Patented Apr. 8, 1952 2,592,204

UNITED STATES PATENT OFFICE

2,592,204

MINERAL OIL COMPOSITION

John F. Socolofsky, Woodbury, and Ralph V. White, Pitman, N. J., assignors to Socony-Vacuum Oil Company, Incorporated, a corporation of New York

No Drawing. Application February 15, 1949, Serial No. 76,647

7 Claims. (Cl. 252—34)

1

This invention relates to lubricants, and it is more particularly concerned with lubricants for ferrous metal surfaces in an environment wherein these surfaces are subject to contact with salt water, and, accordingly, are very prone to rusting.

As is well known to those familiar with the art, rusting of ferrous metal surfaces is frequently encountered during the operation of steam turbines, particularly during the initial operation of new installations. Such rusting is most pronounced at points where the clearance between bearing surfaces is very small, such as in the governor mechanism. Manifestly, this constitutes a menace to the operational life of the turbine. When rusting is very severe, particles of rust may form on the main bearing surfaces, or be carried there by the circulating oil, and severely injure these bearings. This is usually caused by water entering the oil supply, as by condensation, and being entrained by the oil throughout the circulating system.

During the operation of marine turbines, the aforementioned rusting is more frequently encountered. This is due to the fact that in marine operation, the same lubricating oil circulates through the main drive gear system and often, through the hydraulic system, as well as through the steam turbine lubricating system. As a consequence, appreciable amounts of salt water are entrained by the oil and, accordingly, rusting is more severe and occurs more readily.

Many materials have been proposed as addition agents for turbine lubricating oils to prevent rusting of ferrous metal surfaces. For example, the use of organic esters has been suggested, but their use is disadvantageous and undesirable in that these materials produce oils having other undesirable characteristics, such as poor emulsion properties. As is well known, emulsions seriously reduce or impair the high degree of lubricating efficiency required in turbine operation, and are to be carefully avoided.

In United States Letters Patent No. 2,436,272, in which the present inventors are coinventors, there was disclosed a lubricating oil containing an ester reaction product of malic acid with a long-chain alcohol and a short-chain alcohol. This oil afforded excellent protection against rusting in the presence of distilled water and, also, did not give rise to emulsion difficulties. However, when this oil was tested in the presence of salt water, it was found that a large percentage of failures was encountered. These failures were due to two factors, namely, (1) rusting of ferrous metal surfaces and (2) staining of these surfaces during certain periods of the year. The staining was a phenomenon which would occur only on certain occasions. Nevertheless, it is considered deleterious to turbine operation.

2

It has now been discovered that an ester of malic acid derived from a long-chain alcohol and a short-chain alcohol can be prepared which, when blended in a mineral lubricating oil, affords an oil which protects ferrous metal surfaces from rusting in the presence of salt water. It has now been found that an ester reaction product of malic acid with a long-chain alcohol and a short-chain alcohol, prepared in accordance with a procedure to be described hereinafter, and then treated with ammonia or with a basic amine, will blend with a mineral lubricating oil to afford a lubricant which protects ferrous metal surfaces from rusting in the presence of salt water.

Accordingly, it is a broad object of the present invention to provide a new lubricating oil composition. Another object is to provide an improved steam turbine lubricant. Still another object is to provide an ester product which can be added to a lubricating oil to provide an oil which will protect ferrous metal surfaces from rusting in the presence of salt water. A further object is to provide a process for preparing said ester product. A more specific object is to provide an ester reaction product of malic acid with a long-chain alcohol, and a short-chain alcohol which, when added to a mineral lubricating oil, will render said oil capable of protecting ferrous metal surfaces from rusting in the presence of salt water. Other objects and advantages of the present invention will become apparent to those skilled in the art from the following detailed description.

Broadly stated, the present invention provides a mineral oil which normally permits rusting and staining of ferrous metal surfaces in the presence of salt water containing a minor proportion, sufficient to prevent said rusting and staining of ferrous metal surfaces, of an ester product obtained by reacting malic acid with a short-chain, monohydroxy, aliphatic alcohol having between about six and about eight carbon atoms per molecule, and with a long-chain, monohydroxy, aliphatic alcohol having at least about twelve carbon atoms per molecule, in a molar proportion of about 1:0.8–1.6:1.2–0.4, respectively, at a temperature varying between about 120° C. and about 140° C., while removing the water of esterification as it is evolved; adding a hydrocarbon solvent which forms an azeotropic mixture with water, and removing essentially all of the remaining water of esterification formed in the reaction, by azeotropic distillation at a liquid temperature varying between about 130° C. and about 150° C.; treating the resulting reaction mixture with a basic nitrogen compound at a temperature varying between about 90° C. and about 100° C.; and separating the thus-treated ester reaction product from said hydrocarbon solvent.

