

- [54] **PREPARATION OF NEUTRAL AND HIGHLY BASIC ALKYLPHENATES AND SULFURIZED ALKYLPHENATES**
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- [58] Field of Search .....**252/18, 33.4, 42.7; 260/624, 260/608, 609 F**

[56] **References Cited**

**UNITED STATES PATENTS**

2,788,325	4/1957	Meyers et al. ....	252/42.7
2,895,913	7/1959	Carlyle et al. ....	252/42.7 X
2,916,454	12/1959	Bradley et al. ....	252/42.7

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[57] **ABSTRACT**

A process for preparing neutral and highly basic magnesium alkylphenates or sulfurized alkylphenates wherein the process comprises:

- a. forming an admixture of alkylphenol or sulfurized alkylphenol, nonvolatile diluent oil, and a volatile process solvent;
- b. adding a stoichiometric neutralizing amount of a carbonated magnesium alcoholate at a temperature of from about 30°C to about 60°C;
- c. heating the mixture to a reflux temperature of from about 65°C to about 90°C, and maintaining the mixture at this reflux temperature for a period of from about 20 minutes to about 1½ hours; then
- d. removing the volatile materials by heating.

If a highly basic (overbased) product is to be prepared, the same steps are followed as in the production of the neutral phenate, except that after neutralization, and prior to removing the volatile materials from the mixture, carbonated magnesium alcoholate and water are added concurrently to the mixture.

**29 Claims, No Drawings**

**PREPARATION OF NEUTRAL AND HIGHLY BASIC ALKYLPHENATES AND SULFURIZED ALKYLPHENATES**

**BACKGROUND OF THE INVENTION**

**1. Field of the Invention**

This invention relates to the preparation of neutral and overbased alkaline earth metal alkylphenates and sulfurized alkylphenates, and more particularly, to the preparation of low viscosity magnesium alkylphenate compositions which may be neutral, or may be overbased with dispersed magnesium carbonate.

**2. Brief Description of the Prior Art**

The utility of alkaline earth metal alkylphenates and sulfurized alkylphenates as lubricating oil additives has long been known. The presence of these materials in the lubricating oils improves the detergency characteristics, reduces engine wear, minimizes the formation of harmful deposits on engine parts, and improves the resistance of the oil to oxidation. It is further known that the described phenate salts can be advantageously overbased by the dispersion in the neutral phenate compositions of basic alkaline earth metal compounds. The highly basic compositions as thus formulated function effectively in neutralizing harmful acidic compounds formed during the combustion of fuels used in association with the lubricating oils to which the compositions are added.

U.S. Pat. No. 2,788,325 discloses the preparation of neutral magnesium phenates by the reaction under substantially anhydrous conditions of magnesium alkoxo alkoxides with an alkylphenol, with the alkoxides being used in substantially less than the stoichiometric amount required for neutralization of the alkylphenol.

In our co-pending U.S. application Ser. No. 148,262 filed May 17, 1971, we disclose a process for preparing highly basic alkaline earth metal phenates and sulfurized alkaline earth metal phenates by first adding, at a relatively low temperature, a carbonated magnesium alcoholate derived from a glycol ether containing from one to eight carbon atoms, to a mixture which comprises water and an alkylphenol or sulfurized alkylphenol. In this first step, the amount of the magnesium alcoholate used is that which is to be employed in excess of the amount required for neutralization of the phenol, and which is thus the amount to be used in overbasing the product. The water employed in admixture with the alkylphenol to be neutralized is from about 1 to about 2.5 moles of water per mole of dispersed basic alkaline earth metal compound which is to be incorporated in the product. Following the addition of the overbasing amount of magnesium alcoholate to the admixture of water and alkylphenol or sulfurized alkylphenol, an additional amount of magnesium alcoholate which is stoichiometrically required for neutralization of the phenol is added to the reaction mixture. This addition is carried out at a relatively higher temperature than that employed during the initial addition of magnesium alcoholate. Finally, the volatile materials are removed from the reaction mixture by heating.

**BRIEF SUMMARY OF THE PRESENT INVENTION**

Broadly stated, the present invention concerns a process for preparing highly basic magnesium alkylphenates or sulfurized alkylphenates, wherein the process comprises the steps of:

- a. forming an admixture of alkylphenol or sulfurized alkylphenol, nonvolatile diluent oil, and volatile process solvent;
- b. adding to the admixture, at a temperature of from about 30°C to about 60°C, an amount of a carbonated magnesium alcoholate which is stoichiometrically sufficient to neutralize said alkylphenol;
- c. heating the mixture to a temperature of from about 65°C to about 90°C, and maintaining this temperature for a period of from 20 minutes to 1½ hour with stirring; then
- d. removing the volatile materials by heating and stripping with an inert gas.

