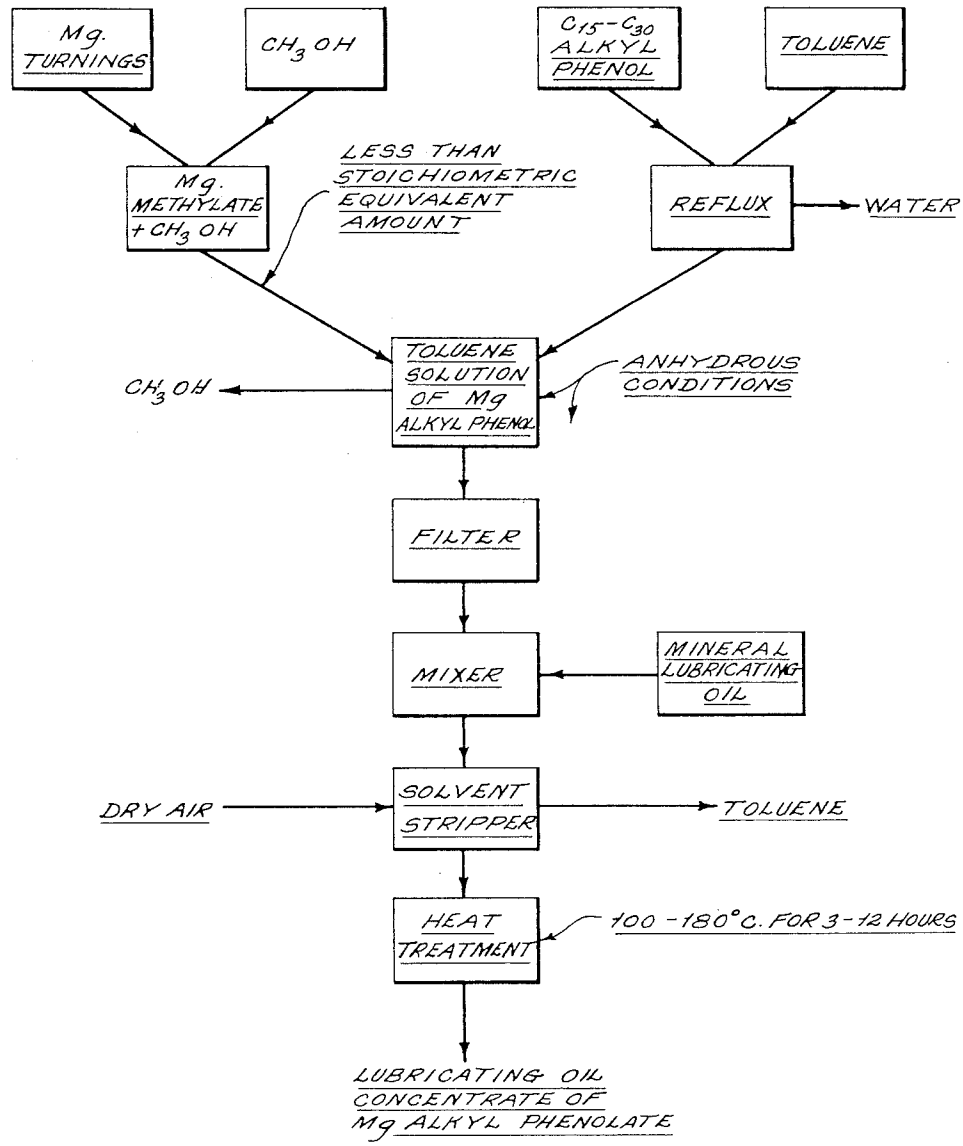
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MANUFACTURE OF MAGNESIUM SALTS OF HIGH MOLECULAR WEIGHT ALKYLATED HYDROXY AROMATIC COMPOUNDS AND THEIR USE IN LUBRICATING OILS

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MANUFACTURE OF MAGNESIUM SALTS OF HIGH MOLECULAR WEIGHT ALKYLATED HYDROXY AROMATIC COMPOUNDS AND THEIR USE IN LUBRICATING OILS

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5 Claims. (Cl. 260—624)

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This invention relates to the manufacture of magnesium salts of high molecular weight alkylated hydroxy aromatic compounds, such as alkyl phenols, wherein the substituent alkyl group on each benzene nucleus contains from 15 to 30 carbon atoms.