The short-chain alcohol reactant may be any

aliphatic, monohydroxy alcohol having between about six and about eight carbon atoms per molecule. It may be saturated or unsaturated, and straight-chained, branched-chained, or cyclic in structure. A saturated, branched chain alcohol is especially preferred, however. Non-limiting examples of the short-chain aliphatic alcohol reactant are hexanol-1, cyclohexanol, 2-methylpentanol-1, 2-ethyl-butanol-1, heptanol-1, 2-methylhexanol-1, octanol-1, and 2-ethylhexanol-1.

The long-chain alcohol reactant may be any aliphatic, monohydroxy alcohol having between about twelve and about eighteen carbon atoms per molecule. As in the case of the short-chain alcohol reactant, this reactant may be saturated or unsaturated, and it may have a straight-chain, a branched-chain, or cyclic structure. The preferred long-chain alcohol reactant is a straight-chain, unsaturated alcohol. By way of non-limiting examples of the long-chain alcohol reactant may be mentioned dodecanol-1, 7-ethyl-2-methylundecanol-1, hexadecanol-1, octadecanol-1, octadecan-9-ol-1, and octadecadien-9,12-ol-1. Oleyl alcohol is especially preferred.

The molar ratio of the reactants is a critical factor. If the ester product is made using a large proportion of short-chain alcohol reactant, the antirust property will be lacking. Conversely, if a large proportion of the long-chain alcohol reactant is used, the ester product will produce an oil with poor emulsion characteristics. Accordingly, and as stated hereinbefore, the reactants must be reacted in a molar ratio of between about 0.8 mol and about 1.6 mols of short-chain alcohol and between about 1.2 mols and about 0.4 mol of long-chain alcohol for each mol of malic acid. The preferred ester product is prepared by using equimolecular proportions of the reactants, i. e., about one mol of short-chain alcohol and about one mol of long-chain alcohol per mol of malic acid. It has been found that small amounts of the short-chain alcohol reactant are unavoidably removed with the water of esterification during the initial stages of the reaction, due to the temperature conditions employed. Accordingly, in practice it is preferred to use a slight molar excess of the short-chain alcohol to compensate for this loss. Excesses, in amounts varying between about 0.02 mol and about 0.05 mol per mol of short-chain alcohol are entirely satisfactory.

In accordance with the present invention, the water of esterification formed in the reaction is removed from the reaction mixture as quickly as it is formed. This is accomplished by heating the reactants initially at temperatures varying between about 120° C. and about 140° C. for a relatively short period of time, in a reaction system from which water can be removed as it is formed. A satisfactory system, for example, is a reaction vessel provided with a reflux condenser having a take-off trap from which water can be removed. As those skilled in the art will readily appreciate, the duration of the initial heating period will vary with the temperature employed. Shorter periods of time will be required when higher temperatures are employed. Generally, the initial heating is carried out for a period of time varying between about 15 minutes and about 60 minutes. The preferred conditions for the initial reaction stage are a temperature varying between about 130° C. and about 140° C. and a period of time of about 30 minutes.

In order to remove the remaining water of esterification, a hydrocarbon solvent which forms an azeotropic mixture with water is added to the reaction mixture at the close of the initial reaction period. Heating is continued at the same or at somewhat higher liquid temperatures until essentially all of the water of esterification which will be produced theoretically to achieve complete esterification has been removed by azeotropic distillation. In general, any hydrocarbon solvent which forms an azeotropic mixture with water can be used in the present process. It is preferred, however, to use an aromatic hydrocarbon solvent of the benzene series. Non-limiting examples of the preferred solvent are benzene, toluene, and xylene. As will be readily appreciated by those skilled in the art, the amount of solvent used is a variable and non-critical factor, and it is dependent on the size of the reaction vessel and the reaction temperature selected. Accordingly, sufficient solvent must be used to support the azeotropic distillation, but a large excess must be avoided since the reaction temperature will be lowered thereby.