The foregoing procedure is used in the preparation of oil soluble neutral magnesium alkylphenates useful as lubricating oil additives. The process of the present invention also contemplates the overbasing of the described neutral phenate compositions by following the neutralization step [step (c) described above] with the gradual concurrent addition to the neutral phenate compositions of an overbasing amount of carbonated magnesium alcoholate and water in a ratio of from about 1.1 to about 4 moles of water per mole of the carbonated magnesium alcoholate. This addition is carried out at a temperature of from about 55°C to about 90°C. After this addition is completed, volatile solvents are removed as described in step (d) above.

We have determined that by using carbonated magnesium alcoholate, a significant improvement in the viscosity of both the neutral and overbased products is realized as compared to the viscosity of products prepared using uncarbonated magnesium alcoholate. Moreover, the carbonated magnesium alcoholates react more rapidly with phenols than uncarbonated magnesium alcoholates, and thus process time requirements are reduced.

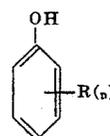
An object of the present invention is to provide an improved process by which neutral and overbased magnesium phenate compositions useful as lubricating oil additives may be prepared.

Another object of the invention is to provide a process for producing neutral and overbased magnesium phenate compositions having low viscosity facilitating ease of blending in lubricating oil formulations.

Additional objects and advantages of the invention will become apparent as the following detailed description of preferred embodiments of the invention is considered.

**DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS OF THE INVENTION**

Suitable alkylphenols for use in the process of the present invention may be represented by the structural formula:

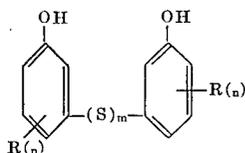


Wherein R is a straight chain or branched chain, saturated or unsaturated, aliphatic hydrocarbon radical having from four to 30 carbon atoms, preferably from nine to 15 carbon atoms, and n is an integer having a

value of 1 or 2. The total number of carbon atoms for the alkyl groups has a minimum value of eight and a maximum value of 40. Thus, when  $n$  is 1, the minimum number of carbon atoms in  $R$  is eight.

Examples of suitable hydrocarbon radicals include alkyl radicals such as butyl, hexyl, octyl, nonyl, decyl, dodecyl, hexadecyl, eicosyl, hexacosyl and triacontyl; radicals derived from petroleum hydrocarbons such as white oil, wax and olefin polymers (e.g., polypropylene and polybutylene).

Suitable sulfurized alkylphenols include those materials prepared by sulfurizing alkylphenols using any of the known methods. For example, it is known that sulfur monochloride can be used to prepare sulfurized alkylphenols. Such a method is described in U.S. Pat. No. 2,409,687. The sulfurized alkylphenols which are suitable in our process can be illustrated by materials represented by the following formula:



wherein  $m$  is a number in range of from 1 to 3, more usually in the range of 1 to 2, and  $R$  and  $n$  are as described in the preceding description of the alkylphenols. It is to be understood that the preceding description is typical of the sulfurized alkylphenols which can be used, and that our process is not limited to materials having the formula shown in the foregoing.

Regardless of the manner in which they are prepared, the sulfurized alkylphenols which are useful in our process suitably contain from about 2 to about 14 percent by weight sulfur. Preferably, the amount of sulfur is from about 4 to about 12 percent by weight. It may be here pointed out that in the preparation of overbased dispersions (as contrasted with neutral dispersions), the utilization of the sulfurized alkylphenols is preferred to use of the unsulfurized alkylphenols.

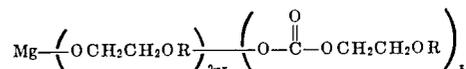
A number of nonvolatile diluent oils are suitable in the process of our invention since the principle requisites for these materials are that they function as a solvent for the alkylphenate or sulfurized alkylphenate, and serve to reduce the viscosity of the final product mixture. Often a small amount of the nonvolatile diluent oil is present in conjunction with the alkylphenol or sulfurized alkylphenol used in the process. The nonvolatile diluent oils have a boiling point in excess of about 160°C.

Examples of suitable nonvolatile diluent oils which can be used include mineral lubricating oils obtained by conventional refining procedures, synthetic lubricating oils such as polymers of propylene, polyox-yalkalenes, polyoxypropylene; dicarboxylic acid esters, and esters of acids of phosphorus, synthetic hydrocarbon lubricating oils, such as di- $n$ -alkylbenzenes and oligomers of  $C_8$ - $C_{14}$   $\alpha$ -olefins; vegetable oils, such as corn oil, cottonseed oil, and castor oil; animal oils, such as lard oil and sperm oil. Mixtures of these materials can also be employed as the nonvolatile diluent.

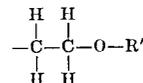
Of the preceding examples of nonvolatile diluent oils, the mineral lubricating oils and the synthetic lubricating oils are considered more suitable, with the mineral lubricating oils being preferred.

The process solvents which are useful in the process of the present invention have a boiling point below about 150°C. Examples of suitable volatile process solvents include aromatic hydrocarbons, such as benzene, toluene and xylene; aliphatic hydrocarbons, such as hexane and heptane; petroleum naphtha, glycol ethers, as hereinafter defined, and primary aliphatic  $C_1$ - $C_6$  alcohols.

