sample	4:15	250° C	127.0 "
	4:45	250° C	130.0 "

Heat off

Thinned to 60% solids with 1355 g xylene

Acid value thinned - 5

Viscosity - B

Drier added; - .3% Pb
.03% Co.

AR4-0-2

Raw Materials:

800.0 g	Dehydrated Castor Acids (Castung Special)	2.86 mols	
320.0 g	Linseed fatty acids	1.14 "	
625.0 g	Pentaerythritol	4.0 "	+ 15% excess
592.0 g	Phthalic anhydride	4.0 "	
2337.0 g	Total		

This material was placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O
8:00	Start	-
9:05	155° C	1st drop
9:15	175° C	15 cc
9:30	190° C	40 "
10:00	190° C	85 "
10:30	200° C	101 "
11:00	198° C	111 "
11:30	205° C	119 "
12:30	210° C	135 "
1:30	210° C	138 "
2:30	238° C	146 "
2:40	238° C	148 "

Heat off

Thinned to 60% solids with 1410 g xylene

Final acid value thinned - 3.2

Drier added: .3% Pb
.03% Co.

AR8-0-2

Raw Materials:

597.4 g	Pentaerythritol	3.5 mols + 23% excess
781.2 g	Phthalic anhydride	5.28 "
576.1 g	Dehydrated castor fatty acids (Castung Special)	2.06 "
64.4 g	Linseed fatty acids	.23 "
161.0 g	Furyl acrylic acid	1.17 "
2180.1 g	Total	

These materials were placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O
8:30	Start	-
9:05	158° C	1st drop
9:30	158° C	21 cc
10:00	160° C	40 "
10:35	184° C	65 "
11:00	195° C	83 "
11:30	198° C	101.5"
12:05	185° C	110.0"
1:00	195° C	125. "

Heat off

Thinned to 60% solids with 1376 g xylene

Final acid value thinned - 31

Viscosity - Z_6+

Drier added: - .3% Pb
.03% Co.

AR9-O-2

Raw Materials:

597.0 g	Pentaerythritol	3.5 mols + 23% excess
781.0 g	Phthalic anhydride	5.25 "
517.0 g	Dehydrated castor fatty acids (Castung Special)	1.82 "
129.0 g	Linseed fatty acids	.49 "
161.0 g	Furyl acrylic acids	1.2 "
2185.0 g	Total	

This material was placed in a reacting setup as described in paragraphs 22 and 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O
8:15	Start	-
9:10	152° C	1st drop
9:30	162° C	21 cc
10:00	168° C	47 "
10:30	174° C	65 "
11:05	190° C	80 "
11:30	198° C	97 "
12:00	198° C	108 "
12:30	198° C	118 "
1:00	210° C	123 "
1:15	210° C	127.5"

Heat off

Thinned to 60% solids with 1355 g xylene.

Final acid value thinned - A.V. 26.5

Viscosity - Z_6

Drier added: - .3% Pb
.03% Co.

AR14-0-2

Raw Materials:

800.0 g	Dehydrated Castor Acids (Castung Special)	2.86 mols
200.0 g	Linseed fatty acids	.71 "
586.4 g	Phthalic anhydride	3.96 "
450.8 g	Pentaerythritol	2.37 " + 15% excess
2037.2 g	Total	

The material was placed in a reacting setup as described in paragraph 23. The solvent process of cooking was used.

Cooking Log:

Time	Temp.	H_2O
9:50	Start	-
10:40	168° C	32 cc
11:30	180° C	65 "
12:05	182° C	83 "
12:30	190° C	91 "
1:00	192° C	98 "
2:00	198° C	109 "
3:00	204° C	118.5"
3:30	204° C	123

Heat off

Thinned to 60% solids with 1273.6 g of xylene

Final acid value (thinned) - 16.3

Viscosity - Z_3+

Drier added: - .3% Pb
.03% Co.

AR15-1-1

Raw Materials:

800.0 g	Dehydrated castor fatty acids (Castung Special)	2.86 mols	
268.8 g	Linseed fatty acids	.96 "	
568.0 g	Phthalic anhydride	3.84 "	
450.1 g	Pentaerythritol	2.88 "	+ 15% excess
2086.9 g	Total		

The material was placed in a reacting setup as described in paragraph 23. The solvent method of cooking was used.

Cooking Log:

	Time	Temp.	H_2O
	9:40	Start	-
	10:45	170° C	1st drop
	11:00	167° C	20 cc
	11:20	174° C	40 "
	12:10	184° C	78 "
	1:10	190° C	88 "
	2:10	195° C	100 "
	3:10	195° C	107 "
Acid value 22.8 Sample	3:50	215° C	112 "
	4:00	215° C	114 "

Heat off

Thinned to 60% solids with xylene

Viscosity - W

Final acid value (thinned) - 14.5

Drier added: - .3% Pb
.03% Co.