As mentioned hereinbefore, this stage of the reaction may be carried out at liquid temperatures somewhat higher than those employed during initial stage. Generally, the liquid temperatures will vary between about 130° C. and about 150° C. The preferred temperature, however, is the same as that used during the initial reaction stage, i. e., varying between about 130° C. and about 140° C. The reaction temperature should not exceed about 150° C., since malic acid decomposes at that temperature.

The second stage of the reaction is carried out until essentially all of the water of esterification is removed, which will be theoretically evolved to achieve complete esterification, i. e., until essentially two mols of water have been evolved for each mol of malic acid. It will be appreciated that it is practically impossible to obtain all of the water theoretically possible, but the amount not evolved will be negligible. Therefore, the duration of the second stage of the reaction will vary with the temperature and with the particular reactants employed. It will vary, generally, between about four hours and about eight hours. For example, when 2-ethylhexanol-1 and oleyl alcohol are used in equimolar quantities as the alcohol reactants, using toluene as the solvent, at the preferred temperature range, it has been found that the second stage of the reaction is complete in between about five and about six hours.

Upon completion of this second stage of the reaction, the reaction product is treated with a basic nitrogen compound. In general, the basic nitrogen compounds contemplated herein are any nitrogen-containing compounds which are basic chemically, and which are capable of adding to the acidic hydrogen atom of an acidic substance in a neutralization reaction. In practice, however, the basic nitrogen compounds are ammonia; compounds having a structure similar to that of ammonia, such as organic amines; and the basic heterocyclic compounds having one or more nitrogen atoms in the ring. Non-limiting examples of the basic nitrogen compound reactant are ammonia, methylamine, dimethylamine, trimethylamine, tri-iso-amylamine, 2-ethylhexylamine, decylamine, benzylamine, benzylethylamine, benzyldiethylamine, aniline, methylaniline, ethylaniline, diethylaniline, β-naphthylamine, hydrazine, pyridine, piperidine, ethanolamine, ethylenediamine, and p-phenylenediamine. The

treatment of the reaction product with these basic nitrogen compounds is accomplished, usually, by cooling the reaction mixture and then adding the desired basic nitrogen compound, with good agitation of the reaction mixture. The preferred temperature for this treatment varies between about 90° C. and about 100° C. The amount of basic nitrogen compound used is small but not too critical. It varies preferably between about 0.005 mol and about 0.01 mol per mol of malic acid used in the reaction.

The separation of the final product is accomplished by removing the solvent and filtering the residue. The solvent is removed, preferably, by distillation of the reaction mixture, under reduced pressure. The temperature of distillation is not too critical and it is a function of the pressure. In no case, however, should the temperature used for solvent removal exceed the highest temperature employed during the reaction. It has been found advantageous, during the initial stages of solvent removal, to use a relatively low vacuum and a temperature similar to the temperature employed during the treatment with the basic nitrogen compound. Also, it has been found advantageous to complete the removal of solvent under a pressure of about 20 millimeters at about 135° C.

The following specific examples are for the purpose of illustrating the process of the present invention and to demonstrate the superiority of lubricants containing the products contemplated herein. It is to be strictly understood, however, that the invention is not to be limited to the specific solvent and reactants set forth hereinafter, or to the operations and manipulations described therein. As will be apparent to those skilled in the art, a wide variety of other reactants and solvents, as set forth hereinbefore, may be used to prepare the products of the present invention.

EXAMPLE 1

A mixture of 93.8 grams (0.7 mol) of malic acid, 187.6 g. (0.7 mol) of oleyl alcohol, and 93.8 grams (0.72 mol) of 2-ethylhexanol-1 was placed in a three-neck flask provided with a reflux take-off, thermometer, and a mechanical stirrer. Heat was applied and the first trace of water was distilled off when a temperature of 133° C. was reached. After thirty minutes, the temperature was 140° C. and 9.6 cubic centimeters of water had been collected in the reflux take-off. Toluene (125 cubic centimeters) was added to the flask, and the heat input was adjusted so that refluxing occurred at a temperature of about 140° C. This refluxing was maintained for five hours, at the end of which time, 24 cubic centimeters of water had been collected. The flask contents were cooled to a temperature of 95–100° C. and 0.12 grams of ammonia gas was introduced. Then the toluene was removed at a temperature of 100–115° C. under a pressure of about one hundred millimeters. The residue remaining was filtered through a layer of diatomaceous silica. Test data for oil blends of this product are set forth in Table I.