The carbonated magnesium alcoholates used in the process of the invention are derived from monoethers of ethylene glycol and diethylene glycol containing up to 8 carbon atoms. The monoethers of ethylene glycol are also known as alkoxy alkanols, and the carbonated magnesium alcoholate complexes thereof are sometimes referred to as magnesium alkoxide-carbonate complexes. The carbonated magnesium alcoholates derived from the monoethers of ethylene glycol have the generic formula:



where  $R$  is either a  $C_1$  to  $C_6$  alkyl group or an organic radical of the formula



where  $R'$  is a  $C_1$  to  $C_4$  alkyl group, and  $x$  is from 0.5 to 1.5, preferably 0.75 to 1.0. The preparation of these complexes is described in detail in U.S. Pat. No. 3,150,089. Preferably, the carbonated magnesium alcoholates are derived from the monoethyl and monomethyl ethers of ethylene glycol. These latter materials are available commercially under the trademarks Cellosolve and methyl Cellosolve, respectively. The complexes employed contain from about 0.5 mole to about 2.0 mole of  $\text{CO}_2$  per mole of metal alcoholate, and preferably from about 0.8 mole to about 1.2 mole per mole of the alcoholate. The magnesium content of the preferred complexes ranges from 3 weight percent to about 10 weight percent. In the practice of the invention, the carbonated magnesium alcoholates are added to the mixture containing the phenol compounds to be neutralized in the form of a solution of the alcoholate in glycol ether. Solutions containing from about 30 weight percent to about 80 weight percent of the alcoholate are preferably utilized.

The water required during the hydrolysis of the overbasing step of the process can be water per se or an azeotrope mixture of water and glycol ether.

Although it is not essential to the practice of the present invention, we have determined that for some usages in which low sedimentation upon dilution of overbased products of the invention is desired, the product is improved by the addition of small amounts of oil-soluble sulfonic acid to the starting material. The amount of sulfonic acid employed in such cases is from about 0.1 to about 50 parts per 100 parts of the al-

alkylphenol or sulfurized alkylphenol starting material. The acid is preferably used in a hexane solution.

Particularly suitable oil-soluble sulfonic acids are those prepared from various synthetic hydrocarbon sulfonation feedstocks. These materials are usually mono- or di-alkylbenzenes and generally have molecular weights of from about 300 to about 1,000. The nature and derivation of the suitable sulfonic acids are further detailed in Nield U.S. Pat. No. 3,525,599 which is incorporated herein by reference.

The amounts of the various materials used in our process, both suitable and preferred ranges, are shown below in parts by weight. Since the invention contemplates the production of both the neutral and overbased phenate compositions, the amounts of materials used in each procedure are set forth. In general, the weight ratio of carbonated magnesium alcoholate used for overbasing to that used for neutralization ranges from 0.5 to 3.0.

#### NEUTRAL PHENATE COMPOSITIONS

	Suitable	Preferred
Alkylphenol or Sulfurized Alkylphenol	5-100	10-70
Volatile Process Solvent	5-120	10-100
Nonvolatile Diluent Oil	2-100	10-90
Water	0.00-1.8	0.00-0.05
Magnesium Alkoxide-Carbonate Complex	2-160	4-100

#### OVERBASED PHENATE COMPOSITIONS

	Suitable	Preferred
Alkylphenol (or sulfurized)	5-100	10-60
Volatile Process Solvent	5-120	10-100
Nonvolatile Diluent Oil	2-100	20-90
Water	0.1-10	0.2-7.5
Magnesium Alkoxide-Carbonate Complex	10-300	20-200

#### PROCESS CONDITIONS

In carrying out the process of the invention, an admixture is initially formed which contains the alkylphenol or sulfurized alkylphenol to be neutralized, nonvolatile diluent oil and a volatile process solvent. Moreover, the admixture may contain a small amount of water which is less than 1 mole of water per mole of the magnesium alkoxide-carbonate complex utilized. Within the broad description of the types of alkylphenols which may be used in the process of the present invention as hereinbefore described, we prefer to utilize mixtures of monoalkyl and dialkylphenols, either sulfurized or unsulfurized, since we have observed that the neutral phenates yielded therefrom exhibit lower viscosity and improved oil solubility with respect to the phenates derived from the monoalkylphenol starting material. Preferably the mixture contains at least 20 weight percent dialkylphenol.

The admixture is stirred and heated to a temperature of from about 30°C to about 60°C. Preferably, the temperature of the admixture is brought within the range of 50°C to 60°C. While the admixture is at this temperature, the stoichiometric amount of magnesium alkoxide-carbonate complex is added thereto in a gradual manner. Although the time period over which the complex is added is not critical, it is desirable that the addition be carried out in a time period of from about 5 minutes to about 120 minutes. Preferably, the addition is carried out in a period of between about 20 minutes

and about 60 minutes. The addition of the complex is made in such a way that the complex is introduced to the admixture beneath the surface thereof, since generally better results are obtained.