ARI6-0-2

Raw Materials:

537.6 g	Dehydrated castor fatty acids (Castung Special)	1.92 mols.
132.4 g	Furyl acrylic acid	.96 "
268.8 g	Linseed fatty acids	.96 "
568.0 g	Phthalic anhydride	3.84 "
450.0 g	Pentaerythritol	2.98 " + 15% excess
1956.8 g	Total	

The material was placed in a reacting setup as described in paragraph 23. The solvent process of cooking was used.

Cooking Log:

Time	Temp.	H_2O
11:15	Heat on	-
11:50	170° C	1st drop
12:20	170° C	21 cc
12:50	180° C	39 "
1:20	204° C	63 "
2:20	210° C	81 "
3:30	205° C	95 "
3:40	205° C	95 "

Heat off

Thinned to 60% solids with 1314 g xylene

Final acid value (thinned) - 24

Viscosity - x to y

Drier added: - .3% Pb
.03% Co.

AR17-0-2

Raw Materials:

605.0 g	Dehydrated castor fatty acids	2.16 mols
149.0 g	Furyl acrylic acids	1.08 "
g	Phthalic anhydride	4.32 "
447.3 g	Glycerine	4.32 " + 12.3% excess
302.4 g	Linseed Fatty acids	1.06 "
2142.7 g	Total	

This material was placed in a reacting setup as described in paragraph 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O
10:25	Start	–
11:00	158° C	1st drop
11:30	168° C	29 cc
12:00	174° C	62 "
12:30	184° C	80 "
1:30	188° C	113 "
2:00	190° C	123 "
2:30	204° C	129 "
3:30	210° C	142.5 "
4:00	210° C	148.0 "

Heat off

Thinned to 60% solids with 1314 g xylene

Final acid value (thinned) - 13.8

Viscosity - F.G.

Drier added: - .3% Pb
.03% Co.

AR19-0-3

Raw Materials:

666.0 g	Phthalic anhydride	4.5 mols.	
289.0 g	Glycerine	3.0 "	+ 5% excess
792.0 g	Bakelite RD-43-26	.9 "	
1.0 g	Litharge		
1748.0 g	Total		

Of these materials,

792.0 g RD-43-26
184.0 g Glycerine
1.0 g Litharge

were placed in a reacting setup as described in paragraph 23 and alcoholized to a mono-glyceride as described in paragraph 25.

Cooking log of alcoholysis:

Time	Temp.
12:00	Start
12:30	200° C
12:45	200° C
1:15	210° C
1:30	210° C - Sample clear with 2:1 MeOH

To the mono-glycerine was added:

666.0 g Phthalic anhydride
104.2 g Glycerine

The cooking was then continued by the solvent method as described in paragraph 23.

Cooking Log:

Time	Temp.	H_2O
1:45	160° C	1st drop
2:20	178° C	25 cc
3:00	188° C	45 "
3:30	194° C	56 "
4:00	210° C	66 "
4:25	210° C	73 "

Heat off

Thinned to 60% solids with 1163 g xylene
Final acid value (thinned) - 24
Viscosity - R
Drier added: - .3% Pb
.03% Co.

AR22-0-2

Raw Materials:

666.0 g	Phthalic anhydride	4.5 mols
278.8 g	Glycerine	3.0 "
792.0 g	G.F. oil (DuPont)	1.07 "
1.0 g	Litharge	
1637.8 g	Total	

Of these materials,

792.0 g G.F. oil
184.8 g Glycerine

were placed in a reacting setup as described in paragraph 23 and alcoholized to the mono-glyceride as described in paragraph 25.

Cooking Log of alcoholysis:

Time	Temp.
11:00	Start
11:20	200° C
11:45	210° C - Sample cloudy with 2:1 MeOH
12:00	210° C - Sample clear with 2:1 MeOH

To the mono-glyceride was added:

666.0 g Phthalic anhydride
105.0 g Glycerine

The cooking was then continued by the solvent method as described in paragraph 23.

Cooking Log:

Time	Temp.	H_2O
1:35	Start	-
2:00	168° C	1st drop
2:30	185° C	18 cc
3:00	190° C	35 "
3:30	190° C	50 "
4:20	220° C	69 "

Heat off

Thinned to 60% solids with 1126 g of xylene
Final acid value (thinned) - 23
Viscosity - T-U
Drier added: - .3% Pb
.03% Co

AR23-1-1

Raw Materials:

666.0 g	Phthalic anhydride	4.5 mols
303.0 g	Glycerine	1.0 " + 10% excess
792.0 g	Armours T-H oil	.9 "
1.0 g	Litharge	
1762.0 g	Total	

Of these materials,
792.0 g T-H oil
184.8 g Glycerine

were placed in a reacting setup as described in paragraph 23 and alcoholized to the mono-glyceride.