The magnesium salts of alkylated hydroxy aromatic compounds, such as alkyl phenols, having one or more high molecular weight alkyl side chains substituted on the ring, are valuable additives for mineral lubricating oil. The magnesium alkyl phenolate of the present invention is particularly useful, when used in conjunction with a zinc alkyl phenolate, as an additive for an airplane engine oil of the highly refined residual type, as disclosed and claimed in the copending application of Frederic C. McCoy, Bill L. Benge, Edwin C. Knowles and Charles C. Towne, Serial Number 143,836, filed of even date herewith.

Polyvalent metal salts of lower molecular weight alkyl phenols or other alkylated hydroxy aromatic hydrocarbons, have generally been prepared by a double decomposition procedure, involving first forming the alkali metal salt of the alkyl phenol, followed by reaction with a soluble polyvalent metal salt. It has been found that this procedure is unsatisfactory in the preparation of the magnesium salts of the present invention, since serious filtration difficulties are encountered, and the quality of the final product is unsatisfactory with respect to solubility and effectiveness as a residual mineral lubricating oil additive.

It has also been proposed to form the polyvalent metal salts of such alkyl phenols by the direct alcoholate procedure, wherein the polyvalent metal alcoholate, such as a methylate or ethylate, is reacted directly with the alkyl phenol. It has been found that this procedure is also unsatisfactory for the synthesis of magnesium salts of the desired solubility and quality for residual lubricating oil additives, when using the high molecular weight C_{15}–C_{30} alkyl phenols, unless certain special and critical processing conditions are employed.

It is accordingly a principal object of the present invention to provide an improved process of synthesizing the magnesium salts of high molecular weight alkylated hydroxy aromatic hydrocarbons, such as alkyl phenols, wherein the substituent alkyl group of the benzene nucleus contains from 15 to 30 carbon atoms, in order to provide products of the required solubility and quality to serve as lubricating oil additives, particularly of the residual lubricating oil type.

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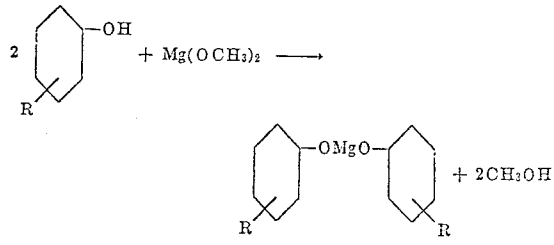
Other objects and advantages of the present invention will be apparent from the following description taken in conjunction with the attached drawing and appended claims.

The drawing is a flow sheet of the method of synthesis of a preferred magnesium alkyl phenolate in accordance with the present invention.

Any suitable alkylated hydroxy aromatic hydrocarbon, wherein the substituent alkyl group on the benzene nucleus contains from 15 to 30 carbon atoms, is a satisfactory starting material for purposes of the present invention. The preferred material from the standpoint of additive manufacture is an alkyl phenol prepared by alkylating a mononuclear monohydroxy aromatic hydrocarbon, such as phenol, cresol or other alkyl phenol, with an olefin polymer, such as a propylene polymer, containing from about 16 to 30 carbon atoms. Also, hydrogenated cardanol producing a C_{15} alkyl phenol is a satisfactory starting material. Likewise, alkylated mononuclear polyhydroxy aromatic hydrocarbons, such as hydroquinone, pyrogallol and other dihydric and trihydric phenols, can be employed. Also, alkylated polynuclear hydroxy aromatic hydrocarbons, such as the naphthols, can be used. From the standpoints of availability and economy, alkyl phenol prepared by alkylating phenol with a C_{18}–C_{30}, and preferably C_{18}–C_{26}, propylene polymer in the presence of anhydrous HF catalyst as disclosed and claimed in the copending application of Louis B. Bos, Serial No. 143,784, filed of even date herewith, or in the presence of $AlCl_2 \cdot HSO_4$ complex catalyst as disclosed and claimed in the copending application of Fred W. Moore and Herman D. Kluge, Serial No. 143,782, filed of even date herewith, is preferred. For the purpose of ease in description, a C_{15}–C_{30} alkyl phenol of the foregoing type will be referred to in the following text; but it is to be understood that this is solely by way of example, and that the description applies to the other alkylated hydroxy aromatic hydrocarbons specified.