The amount of the complex added is, as has been previously explained, an amount which is stoichiometrically sufficient to neutralize the alkylphenol or sulfurized alkylphenol present in the admixture. The use of a stoichiometric quantity of the complex is to be contrasted with the procedure described in U. S. Pat. No. 2,788,325, and is believed to be made possible in the process of the present invention as a result of the enhanced reactivity of the carbonated alcoholate as contrasted with the noncarbonated alcoholate.

Upon completion of the addition of the complex, the reaction mixture is heated to reflux temperature, which is generally between about 65°C to about 90°C, and is maintained at this temperature for a period of from about 20 minutes to about 1½ hours with concurrent stirring. This step is important to the procurement of complete reactivity of the stoichiometric amount of the magnesium alkoxide-carbonate complex with the alkylphenol.

If an overbased product is to be produced, rather than a neutral composition, an overbasing amount of magnesium alkoxide-carbonate complex is introduced at this point in the process, along with water or a water-alcohol azeotrope. The amount of the magnesium alkoxide-carbonate complex used for the purpose is varied according to the specific complex used and to the extent of overbasing desired, but in general is from about 15 weight percent to about 40 weight percent based on the total weight of the admixture to which it is added. An amount of water is added concurrently with the complex in an amount which is a stoichiometric excess of that required to hydrolyze the complex to the alkoxy alcohol and magnesium carbonate. In general, from about 1.1 moles to about 4 moles of water is utilized per mole of the alkoxide complex. The concurrent addition is carried out slowly with the water preferably being added in the form of an azeotrope with the alkoxy alcohol from which the complex is derived.

Either after completion of the overbasing step by concurrent addition of complex and water, or, where a neutral product is the objective, after completion of period of heating at reflux, the volatile components of the mixture are removed by distillation. In general, the distillation is continued until a bottoms temperature reaches about 140°C to about 160°C, and preferably about 150°C. It should be here pointed out that nonvolatile diluent oil may be added to the reaction mixture at any time prior to this point in the process, provided the total amount present does not exceed the 100 weight parts previously referred to.

Upon the attainment of the described bottoms temperature, this temperature is maintained and the reaction mixture is then subjected to stripping of residual volatile materials therefrom by bubbling an inert gas, and preferably, carbon dioxide, through the mixture for a period of from about 30 minutes to 1½ hours.

If it should be desired to adjust the activity (i.e., concentration of alkylphenate and dispersoid material) in the final product, an additional amount of a nonvolatile diluent oil can be added to the product at this time.

For the purpose of disclosing more completely the manner in which the present invention is practiced, the following examples are given. The specific conditions and details set forth in these examples are not considered to be limiting relative to the scope of the invention, except insofar as such limitations and details are specified in the appended claims.

#### EXAMPLE 1

This example illustrates the preparation of a neutral magnesium alkylphenate solution using the process of the present invention.

Materials Used	Parts by Weight
Alkylphenol*	117.8
n-Hexane	100.0
Nonvolatile Diluent Oil**	176.5
Methoxy Ethanol Solution of Magnesium Ethoxy Ethoxide-Carbonate Complex***	79.8

\*This alkylphenol contained about 97 weight percent mono-p-nonylphenol having a molecular weight of 220.3 and containing 3 weight percent free oil. The nonyl side chain was branched.

\*\*This oil was a commercially available, nonvolatile naphthenic oil.

\*\*\*This material contained 7.9 weight percent magnesium and 14.3 weight percent carbon dioxide.

#### PROCESS

The alkylphenol, oil and hexane were charged to a 1-liter, 3-neck flask equipped with mechanical stirrer, thermometer and condenser. The mixture was stirred and heated to 50°C. The magnesium ethoxy ethoxide-carbonate complex was then added slowly over a period of 30 minutes by means of a tube immersed in the reaction mixture. Upon completion of the addition of the complex, the admixture was then heated to reflux temperature of about 72°C, and was refluxed and stirred for 1 hour. The volatile solvents, including water of neutralization, methoxy ethanol and hexane were then removed by distilling the mixture to a bottoms temperature of 150°C. The mixture was stripped with CO<sub>2</sub> for a period of 1 hour at about 150°C.

300 grams of neutral magnesium alkylphenate composition was produced which was calculated to contain 40 weight percent of magnesium mono-p-nonylphenate. The product had a B.S.&W. value of 0.015 volume percent, an acetic base number of 89 and a viscosity in centistokes at 210°F of 93.4.

#### EXAMPLE 2

This example illustrates the preparation of a neutral magnesium alkylphenate composition utilizing an uncarbonated magnesium alcoholate for neutralization.