Cooking Log of alcoholysis:

Time	Temp.
10:30	Start
10:50	210° C
11:45	210° C - Sample clear with 2:1 MeOH

To the mono-glyceride was added:
666.0 g Phthalic anhydride
118.2 g Glycerine

The cooking was then continued, using the solvent method as described in paragraph 23.

Cooking Log:

Time	Temp.	H_2O
11:55	Start	-
12:25	162° C	20 cc
12:45	181° C	35 "
1:35	181° C	53.5"
2:00	210° C	63.0"
3:15	210° C	64.0"

Heat off.

Thinned to 60% solids with 1126 g xylene
Final acid value (thinned) - 13
Viscosity - U-V
Drier added: - .3% Pb
.03% Co.

AR31-0-1

Raw Materials:

880.0 g	Dehydrated Castor Oil (Castung Special)	1 mol
550.0 g	Phthalic anhydride	3.7 "
262.0 g	Glycerine	2.5 " + 15% excess
1.0 g	Litharge	
1693.0 g	Total	

Of these materials,

880.0 g Castung Special
148.0 g Glycerine
1.0 g Litharge

were placed in a reacting setup as described in paragraph 23.

Cooking Log:

Time	Temp.
10:45	190° C
11:40	210° C Sample not clear in 2:1 MeOH
12:30	210° C Sample clear in 2:1 MeOH

To the mono-glyceride was added:

550.0 g Phthalic anhydride
78.0 g Glycerine

The cooking was continued by the solvent method as described in paragraph 23.

Cooking Log:

Time	Temp.	H_2O
1:00	Start	-
1:20	178° C	8 cc
2:15	180° C	42 "
3:30	185° C	59 "
4:10	187° C	63 "

Heat off

Thinned to 60% solids with 1095 g xylene

Final acid value (thinned)- 14,2

Viscosity - G-H

Drier added: - .3% Pb
.03% Co.

AR4-5-1

Raw Materials:

700.0 g	Dehydrated castor fatty acids (Isoline)	2.5 mols
280.0 g	Linseed fatty acids	1.0 "
483.0 g	Pentaerythritol	3.5 "
518.0 g	Phthalic anhydride	3.5 "
1981.0 g	Total	

This material was placed in a reacting setup as described in paragraph 23. The solvent method of cooking was used.

Cooking Log:

	Time	Temp.	H_2O
	8:10	Start	-
	9:10	160° C	1st drop
	9:30	176° C	20 cc
	9:55	190° C	49 "
	10:55	210° C	89 "
	12:35	210° C	108 "
Acid Value 18 - Sample	2:00	209° C	112 "
	2:50	210° C	113 "

Heat off

Thinned to 60% solids with 1238 g xylene

Final acid value thinned - 7.9

Viscosity - Z_3

Drier added: - .3% Pb
.03% Co.

AR2-1-1

Raw Materials:

351.9 g	Pentaerythritol	2.25 mols + 15% excess
333.0 g	Phthalic anhydride	2.25 "
207.0 g	Furyl acrylic acid	1.5 "
840.0 g	Dehydrated castor fatty acids (Isoline)	3.0 "
1731.9 g	Total	

The material was placed in a reacting setup as described in paragraph 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O
8:35	Start	-
9:05	160° C	1st drop
9:30	169° C	22 cc
10:40	195° C	73 "
1:15	200° C	96 "
1:40	215° C	98 "
2:40	214° C	105 "
3:40	214° C	108.5 "
4:00	214° C	109 "

Heat off

Thinned to 60% solids with 1070 g xylene

Final acid value (thinned) - 15.6

Viscosity - G-H

Drier added: - .3% Pb
.03% Co.

AR3-3-1

Raw Materials:

800.0 g	Dehydrated castor fatty acids (Isoline)	2.86 mols
558.0 g	Phthalic anhydride	3.84 "
407.0 g	Pentaerythritol	2.6 " + 15% excess
1765.0 g	Total	

The material was placed in a reacting setup as described in paragraph 23. The solvent method of cooking was used.

Cooking Log:

	Time	Temp.	H_2O
	9:00	Start	–
	9:40	167° C	1st drop
	10:02	172° C	20 cc
	10:27	172° C	41 "
	11;45	185° C	68 "
	1:05	203° C	89 "
Thinned (acid	2:40	205° C	100 "
value 19.7 60% solids) Sample Visc. E–F	3:25	203° C	103 "

Heat off

Thinned to 60% solids with 1100 g xylene

Final acid value (thinned) – 17.9

Viscosity – Z_1

Driers added: – .3% Pb
.03% Co.