The synthesis of the magnesium salt of the C_{15}–C_{30} alkyl phenol in accordance with the present invention involves an improvement on the previously suggested direct alcoholate procedure, wherein the magnesium alcoholate of a C_1–C_5 anhydrous aliphatic alcohol is reacted directly with the alkyl phenol. Preferably magnesium methylate, prepared by reacting magnesium turnings with an excess of anhydrous methyl alcohol, is employed. However, the same procedure applies to the other magnesium alcoholates of the

C_2–C_5 anhydrous aliphatic alcohols. The reaction between the alkyl phenol and magnesium methylate to produce the magnesium salt is represented by the following equation:



In accordance with the present invention, the reaction is carried out under substantially anhydrous conditions, employing somewhat less than the stoichiometric equivalent amount of magnesium methylate or other alcoholate in solution in an excess of the corresponding anhydrous alcohol, than is required to completely neutralize the amount of alkyl phenol present. This has been found to be important in securing a magnesium salt of the proper ash content and of the proper solubility characteristics. The reaction is carried out in the presence of a suitable organic solvent for the alkyl phenol as well as the magnesium alkyl phenolate reaction product, which solvent has a higher boiling point than the anhydrous alcohol as well as water. The reaction takes place under mildly elevated temperature conditions at which the alcohol is distilled off, leaving an organic solvent solution of the magnesium alkyl phenolate which is then filtered while hot. A mineral lubricating oil is then added to the filtrate, the organic solvent is stripped off with the aid of a dry gas to maintain the anhydrous conditions, thereby leaving a mineral lubricating oil concentrate of the magnesium alkyl phenolate. Finally, this mineral oil concentrate is subjected to a controlled heat treatment at about 100–180° C. for at least 3 hours, and up to about 12 hours, preferably with stirring at about 125° C. for 4–8 hours. This has been found to prevent the concentrate from solidifying to a gel upon cooling, and also to improve the solubility of the magnesium salt in a residual mineral lubricating oil. This heat treatment is an important step in obtaining a magnesium alkyl phenolate additive of the desired quality for a residual lubricating oil, such as an airplane engine oil.

Referring to the drawing, the C_{15}–C_{30} alkyl phenol is mixed with about an equal volume of a suitable organic solvent, such as toluene, xylene, or any other organic liquid which is a solvent for both the alkyl phenol and magnesium salt and which boils above the boiling point of water as well as the aliphatic alcohol employed in the process, and yet can be readily stripped from the reaction product at a later stage. As shown, toluene is generally preferred for this purpose. The alkyl phenol solution in toluene is charged to a stirred reactor fitted with a condenser having a water trap; and the reactants are rendered substantially anhydrous by drying at a toluene refluxing temperature, with any separated water collecting in the water trap being removed.

The magnesium alcoholate of a C_1–C_5 anhydrous aliphatic alcohol is prepared in a separate reactor equipped with a condenser. As shown, absolute methyl alcohol is preferred for this purpose. Magnesium turnings are added to the absolute methyl alcohol in the proportion of about one atomic quantity of magnesium per liter of

alcohol; and the latter is heated to refluxing conditions. The clear solution of the resulting magnesium methylate dissolved in excess methyl alcohol is then added dropwise at about 40–50° C. to the first mentioned stirred reactor until slightly less than the stoichiometric equivalent amount of magnesium methylate (½ mol per 1 mol of the alkyl phenol) has been added. Anhydrous conditions are maintained in this reaction and throughout the balance of the process.