In this example, the same alkylphenol was employed as that which was used in Example 1, and this material was mixed with the same nonvolatile naphthenic oil used in Example 1 in amounts equivalent to those there used. The admixture also contained 100 grams of n-hexane. To the admixture as thus constituted were added 68.5 grams of magnesium ethoxy ethoxide (magnesium methyl Cellosolve) which contained 9.2 weight percent magnesium and 0.4 weight percent carbon dioxide. The addition was carried out as described in Example 1, and was followed by heating the mixture to reflux temperature and retaining it at this temperature for a period of 1 hour with concurrent stirring. After this, the volatile materials were removed from the mix-

ture by elevating the temperature to 150°C. The mixture was then stripped with nitrogen gas for a period of 1 hour at about 150°C.

310.1 grams of neutral magnesium alkylphenate composition were produced, and were calculated to contain 40 weight percent magnesium mono-p-nonylphenate. The product had a B.S.&W. value of 0.002 volume percent, an acetic base number of 90 and a viscosity in centistokes at 210°F of 3513.2. It may thus be readily perceived that when the uncarbonated magnesium alcoholate is employed for neutralization, the product obtained has a viscosity which is dramatically higher than the viscosity of the product obtained by the practice of the present invention, using the carbonated magnesium alcoholate material for neutralization.

#### EXAMPLE 3

This example describes two runs carried out for comparative purposes, utilizing in each case, a commercially available alkylphenol composition which contained 7 weight percent free oil, with the balance being active alkylphenols. The alkylphenols in the composition comprised 65 weight percent mono-p-nonylphenol and 35 weight percent dinonylphenol. The average molecular weight of the phenol materials in the composition was 250. In each of the comparative runs, 123.5 grams of the alkylphenol composition was employed in admixture with 171.4 grams of nonvolatile naphthenic oil and 100 grams of n-hexane. One of the runs was carried out using a magnesium alkoxide-carbonate complex which contained 7.9 weight percent magnesium and 14.3 weight percent carbon dioxide. In the other run, 60.7 grams of uncarbonated magnesium alcoholate was utilized. In the two runs, the procedures described in Examples 1 and 2 were utilized, employing carbon dioxide for stripping residual volatile materials from the run carried out using the carbonated complex, and using nitrogen gas for stripping volatile materials from the run carried out using uncarbonated magnesium alcoholate for neutralization.

In the run in which the carbonated complex was used for neutralization, 307 grams of product were yielded having a B.S.&W. value of 0.03 volume percent, an acetic base number of 82 and a viscosity at 210°F of 82 centistokes. In the run carried out using the uncarbonated neutralizing material, 313.2 grams of product were produced, having a B.S.&W. value of 0.04 volume percent, an acetic base number of 83 and a viscosity at 210°F of 398 centistokes. The much lower viscosity of the product produced using the carbonated magnesium alkoxy alkoxide complex for neutralization will be readily apparent. Moreover, comparison of the results obtained in the two runs described in this example, in which a mixture of the mono- and di-nonylphenols were used as a starting material, with the results obtained in Examples 1 and 2 will reveal the improvement in viscosity obtained as a result of the use of the mixed alkylphenol starting material.

#### EXAMPLE 4

A commercially available alkylphenol composition which contained 95 weight percent mono-p-dodecylphenol having a molecular weight of 262.4 and 5 weight percent of free oil was subjected to two neutralization runs in one of which, the carbonated

magnesium methoxy ethoxide complex described in Example 1 was used for neutralization, and in the other of which an uncarbonated magnesium methoxy ethoxide of the type described in Example 2 was utilized for neutralization. 121.2 grams of the alkylphenol composition was employed in admixture of 173.9 grams of nonvolatile naphthenic oil and 100 grams of hexane in each of the runs. In the run utilizing the magnesium methoxy ethoxide-carbonate complex, 67.5 grams of the complex was used for neutralization. In the run utilizing the uncarbonated magnesium alcoholate for neutralization, 58.0 grams of the alcoholate were used for neutralization. The process conditions were those described in the preceding examples, with nitrogen being used for the stripping of the neutral phenate prepared using the uncarbonated neutralizing material, and carbon dioxide being utilized to strip the neutral phenate prepared by the use of the carbonated complex.

In the run carried out using the carbonated complex, 307 grams of product were yielded having an acetic base number of 75 and a viscosity at 210°F of 1795.0 centistokes. 306.3 grams of product were prepared in the run utilizing the uncarbonated magnesium alcoholate for neutralization, and this product had an acetic base number of 77 and a viscosity at 210°F exceeding 5,000 centistokes.

#### EXAMPLE 5

Two runs were carried out utilizing the procedure described in the preceding examples and each employing 143.7 grams of a commercially available alkylphenol composition which contained 3 weight percent free oil, 27 weight percent di-nonylphenol and 70 weight percent mono-p-nonylphenol. In one of the described runs, the alkylphenol composition described was mixed with 100 grams of hexane and 143.8 grams of 150 SSU naphthenic oil. In the other run, the alkylphenol composition was admixed with 100 grams of hexane and 147.6 grams of 150 SSU naphthenic oil.