AR2-0-5

Raw Materials:

351.0 g	Pentaerythritol	2.25 mols + 15% excess
333.0 g	Phthalic anhydride	2.25 "
207.0 g	Furyl acrylic acids	1.5 "
840.0 g	Dehydrated castor fatty acids (Castung Special)	3.0 "
1731.0 g	Total	

The material was placed in a reacting setup as described in paragraph 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O over
8:40	Start	–
9:15	162° C	1st drop
9:30	190° C	51 cc
10:00	200° C	66 "
10:30	212° C	86 "
12:00	215° C	103 "
12:30	212° C	107 "
2:00	223° C	111 "

Heat off

Thinned to 60% solids with 1070 g xylene

Final acid value (thinned) - 12.5

Viscosity - Y

Drier added: - .3% Pb
.03% Co.

AR8-1-1

Raw Materials:

710.0 g	Phthalic anhydride	4.8 mols
630.0 g	Dehydrated castor fatty (acids (Isoline)	2.25 "
71.5 g	Linseed fatty acids	.25 "
179.0 g	Furyl acrylic acids	1.3 "
519.0 g	Pentaerythritol	3.33 " + 15% excess
2109.5 g	Total	

The material was placed in a reacting setup as described in paragraph 23. The solvent method of cooking was used.

Cooking Log:

Time	Temp.	H_2O over
9:30	Start	
10:00	165° C	1st drop
10:25	172° C	21 cc
10:45	172° C	42 "
11:20	185° C	63 "
12:00	188° C	83 "
1:00	200° C	100 "
2:00	206° C	115 "
3:00	210° C	120 "

added 10 cc Butanol and thinned down with xylol from amount to be used as thinner until refluxing temperature was 140° C.

3:40	140° C	124 cc

Heat off

Thinned to 60% solids with 1300 g solvent

Final acid value (thinned) – 29.9

Viscosity – Z_6+

Drier added: – .3% Pb
.03%Co.

APPENDIX B

DR7-0-2

Raw Materials:

270.0 g	Esskol
225.0 g	BR-254 (Bakelite)
90.4 g	ZnO
585.4 g	Total

The oil and resin were placed together in a stainless steel kettle equipped with a thermometer and stirring paddle.

Cooking Log:

Time	Temp.
1:45	Start
2:00	305°C
2:27	305°C very heavy string; added ZnO and stirred in thoroughly.
2:35	275°C Heat off.

Thinned with 585 g xylene.

DR10-0-2

Raw Materials:

216.0 g	G.F. oil
180.0 g	BR-254 (Bakelite)
72.0 g	ZnO
468.0 g	Total

The oil and resin were placed together in a 1500 cc beaker equipped with a thermometer and stirring apparatus.

Cooking Log:

Time	Temp.
9:45	Start
9:55	305°C
10:30	305°C began to string; added ZnO and stirred in thoroughly.
10:40	220°C Heat off.

Thinned with 468 g of xylene.

DR10-0-3

Raw Materials:

270.0 g	G.F. oil
255.0 g	BR-254 (Bakelite)
90.0 g	ZnO
615.0 g	Total

The oil and resin were placed in a stainless steel kettle equipped with a thermometer and stirring apparatus.

Cooking Log:

Time	Temp.
3:25	Start
3:55	305°C
4:05	305°C began to string; added ZnO and stirred in thoroughly.
4:15	280°C Heat off

Thinned with 585 g of xylene.

DR11-0-3

Raw Materials:

216.0 g	G.F. oil
180.0 g	BR-254 (Bakelite)
72.0 g	ZnO
468.0 g	Total

The oil and resin were placed in a stainless steel kettle equipped with a thermometer and stirring apparatus.

Cooking Log:

Time	Temp.
9:55	190°C
10:05	190°C
10:15	305°C
10:55	305°C
11:00	305°C began to string; added ZnO and stirred in thoroughly.
11:10	305°C
11:20	280°C Heat off

Thinned with 468 g of xylene.

DR11-0-4

Raw Materials:

432.0 g	G.F. oil
360.0 g	BR-254 (Bakelite)
154.0 g	ZnO
946.0 g	Total

The oil and resin were placed together in a stainless steel kettle equipped with a thermometer and stirring apparatus.

Cooking Log:

Time	Temp.
10:35	Start
11:05	260°C
11:19	305°C
11:30	305°C began to string; added ZnO and stirred in well.
11:38	305°C
11:48	280°C Heat off.

Thinned to 50% solids with 964 g of xylene