Following the addition of the magnesium methylate solution, the temperature is then increased, and the methyl alcohol distilled over and removed by the trap. The resulting toluene solution of magnesium alkyl phenolate is then filtered while hot. Sufficient mineral lubricating oil, such as a refined paraffin base distillate motor oil of about SAE 10 grade, or a refined naphthene base oil having an SUS viscosity at 100° F. of about 300, is added to produce an ultimate concentrate of the magnesium alkyl phenolate in the lubricating oil of about 10–50%, preferably about 25%. The toluene solvent is then stripped from the lubricating oil concentrate by distillation with the aid of a dry gas, such as dry air. The resulting concentrate is then subjected to the controlled heat treatment with stirring at a temperature of about 100–180° C. for in excess of 3 hours and up to about 12 hours, in order to prevent gel formation and maintain the lubricating oil concentrate completely fluid when cooled to room temperature, and also to improve the solubility of the magnesium alkyl phenolate in a residual mineral lubricating oil, such as an airplane engine oil.

The following examples are given to further illustrate the present invention:

EXAMPLE 1

206 grams of alkyl phenol (½ mol), wherein the substituent alkyl group on the benzene nucleus contained an average of 23 carbon atoms, were dissolved in 800 mls. of toluene, and 212 grams of a naphthene base distillate lubricating oil having an SUS viscosity at 100° F. of 315. The resulting mix was refluxed until apparently dry. 15 grams of magnesium turnings were reacted in a separate vessel with 500 mls. of absolute methyl alcohol. A sample of the resultant magnesium methylate solution was withdrawn and titrated with standard HCl, from which the quantity of the magnesium methylate solution was calculated in order to give slightly less than the stoichiometric equivalent amount of magnesium for complete neutralization of the quantity of alkyl phenol present. The stoichiometric equivalent amount required 6.1 grams (¼ mol) of magnesium; and sufficient magnesium methylate solution was added dropwise at 50° C. to the reactor containing the toluene-lubricating oil solution of the alkyl phenol to provide approximately 6.0 grams of magnesium. Ordinarily, only a slight deficiency of the magnesium methylate solution is satisfactory, such as about 0.1–1.0% calculated as magnesium. The reactants were heated to distill off the methyl alcohol, and the hot solution filtered. The toluene was then stripped, and the lubricating oil concentrate allowed to cool to room temperature without the previously described heat treatment. The product solidified to a gel, which analyzed to an ash content of 2.42, check 2.35%, the theoretical ash content being 2.35%. The gel was difficultly soluble in a paraffin base distillate lubricating oil of an SAE 10 grade such that less than 1% by weight of the magnesium

alkyl phenolate could be maintained in solution therein on storage.

In a previous preparation utilizing the same procedure, except that a slight excess (¼ mol + 0.9 gram) of the magnesium methylate solution had been added to the alkyl phenol, the resulting gel which formed on cooling of the final lubricating oil concentrate of the magnesium alkyl phenolate analyzed to an ash content of 2.78%, as compared to a theoretical ash content of 2.35%. This gel with high ash content was substantially insoluble in the distillate paraffin base lubricating oil of SAE 10 grade.

EXAMPLE 2

The gel product from Example 1 (first described procedure) was dissolved in 342 grams of benzene, and an additional 342 grams of the naphthene base distillate lubricating oil sufficient to make an ultimate 25% concentrate were added. The benzene was then stripped, and a yield of 680 grams of resulting concentrate was obtained which remained fluid when cooled to room temperature. The product analyzed to an ash content of 1.22%, with a theoretical ash content of 1.18%, and was readily soluble in the SAE 10 paraffin base lubricating oil to the extent of 1% of the magnesium alkyl phenolate. Apparently the additional heating altered the character of the product to improve its fluidity and solubility. This product was engine tested, but the results were inferior as set forth in greater detail hereinbelow. Apparently the prior addition of the lubricating oil before the completion of the reaction interfered with the attainment of a product of desired quality, probably because of the difficulty of maintaining the reactants anhydrous in the presence of the lubricating oil.