In the first run, 93.3 grams of magnesium alkoxide-carbonate complex were added to the mixture for neutralization purposes. This complex contained 7.49 weight percent magnesium and 14.0 weight percent carbon dioxide. Upon completion of stripping with carbon dioxide, 300 grams of neutral phenate product were yielded which contained 50 weight percent of the magnesium alkylphenate per se. The product composition had a viscosity at 210°F of 17.5 centistokes.

In the other run (in which the starting composition contained 147.6 grams of nonvolatile naphthenic oil), 76 grams of an uncarbonated magnesium alcoholate containing 9.20 percent magnesium and 0.30 weight percent carbon dioxide were utilized for neutralization. The 300 grams of product composition yielded following nitrogen stripping contained 50 weight percent magnesium alkylphenate, and had a viscosity exceeding 500 centistokes.

#### EXAMPLE 6

This example illustrates the preparation of magnesium alkylphenate compositions derived from sulfurized phenols. Two comparative runs were carried out utilizing, in each case, a commercially available, sulfurized alkylphenol composition which contained about 48.5

weight percent sulfur bridged mono-p-nonylphenol, 18.5 weight percent sulfur bridged dinonylphenol and 33 weight percent free nonvolatile oil. In each of the two runs carried out for comparison purposes, 221.6 grams of the described sulfurized phenol composition were mixed with 100 grams of hexane. In the run in which a carbonated magnesium alcoholate was used for neutralization, the starting mixture further contained 85 grams of 150 SSU naphthenic oil. In the run subjected to neutralization with an uncarbonated magnesium alcoholate of the type described in Example 5 (containing 9.20 weight percent magnesium), the starting mixture contained 75.8 grams of 150 SSU naphthenic oil. For neutralization, in the case of the first run, 88.3 grams of magnesium alkoxide-carbonate complex containing 7.49 weight percent magnesium and 14.0 weight percent carbon dioxide were employed for neutralization. In the second run, 71.8 grams of uncarbonated magnesium methoxy ethoxide containing 9.20 weight percent magnesium were utilized.

In each of the runs, 300 grams of product were yielded. In the run in which the magnesium alkoxide-carbonate complex was employed, the product had a viscosity at 210°F of 36 centistokes. In the second run using the uncarbonated magnesium alcoholate, the product had a viscosity at 210°F of 111.1 centistokes.

#### EXAMPLE 7

This example illustrates the employment of the present invention in the production of an overbased magnesium alkylphenate dispersion, using a small amount of an oil-soluble sulfonic acid in the starting material.

Materials Used	Parts
Alkylphenol*	145.3
Nonvolatile Naphthenic Diluent Oil	127.0
n-Hexane	147.1
Water**	8.4
Hexane Solution of Sulfonic Acid***	15.4
Methoxy Ethanol Solution of Magnesium Ethoxy-Ethoxide Carbonate Complex****	175.5

\*Same as alkylphenol used in Example 3

\*\*Added as an azeotrope with an equal amount of methoxy ethanol (methyl Cellosolve)

\*\*\*The hexane solution contained 29 weight percent of a sulfonic acid composition derived from alkylbenzenes and having a combining weight of 488. The sulfonic acid acidity of the solution was 0.587 meq./g., and the acid contained 10.5 weight percent nonvolatile mineral oil.

\*\*\*\*This material contained 7.49 weight percent magnesium and 13.6 weight percent carbon dioxide.

#### PROCESS

To a 1-liter, 3-neck flask were added the alkylphenol, n-hexane, and hexane solution of sulfonic acid. The mixture was heated to a temperature of 35°C, and while the temperature was maintained between about 30°C and about 40°C, 95.5 parts by weight (54.4 percent) of the methoxy ethanol solution of the complex was added over a period of 15 minutes. The mixture was then heated to a reflux temperature of 73°C and refluxing at this temperature was continued for a period of 1 hour.

There were then concurrently added to the reaction mixture, the remainder of the complex solution and the

water azeotrope. The addition of complex was completed in 90 minutes and the azeotrope addition was completed in 75 minutes. 50 parts by weight of the nonvolatile naphthenic diluent oil and 25 weight parts of toluene (added for processing purposes) were then added, and removal of volatile solvents by distillation was begun. At a pot temperature of 131°C, the remainder of the nonvolatile diluent oil was added. Distillation was then continued to a bottoms temperature of 150°C. The resulting product was blow with CO<sub>2</sub> for a period of 30 minutes at a temperature of between 140°C and 160°C, after which it was filtered through diatomaceous earth and cooled. The acetic base number of the finished product was 162.

#### EXAMPLE 8

In this example, an overbased composition was prepared by the process of the present invention, using a sulfurized alkylphenol starting material admixed with a minor amount of an oil soluble sulfonic acid.