EXAMPLES 2, 3, 4, 5, 6 AND 7

Other runs were made in the same manner as set forth in Example 1. Pertinent test data for lubricating oils containing these products are given in Table I. In two of these runs, namely, runs 2 and 3, benzene was used in place of toluene for the azeotropic distillation. These runs are identified by the notation (1) in Table I.

The runs in the preceding examples were made in accordance with the procedure of the present invention and they are illustrative of the technique involved. The following examples are included for comparison purposes. They set forth illustrations of runs made by the procedure set forth in the aforementioned United States Letters Patent, No. 2,436,272.

EXAMPLE 8

A mixture of 20.1 grams (0.15 mol) of malic acid, 40.2 grams (0.15 mol) of oleyl alcohol, and 23.5 grams (0.18 mol) of 2-ethylhexanol-1 were digested for 3.5 hours at 115–120° C., in a three-neck flask provided with a reflux take-off, a thermometer, and a mechanical stirrer. Then, about 30 cubic centimeters of toluene were added to the reaction vessel, and the heat input was regulated so that toluene reflux occurred with the flask contents at a temperature of about 140° C. After seven hours, 4.9 cubic centimeters of water had been collected in the reflux take-off, and the evolution of water had apparently ceased. The reaction was stopped, and the reaction mixture was filtered. Toluene was removed from the reaction product at a pot temperature of 100° C. under a pressure of 4–5 millimeters. Test data for this product blended in a lubricating oil are set forth in Table II.

EXAMPLES 9, 10, 11, 12, 13 AND 14

Several additional runs were made by a procedure identical with that used in Example 8, to provide additional samples for comparison purposes. The test results for oil blends of these products appear in Table II.

TABLE I

Blends containing malic acid ester product made by new procedure

Run	N. N. of Product [1]	Conc. in Oil, Percent	Emulsion Test, Break, Min.		Rust Test—No. of Tests Which:	
			Dist. Water	Percent NaCl	Pass	Fail
1	28.0	0.25	16	21	4	0
2 [2]	23.0	0.25	14	16	4	0
3 [2]	28.0	0.25	13	21	4	0
4	22.7	0.25	16	23	3	0
5	23.9	0.25	15	17	3	0
6	22.0	0.25	18	21	6	0
7	23.0	0.25	18	21	4	2

[1] Number of milligrams KOH required to neutralize one gram of product.
[2] Benzene was used instead of toluene for the azeotropic distillation.

TABLE II

Blends containing malic acid product made by prior procedure

Run	N. N. of Product [1]	Conc. in Oil, Percent	Emulsion Test, Break, Min.		Rust Test—No. of Tests Which:	
			Dist. Water	Percent NaCl	Pass	Fail
8	25.0	0.25	14	12	0	2
9	18.0	0.25	16	16	50%	
10	18.0	0.25	24	22	0	4
11	24.5	0.25	14	23	5	[1] 13
12	25.5	0.25	17	22	1	[2] 10
13	20.8	0.25	13	15	0	[2] 12
14	24.0	0.25	14	12	0	[3] 4

[1] Number of milligrams KOH required to neutralize one gram of product.
[2] Seven fails due to stain.
[3] Eight fails due to stain.
[4] Fails due to stain.

To demonstrate the outstanding character of the oil compositions contemplated herein, typical rust test and emulsion test data were obtained for oil blends of the reaction product described in the examples. The oil used for test purposes was a blend of solvent-refined, Mid-Continent residual stock with a solvent-refined, Mid-Continent Rodessa distillate stock. It had an A. P. I. gravity of 30.8, a flash point of 445° F., and a Saybolt Universal viscosity of 407.7 seconds at 100° F. There were blended in this oil 0.2 per cent of 2,6-di-t-butyl-4-methylphenol and 0.1 per cent of phenyl-α-naphthylamine, well-known antioxidants for lubricating oils. This oil is suitable for use in steam turbines and the like, and the tests run on the oil and oil blends simulate conditions existing in turbine operations and analogous operations.

The test method used to distinguish the rusting characteristics of lubricating oil blends is derived from the Bureau of Ships Ad Interim Specification 14–0–15 (INT), dated March 1, 1943. In this test a flat polished steel specimen is suspended in 300 cubic centimeters of the oil under test, at a temperature of 140° F. After 30 minutes, 30 cubic centimeters of synthetic sea water are added and the mixture is stirred with an agitator revolving at 750 R. P. M. throughout the test period. After 48 hours have elapsed, the specimen is removed and examined for evidence of rust and/or staining. The oil passes the test when there is no evidence of rust or staining thereon. The synthetic sea water used in this test contains 25 grams of sodium chloride, 11 grams of magnesium chloride hexahydrate, 4 grams of sodium sulfate, and 1.2 grams of calcium chloride per liter of water; and it simulates the composition of actual sea water.