EXAMPLE 3

642 grams of C_{24} alkyl phenol (1½ mols) were dissolved in 2 liters of toluene and refluxed until dry. 30 grams of magnesium turnings were reacted in 1000 mls. of absolute methyl alcohol. The magnesium content was calculated and slightly less than the stoichiometric equivalent amount of the magnesium methylate solution was added dropwise at 40–50° C. The temperature was increased and the methyl alcohol removed by the trap. The material was filtered hot, and 1953 grams of the aforesaid naphthene base distillate lubricating oil were added and the solvent stripped. The heat treatment was not employed. 2530 grams of a lubricating oil concentrate of the magnesium alkyl phenolate were obtained which became quite viscous but remained fluid on cooling to room temperature. The product analyzed to an ash content of 1.19% with a theoretical ash content of 1.15% and was soluble to the extent of 1% of the pure magnesium alkyl phenolate in the SAE 10 paraffin base motor oil. The product was employed in engine testing, but the results were still not satisfactory as discussed more fully hereinbelow.

EXAMPLE 4

The same procedure was employed as in Example 3 above, using the following quantities of reactants:

C_{24} alkyl phenol	1260 grams (3 mols)
Magnesium turnings	50 grams
Methyl alcohol absolute	2000 mls.
Toluene	4 liters
Distillate naphthene base lubricating oil	3879 grams

A yield of 4643 grams of a lubricating oil concentrate of the magnesium alkyl phenolate was obtained which was very viscous when cooled to room temperature. While the product was soluble to the extent of 1% of the magnesium alkyl phenolate in the SAE 10 distillate lubricating oil, it was not soluble to the extent of 1% in a refined paraffin base residual lubricating oil of the airplane engine oil type. This residual lubricating oil was refined by solvent deasphalting, solvent refining, solvent dewaxing, clay contacting and clay filtering. Airplane engine oils of this type generally have a SUS viscosity at 210° F. of about 80–130. The particular oil selected for this test had a SUS viscosity at 210° F. of about 120. This product was also engine tested, but the results were not satisfactory as discussed further hereinbelow.

EXAMPLE 5

Approximately 3,000 grams of the viscous lubricating oil concentrate obtained in Example 4 above were placed in beakers in a steam chest at 125° C. for 4–6 hours. Upon cooling, the product was fluid and soluble to the extent of 1% of the pure magnesium alkyl phenolate in the previously mentioned residual airplane engine oil. The product was engine tested with very satisfactory results as described hereinbelow.

EXAMPLE 6

Based on the foregoing results including the engine testing, the following is given as an example of the standard procedure developed in synthesizing the magnesium alkyl phenolate of the desired solubility and quality, in accordance with the present invention. 416 grams of C_{23} alkyl phenol were dissolved in 2 liters of toluene, and the reactants refluxed until dry. 25 grams of magnesium turnings were reacted in 1,000 mls. of absolute methyl alcohol. The magnesium content was calculated, and slightly less than the theoretical amount of the magnesium methylate solution added dropwise at 40–50° C., followed by distilling off the methyl alcohol. The toluene solution was filtered, and 1280 grams of the naphthene base distillate lubricating oil added. The solvent was then stripped with the aid of dry air. The resulting lubricating oil concentrate was then heat treated at 125° C. for 4–6 hours. 1647 grams of a 25% concentrate of the magnesium alkyl phenolate were obtained, which had substantially theoretical ash content, possessed good solubility properties, and gave excellent engine test results as hereinafter described.