Materials Used	Parts
Sulfurized Alkylphenol*	1680.0
Nonvolatile 80 Pale Oil Diluent	127.0
n-Hexane	1200.0
Hexane Solution of Sulfonic Acid**	194.7
Water	119.8
Methyl Cellosolve	400.0
Methoxy Ethanol Solution of Magnesium Ethoxy-ethoxide Carbonate Complex***	1144.0

\*Same as sulfurized alkylphenols used in Example 6

\*\*The hexane solution contained 26.3 weight percent of a sulfonic acid composition derived from alkylbenzenes and having a combining weight of 440. The sulfonic acid acidity of the solution was 0.59 meq./g., and the acid contained 17.5 weight percent nonvolatile mineral oil.

\*\*\*This material contained 7.86 weight percent magnesium and 16.0 weight percent carbon dioxide.

#### PROCESS

The sulfurized alkylphenol, n-hexane, 80 pale oil, hexane sulfonic acid solution, and methyl Cellosolve were charged to a 12 liter, 3 necked creased flask equipped with stirrer, heater, thermometer and reflux condenser. Over a 1 hour period, one half of the total methoxy ethanol-complex solution was added to the reaction mixture while gradually increasing the temperature to 55°C. Upon completion of the addition of this neutralizing amount of the complex, the mixture was heated to reflux, and refluxing was then continued for 20 minutes.

There was then added concurrently to the mixture while it was maintained at reflux temperature, the balance of the methoxy ethanol solution of complex and the water. The addition of the complex was accomplished in 60 minutes, and the water was added over a period of 45 minutes.

The volatile solvents were then removed by distillation to a bottoms temperature of 150°C. The product was stripped by bubbling CO<sub>2</sub> therethrough for a period of 1 hour while maintaining the temperature between 140°C and 160°C. The final product had a base number of 248, and a viscosity at 210°F of 248 centistokes.

Although certain preferred embodiments of the invention have been herein described for illustrative purposes, it will be understood that various modifications

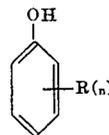
in the process conditions and reactants used can be effected without departure from the basic principles of the invention. Such changes which continue to be based upon such principles are therefore deemed to be within the spirit and scope of the invention except as the same may be necessarily limited by the appended claims or reasonable equivalents thereof.

What is claimed is:

1. A process for preparing a magnesium phenate-containing composition comprising:

a. forming an admixture comprising:

i. about 5 to about 100 parts by weight of alkylphenol or sulfurized alkylphenol, wherein the alkylphenol is represented by the formula

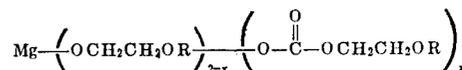


wherein R is a straight or branched chain, saturated or unsaturated, aliphatic hydrocarbon radical having from four to 30 carbon atoms, and n is an integer having a value of 1 or 2, said alkylphenol being characterized further in that the total number of alkyl group carbon atoms is from eight to 40;

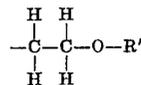
ii. from 2 to 100 parts by weight of a nonvolatile diluent oil having a boiling point in excess of about 160°C; and

iii. from 5 to 120 parts by weight of a volatile process solvent having a boiling point below about 150°C;

b. while the temperature is in the range of from about 30°C to about 60°C, adding to the admixture of alkylphenol, nonvolatile diluent oil and volatile process solvent, a stoichiometric neutralizing amount of carbonated magnesium alcoholate represented by the formula



where R is either a C<sub>1</sub> to C<sub>6</sub> alkyl group, or an organic radical of the formula



where R' is a C<sub>1</sub> to C<sub>4</sub> alkyl group, x is from 0.5 to 1.5, and wherein said magnesium alcoholate contains from about 0.5 mole to about 2.0 moles of CO<sub>2</sub> per mole of metal alcoholate;

c. heating the mixture to a reflux temperature of from about 65°C to about 90°C, and maintaining the mixture at this reflux temperature for a period of from about 20 minutes to about 1½ hours; then

d. removing the volatile materials boiling below about 140°C from the reaction mixture.

2. The process of claim 1 wherein x in said carbonated magnesium alcoholate formula is from about 0.75 to about 1, and wherein said magnesium alcoholate contains from about 0.8 mole to about 1.2 mole of CO<sub>2</sub> per mole of magnesium alcoholate.

3. The process of claim 1 wherein the alkylphenol employed is a mixture containing at least 20 weight percent dialkylphenol with the balance being a monoalkylphenol.

4. The process defined in claim 1 wherein the non-volatile diluent oil is selected from the group consisting of mineral lubricating oils and synthetic lubricating oils.

5. The process defined in claim 1 wherein the admixture of step (a) contains additionally from about 0.1 to about 50 parts of oil soluble sulfonic acid per 100 parts of alkylphenol or sulfurized alkylphenol.