The emulsion test used herein is the emulsion test for lubricating oils (Method 320.13) set forth in the Federal Stock Catalog, section IV, part 5, Federal Specifications VV–L–791*b*, February 19, 1942. In this test, 40 cubic centimeters of oil to be tested and 40 cubic centimeters of emulsant, either distilled water or a one per cent aqueous solution of sodium chloride, are stirred with a paddle at 1500 R. P. M. for 5 minutes in a 100-cubic centimeter cylinder, at a temperature of 130° F. Separation of the emulsion formed is observed while the cylinder is kept at 130° F., for a specified time interval. Figures given in the tables are the number of minutes which elapse before there is no continuous layer of emulsion between the oil and the emulsant. With an oil having the viscosity characteristics of the oil used herein, specifications required that there be no continuous layer of emulsion at the interface after a 30-minute period.

It will be apparent from a comparison of the data set forth in Tables I and II that the present invention provides a process for making a malic acid ester-type antirust additive which, is entirely satisfactory for use in steam turbine lubricants which are to be used under operating conditions wherein there is great likelihood of rusting due to the action of sea water. In addition, staining of the steel has been completely eliminated.

As is well known to those skilled in the art, other additives may be added to the lubricating oil in addition to the antirust additive, to impart to the oil greater resistance to oxidation, greater film strength, etc. Accordingly, there may be added well-known phenols, amines, organic compounds containing phosphorous and/or halogens, etc.

The malic acid esters of the present invention may be used in oils in amounts varying between about 0.1 per cent and up to about 10.0 per cent by weight. Preferred concentrations vary between about 0.1 per cent and about 1.0 per cent. The higher concentrations, i. e., above one per cent, are preferred for use in cutting oils and the like.

Mineral oil concentrates are also contemplated in this invention, such concentrates containing substantially larger amounts of the ester product than set forth hereinbefore. Thus, relatively large amounts, i. e., upwards of about ten per cent by weight and up to about 49 per cent of said ester product, may be incorporated in an oil fraction. The oil concentrate thus obtained may thereafter be diluted with a suitable quantity of mineral oil, prior to use, to produce a mineral oil composition containing the desired optimum concentration of ester product.

Although the present invention has been described with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of this invention, as those skilled in the art will readily understand. Such variations and modifications are considered to be within the purview and scope of the appended claims.

We claim:

1. A mineral oil which normally permits rusting and staining of ferrous metal surfaces in the presence of salt water, containing a minor proportion, sufficient to prevent said rusting and staining of an ester product of malic acid obtained by reacting malic acid with a short-chain, aliphatic alcohol having between about six and about eight carbon atoms per molecule, and with a long-chain, aliphatic alcohol having at least about twelve carbon atoms per molecule, in a molar proportion of about 1:8–1.6:1.2–0.4, respectively, at a temperature varying between about 120° C. and about 140° C., concurrently removing the water of esterification as it is evolved; adding a hydrocarbon solvent which forms an azeotropic mixture with water, removing essentially all of the remaining water of esterification formed in the reaction by azeotropic distillation at a liquid temperature varying between about 130° C. and about 150° C.; treating the resulting reaction mixture with a basic nitrogen compound selected from the group consisting of ammonia and organic amines, at a temperature varying between about 90° C. and about 100° C.; and separating the thus treated ester product from said hydrocarbon solvent.

2. A mineral oil which normally permits rusting and staining of ferrous metal surfaces in the presence of salt water, containing a minor proportion, sufficient to prevent said rusting and staining, of an ester product of malic acid obtained by reacting malic acid with a branched, short-chain, aliphatic alcohol having between about six and about eight carbon atoms per molecule, and with a long-chain aliphatic alcohol having between about twelve and about eighteen carbon atoms per molecule, in a molar proportion of about 1:0.8–1.6:1.2–0.4, respectively, at a temperature varying between about 120° C. and about 140° C., concurrently removing the water of esterification as it is evolved; adding a hydrocarbon solvent which forms an azeotropic mixture with water, removing essentially all of

the remaining water of esterification formed in the reaction by azeotropic distillation at a liquid temperature varying between about 130° C. and about 150° C.; treating the resulting reaction mixture with a basic nitrogen compound selected from the group consisting of ammonia and organic amines, at a temperature varying between about 90° C. and about 100° C.; and separating the thus treated ester product from said hydrocarbon solvent.