As set forth more fully in the above mentioned copending application of Frederic C. McCoy, Bill L. Benge, Edwin C. Knowles and Charles C. Towne, the magnesium alkyl phenolate produced in accordance with the present invention is employed in conjunction with a second metal alkyl phenolate, namely zinc alkyl phenolate, in an airplane engine oil, to maintain engine cleanliness in high temperature heavy duty service, and also to prevent the formation of objectionable combustion chamber deposits. Final proof of the quality of the magnesium alkyl phenolate preparation was therefore secured by the actual engine testing of the preparation in conjunction with a second metal alkyl phenolate in an airplane engine oil, as set forth in the following Table I. In that table, MP designates the magnesium alkyl phenolate, ZP the corresponding zinc salt and TP the corresponding tin salt. The numeral following the designation for the metallic phenolate

signifies the average carbon atom content of the alkyl side chain on each benzene nucleus of the salt

The CFR high speed engine test of the table is a reliable screening test for determining the ability of a crank case lubricating oil to reduce engine deposits under severe operating conditions. This test is carried out with a standard CFR single cylinder engine operating under the following conditions:

Compression ratio	6.5:1
Speed, R. P. M.	1800
Air/fuel ratio	13:1
Jacket temperature	285° F.
Oil in temperature	185° F.
Oil out temperature	200° F.
Fuel	100 octane (minimum) aviation type containing 4 cc. TEL/gal.
Duration of run	50 hours

In this test, electrical measurements are taken of the piston skirt deposit with respect to the extent or percentage of surface area of the piston skirt which is covered by the lacquer deposit at the termination of each run, together with the average thickness of the deposit expressed in inches $\times 10^{-4}$. Good reproducibility from run to run is obtained in the piston skirt lacquer deposit; and the test is therefore recognized as an accurate indication of the engine cleanliness of the oil composition under test.

The following results were obtained in this CFR high speed engine test, utilizing the magnesium alkyl phenolate preparations as described herein in conjunction with either tin phenolate or zinc phenolate in the proportions indicated in a residual airplane engine oil having an SUS viscosity at 210° F. of about 120 and designated "AEO 120."

Table I

Mg Alkyl Phenolate Procedure	Percent Conc.	Secondary Additive	Percent Conc.	CFR High Speed Engine Test Piston Skirt Deposit	
				Covered, Percent	Thickness in. $\times 10^{-4}$
1. Na Amylate—MP-23	0.5	TP-24	1.0	42	0.19
2. Mg Methylate MP-23 (prior addition of lube oil)	1.0	TP-24	1.0	16	0.02
3. Mg Methylate MP-24 (without heat treatment)	1.0	TP-24	0.5	15	0.12
4. Mg Methylate MP-24 (without heat treatment)	1.0	ZP-24	0.5	12	0.03
5. Mg Methylate MP-24 (without heat treatment)	1.0	ZP-23	0.5	18	0.08
6. Mg Methylate—Product of Run 5 with heat treatment	1.0	ZP-24	0.5	0	0.00
7. Standard—MP-23	1.0	ZP-23	0.5	0	0.00
8. Standard—MP-20	1.0	ZP-20	0.5	0	0.00
9. Standard—MP-21	1.0	ZP-21	0.5	0	0.00
10. Base Oil Alone AEO 120				80	2.0

Run 10 of the foregoing table shows that the base oil alone fails to maintain engine cleanliness, since the piston was 80% covered with a comparatively thick lacquer deposit. On the other hand, a magnesium alkyl phenolate of the proper quality in conjunction with either a zinc alkyl phenolate or a tin alkyl phenolate of the proper quality, when employed in proportion ranges of the order shown in the table, will maintain good engine cleanliness, and give in this engine test a piston substantially without measurable lacquer deposit on the skirt. The zinc and tin phenolate additives employed in the runs of this table were known to be of good quality; so the test results are an accurate indication of the quality of the magnesium phenolate products.

Run 1 of the table employed a magnesium C_{23} alkyl phenolate produced by a double decomposition reaction, wherein sodium amylate was first reacted with the alkyl phenol, and then the resulting sodium alkyl phenolate reacted with magnesium chloride. Other attempts to prepare the magnesium salt by this double decomposition reaction, including the use of sodium butylate and also magnesium acetate in the reaction, had resulted in failure due to filtration difficulties. This sodium amylate procedure resulted in a magnesium alkyl phenolate of fairly satisfactory ash content and solubility so that it could be employed for engine testing; but the engine test showed the product to be of unsatisfactory quality as a detergent additive for crank case lubricating oil, since it failed to maintain the desired engine cleanliness.