6. The process defined in claim 1 wherein the amounts of materials admixed in step (a) are:

	Parts by Weight
i. alkylphenol or sulfurized alkylphenol	10 - 70
ii. nonvolatile diluent	10 - 90
iii. volatile process solvent	10 - 100

7. The process defined in claim 1 wherein said carbonated magnesium alcoholate is derived from the monomethyl ether of ethylene glycol or from the monoethyl ether of ethylene glycol.

8. The process defined in claim 1 wherein said volatile process solvent consists essentially of hexane.

9. The process defined in claim 1 wherein said volatile material removal is accomplished by

a. distilling the reaction mixture to a bottoms temperature of at least 140°C; then

b. stripping the mixture with CO<sub>2</sub> while maintaining the temperature of the mixture between about 140°C and 160°C.

10. The process defined in claim 9 wherein said magnesium alcoholate is derived from the monomethyl ether of ethylene glycol or the monoethyl ether of ethylene glycol, and contains from about 0.8 mole to about 1.2 mole of CO<sub>2</sub> per mole of magnesium alcoholate.

11. The method defined in claim 10 wherein the alkylphenol employed is a mixture containing at least 20 weight percent dialkylphenol with the balance being a monoalkylphenol.

12. The process defined in claim 11 wherein said nonvolatile diluent oil is selected from the group consisting of mineral lubricating oils and synthetic lubricating oils.

13. The process defined in claim 12 wherein the amounts of materials admixed in step (a) are:

	Parts by Weight
i. alkylphenol or sulfurized alkylphenol	10 - 70
ii. nonvolatile diluent	10 - 90
iii. volatile process solvent	10 - 100

14. The process defined in claim 1 and further characterized to include the step of adding to the reaction mixture after refluxing as described in step (c) and prior to removing volatile materials as described in step (d), an overbasing amount of said carbonated magnesium alcoholate, and concurrently with said addition, adding to the reaction mixture an amount of water exceeding the stoichiometric amount required to hydrolyze the overbasing amount of carbonated magnesium alcoholate.

15. The process defined in claim 14 wherein an amount of carbonated magnesium alcoholate is added which is in a weight ratio to the neutralizing amount of alcoholate added in step (b) of claim 1 of from about 0.5 to 3.0.

16. The process defined in claim 14 wherein from about 0.1 part by weight to about 10 parts by weight of water are added to the reaction mixture concurrently with said overbasing amount of magnesium alcoholate.

17. The process defined in claim 14 wherein sulfurized alkylphenol is mixed with nonvolatile diluent oil and volatile process solvent in step (a) of claim 1.

18. The process defined in claim 14 wherein the overbasing amount of carbonated magnesium alcoholate used is from about 10 parts by weight to about 300 parts by weight.

19. The process defined in claim 14 wherein the amounts of materials admixed in step (a) are:

	Parts by Weight
i. alkylphenol or sulfurized alkylphenol	10 - 70
ii. nonvolatile diluent oil	10 - 90
iii. volatile process solvent	10 - 100

20. The process defined in claim 14 wherein the admixture in step (a) contains additionally from about 0.1 to about 50 parts of oil soluble sulfonic acid per 100 parts of alkylphenol or sulfurized alkylphenol.

21. The process defined in claim 14 wherein the non-volatile diluent oil is selected from the group consisting of mineral lubricating oils and synthetic lubricating oils.

22. The process defined in claim 15 wherein the admixture of step (a) contains additionally from about 0.1 to about 50 parts of an oil-soluble sulfonic acid per 100 parts of alkylphenol or sulfurized alkylphenol.

23. The process defined in claim 16 wherein from about 1.1 moles to about 4 moles of water are added to the reaction mixture per mole of the overbasing amount of carbonated magnesium alcoholate added thereto.

24. The process defined in claim 17 wherein the sulfurized alkylphenol content of said admixture is a mixture of sulfurized monononylphenol and sulfurized dinonylphenol.

25. The process defined in claim 19 wherein an amount of carbonated magnesium alcoholate is added which is in weight ratio to the neutralizing amount of alcoholate added in step (b) of claim 1 of from 0.5 to 3.0.

26. The process defined in claim 25 wherein from about 0.1 part by weight to about 10 parts by weight of water are added to the reaction mixture concurrently with said overbasing amount of magnesium alcoholate.

27. The process defined in claim 26 wherein a mixture of sulfurized monoalkylphenol and sulfurized dialkylphenol is mixed with nonvolatile diluent oil and volatile process solvent in step (a) of claim 1.

28. The process defined in claim 27 wherein the admixture of step (a) contains additionally from about 0.1 to about 50 parts of an oil soluble sulfonic acid per 100 parts of alkylphenol or sulfurized alkylphenol.

29. The process defined in claim 14 wherein less than 70 weight parts of nonvolatile diluent oil is present in

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said admixture formed in step (a), and further characterized as including the step of adding an additional amount of nonvolatile diluent oil to the reaction mixture prior to the removal of all of said volatile materials

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therefrom in step (e), said additional amount of nonvolatile diluent oil together with said amount in said admixture totaling less than 70 weight parts.

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