3. A mineral oil which normally permits rusting and staining of ferrous metal surfaces in the presence of salt water, containing a minor proportion, sufficient to prevent said rusting and staining, of an ester product of malic acid obtained by reacting malic acid with 2-ethylhexanol-1, and with oleyl alcohol, in a molar proportion of about 1:0.8–1.6:1.2–0.4, respectively, at a temperature varying between about 130° C. and about 140° C., concurrently removing the water of esterification as it is evolved; adding a hydrocarbon solvent which forms an azeotropic mixture with water, removing essentially all of the remaining water of esterification formed in the reaction by azeotropic distillation at a liquid temperature varying between about 130° C. and about 140° C.; treating the resulting reaction mixture with a basic nitrogen compound selected from the group consisting of ammonia and organic amines, at a temperature varying between about 90° C. and about 100° C.; and separating the thus treated ester product from said hydrocarbon solvent.

4. A mineral oil which normaly permits rusting and staining of ferrous metal surfaces in the presence of salt water, containing between about 0.1 per cent and about 10.0 per cent of an ester product of malic acid obtained by reacting malic acid with 2-ethylhexanol-1, and with oleyl alcohol, in a molar proportion of about 1:1:1, respectively, at a temperature varying between about 130° C. and about 140° C., concurrently removing the water of esterification as it is evolved; adding toluene, removing essentially all of the remaining water of esterification formed in the reaction by azeotropic distillation at a liquid temperature varying between about 130° C. and about 140° C.; treating the resulting reaction mixture with ammonia at a temperature varying between about 90° C. and about 100° C.; and separating the thus treated ester product from said toluene.

5. A process for the production of an ester product of malic acid, which comprises reacting malic acid with a short-chain, aliphatic alcohol having between about six and about eight carbon atoms per molecule, and with a long-chain, aliphatic alcohol having at least about twelve carbon atoms per molecule, in a molar proportion of about 1:0.8–1.6:1.2–0.4, respectively, at a temperature varying between about 120° C. and about 140° C., concurrently removing the water of esterification as it is evolved; adding a hydrocarbon solvent which forms an azeotropic mixture with water, removing essentially all of the remaining water of esterification formed in the reaction by azeotropic distillation at a liquid temperature varying between about 130° C. and about 150° C.; treating the resulting reaction mixture with a basic nitrogen compound at a temperature varying between about 90° C. and about 100° C.; and separating the thus treated ester product from said hydrocarbon solvent.

6. A process for the production of an ester product of malic acid, which comprises reacting malic acid with a short-chain, aliphatic alcohol having between about six and about eight carbon atoms per molecule, and with a long-chain, aliphatic alcohol having between about twelve and about eighteen carbon atoms per molecule, in a molar proportion of about 1:0.8–1.6:1.2–0.4, respectively, at a temperature varying between about 120° C. and about 140° C., concurrently removing the water of esterification as it is evolved; adding a hydrocarbon solvent which forms an azeotropic mixture with water, removing essentially all of the remaining water of esterification formed in the reaction by azeotropic distillation at a liquid temperature varying between about 130° C. and about 150° C.; treating the resulting reaction mixture with a basic nitrogen compound at a temperature varying between about 90° C. and about 100° C.; and separating the thus treated ester product from said hydrocarbon solvent.

7. A process for the production of an ester product of malic acid, which comprises reacting malic acid with 2-ethylhexanol-1, and with oleyl alcohol, in a molar proportion of about 1:1:1, respectively, at a temperature varying between about 130° C. and about 140° C., concurrently removing the water of esterification as it is evolved; adding a hydrocarbon solvent which forms an azeotropic mixture with water, removing essentially all of the remaining water of esterification formed in the reaction by azeotropic distillation at a liquid temperature varying between about 130° C. and about 140° C.; treating the resulting reaction mixture with ammonia at a temperature varying between about 90° C. and about 100° C.; and separating the thus treated ester product from said hydrocarbon solvent.

JOHN F. SOCOLOFSKY.
RALPH V. WHITE.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,344,016	Anderson	Mar. 14, 1944
2,426,496	Farley	Aug. 26, 1947
2,436,272	Snyder	Feb. 17, 1948
2,458,425	Rocchini	Jan. 4, 1949