Run 2 of the table sets forth the results secured in the engine testing of the magnesium C_{23} alkyl phenolate produced by the methylate procedure of Example 2 above, wherein the mineral lubricating oil was added prior to the reaction. Even though this product was prepared by using slightly less than the stoichiometric equivalent amount of magnesium methylate solution, and was in effect heat treated by the second addition of solvent and lubricating oil to the gel product of Example 1, nevertheless this preparation also failed to exhibit the desired engine cleanliness properties.

Run 3 of the table employed the magnesium C_{24} alkyl phenolate product produced by Example 3, involving the so-called standard methylate procedure except omitting the final heat treating step. Run 4 likewise employed a product made by this same procedure. Run 5 employed the product of Example 4 above. In each case, the product is seen to be of inferior quality with respect to maintaining the desired engine cleanliness.

Run 6 employed the heat treated product of Example 5, and the engine test showed this product to be of good quality as evidenced by the lack of measurable piston skirt deposit. Run 7 employed the product of Example 6; and Runs 8 and 9 employed other preparations of magnesium alkyl phenolate produced by the so-called standard procedure of Example 6. In each case, the product was of the desired high quality as shown by the satisfactory engine test.

The characteristics of the magnesium alkyl phenolates employed in Runs 2–9 inclusive of

Table I, with respect to ash and magnesium content, and storage data on solubility, are set forth in the following Table II:

Table II

Mg Alkyl Phenolate Preparation	Ash Weight Percent	Ash Percent Theory	Mg Weight Percent	Mg Percent Theory	Storage Data
Run 2 of Table I	1.22	1.18			1% soluble in SAE 10 oil.
Run 3 of Table I	1.19	1.15			Do.
Run 4 of Table I	1.18	1.12	0.72	0.68	Do.
Run 5 of Table I	1.25	1.12	0.76	0.68	1% soluble in SAE 10 oil but not completely soluble in AEO 120.
Run 6 of Table I					1% in AEO 120 clear after 3 months.
Run 7 of Table I	1.23	1.18	0.72	0.71	Do.
Run 8 of Table I	1.32	1.28	0.79	0.78	Do.
Run 9 of Table I	1.28	1.24	0.76	0.75	Do.

In each case, the ash and magnesium contents of the products analyzed close to theoretical. This is to be compared with the product of Example 1 above using an equivalent or excess amount of magnesium methylate. In the latter case, the ash content is quite high; and products made by this procedure were uniformly unsatisfactory with respect to solubility and gelling properties. It is postulated that, where the alkyl phenol is not present in the reacting mix in excess of the stoichiometric equivalent amount of magnesium methylate, some complex other than the desired magnesium alkyl phenolate, such as a complex containing some methylate groups attached to some magnesium monophenolate, forms in the reaction. The presence of such complex in the product would of course increase the ash content above the theoretical for magnesium alkyl phenolate, and apparently contributes to the insolubility and other undesirable properties of the resulting product.

While the ash and magnesium contents of the products of Runs 3–5 inclusive of Table I are fairly close to theoretical, it is noted from Table II that the solubility of these products which were not heat treated is not satisfactory in the residual type of mineral lubricating oil. The products prepared by this procedure omitting the heat treating step were mainly gels of unsatisfactory solubility in the residual lubricating oil so that they could not even be employed for engine testing. And the few products of this character which could be employed for engine testing uniformly gave inferior results with respect to engine cleanliness. It is postulated that some polymerization reaction tends to take place in the reaction forming product which is responsible for the gel formation or substantial viscosity increase, as well as the poor solubility and engine detergent characteristics; and that the heat treatment breaks down this polymer and converts the reaction product to the desired magnesium salt.

On the other hand, the products made by the standard procedure including the heat treatment all have proper ash and magnesium contents, and have excellent solubility in the residual oil as evidenced by the fact that 1% of the magnesium alkyl phenolate in the airplane engine oil of grade 120 remained as a clear solution after storage of three months. The synthesis procedure of the present invention has been found to uniformly give satisfactory products of high quality without difficulty.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof, and, therefore, only such limitations should be imposed as are indicated in the appended claims.

I claim:

1. In the manufacture of a magnesium salt of an alkyl substituted hydroxy aromatic hydrocarbon consisting of carbon, hydrogen and oxygen, wherein the alkyl substituent on the ring contains from 15 to 30 carbon atoms, the method which comprises reacting under substantially anhydrous conditions the alkyl substituted hydroxy aromatic hydrocarbon consisting of carbon, hydrogen and oxygen in solution in an organic solvent with a magnesium alcoholate solution consisting of magnesium alcoholate dissolved in an excess of the corresponding anhydrous alcohol selected from the group consisting of the lower molecular weight aliphatic alcohols containing from 1 to 5 carbon atoms in the molecule, said organic solvent having a higher boiling point than that of water as well as said alcohol, heating said reaction mix at mildly elevated temperatures at which the aliphatic alcohol is distilled off but below the boiling point of said organic solvent, to obtain a solution of the magnesium salt of the alkyl substituted hydroxy aromatic hydrocarbon in the said organic solvent, the quantity of magnesium alcoholate being less than the calculated stoichiometric amount required for neutralization of the quantity of alkyl substituted hydroxy aromatic hydrocarbon present.

2. The method according to claim 1, wherein the alkyl substituted hydroxy aromatic hydrocarbon is a C_{18}–C_{26} alkyl phenol.

3. The method according to claim 1, wherein the organic solvent solution is filtered, mineral lubricating oil is added, and the organic solvent removed by stripping with dry gas to recover a concentrate of the magnesium alkyl substituted hydroxy aromatic hydrocarbon in the mineral lubricating oil.

4. In the manufacture of a magnesium salt of a C_{15}–C_{30} alkyl substituted hydroxy aromatic hydrocarbon consisting of carbon, hydrogen and oxygen, the method which comprises reacting a C_{15}–C_{30} alkyl substituted hydroxy aromatic hydrocarbon consisting of carbon, hydrogen and oxygen in solution in an organic solvent of higher boiling point than water with slightly less than a stoichiometric equivalent quantity of magnesium methylate dissolved in anhydrous methyl alcohol at a mildly elevated temperature at which the methyl alcohol is distilled off but below the boiling point of said organic solvent, thereby leaving the magnesium salt of the C_{15}–C_{30} alkyl hydroxy aromatic hydrocarbon in solution in the organic solvent, filtering the last mentioned solution, adding mineral lubricating oil, removing the solvent to obtain a mineral oil concentrate of

the said magnesium salt, and heat treating the said concentrate at a temperature of about 100–180° C. for a period of at least 3 hours until the concentrate remains fluid upon cooling to room temperature and becomes soluble in a refined residual mineral lubricating oil to the extent of dissolving at least 1% by weight of the magnesium salt therein.

5. The method in the manufacture of magnesium C_{18}–C_{26} alkyl phenol, which comprises reacting under substantially anhydrous conditions a C_{18}–C_{26} alkyl phenol in solution in toluene with slightly less than the stoichiometric equivalent amount of magnesium methylate in solution in anhydrous methyl alcohol at a mildly elevated temperature above the boiling point of methyl alcohol to distill off the alcohol, but below the boiling point of toluene to obtain a toluene solution of the magnesium C_{18}–C_{26} alkyl phenol, filtering the last mentioned solution while hot,

adding a distillate mineral lubricating oil, removing the toluene by stripping with the aid of dry air to obtain a lubricating oil concentrate of the said magnesium salt, and heat treating the said concentrate with stirring at a temperature of about 100–180° C. for about 4–8 hours to prevent gel formation of the concentrate upon cooling and to improve the solubility of the magnesium salt in a residual mineral lubricating oil.

JOHN W. HUTCHESON.

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