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SYNTHETIC LUBRICANT

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This invention has to do with the art of lubrication and, more particularly, has to do with the preparation of synthetic lubricants.

In the lubricant art considerable progress has been realized in recent years in the production of lubricants characterized by one or more specific properties and adapted for particular uses. In the main, this progress can be attributed to two developments: the first, new refining procedures and, the second, addition agents capable of imparting particular properties to available lubricants. Although these lubricants are somewhat superior to those formerly available in the art, in general, they too suffer in one or more respects. For example, while such oils may have superior viscosity characteristics, etc., they may have undesirable oxidation characteristics, etc. Recently, however, in an effort to obtain superior lubricants endowed with specific and superior characteristics, a new field has been explored, namely, the synthesis of lubricants from various materials. The products so obtained have been aptly described in the art as "synthetic lubricants."

While many of the synthetic lubricants recently prepared have been found desirable for various uses in view of a superior characteristic such as improved oiliness, viscosity, resistance to oxidation, etc., they invariably have extremely high pour points which naturally militate against their wide use in the art. Generally they suffer too from the disadvantage of a failure to respond to pour point depressants when the latter are incorporated therewith in relatively small amounts.

The present invention is predicated upon the discovery of a method for the preparation of a new and novel class of synthetic lubricants which are characterized by relatively low pour points and which are responsive to substantial pour point depression upon the addition of small amounts of a pour point depressant and paraffin wax thereto.

This is an anomalous discovery inasmuch as paraffin wax has always been removed, not added, to a lubricant in order to improve its pour characteristics; nonetheless, the results provided hereinafter clearly establish the fact that small amounts of a pour point depressant and of paraffin wax cooperate to substantially lower the pour points of the synthetic lubricants of the present invention.

As used herein the expression "substantial pour point lowering" obtained with a small amount of a pour point depressant and a small amount of paraffin wax demonstrates a lowering of the A. S. T. M. pour point of a lubricant of 10° F. or more. As appreciated by those familiar with the art, the A. S. T. M. pour test is accurate to only about 5° F.

Not only are the new and novel synthetic lubricants of this invention characterized as indicated

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above but they are further characterized by outstanding viscosity index values and oxidation properties.

The method contemplated herein for the preparation of the new and novel synthetic lubricants involves reacting certain partially halogenated paraffin waxes with certain aromatic compounds in the presence of a Friedel-Crafts catalyst at a condensation temperature and thereafter separating the catalyst and any unchanged reactants from the reaction products so obtained. While the present method may be briefly summarized by the foregoing statement, it is much more complex than as so stated, inasmuch as several of the factors recited therein are critical in nature and must be followed religiously lest products of a somewhat inferior character be formed. Specifically, the several critical factors, all of which are interrelated, include the following:

1. Type of paraffin wax,
2. Degree of halogenation of the paraffin wax,
3. Aromatic hydrocarbon, and
4. Proportions of halogenated paraffin wax and of aromatic hydrocarbon.

Of importance in the present method is the type of paraffin wax used. We have found that when a crystalline paraffin wax having a melting point not greater than about 140° F. is used, synthetic lubricants of relatively low pour point and susceptible to pour point depression are obtained. Particularly preferred waxes of this type, however, are those having melting points from about 90° F. to about 130° F. These waxes have a crystalline structure, as opposed to other paraffin waxes which do not have a well-defined crystal structure and are amorphous in character. The latter are commonly referred to as "amorphous" waxes, and are considered to be undesirable for the purposes of this invention, for such waxes would result in products having relatively high melting points and having comparatively little susceptibility to pour point depression. Of the waxes used herein we have found that particularly outstanding results are obtained with a crystalline paraffin wax containing on the average of 24 carbon atoms and having a melting point of about 126° F. It is to be understood, however, that all crystalline paraffin waxes characterized by melting points not greater than about 140° F., and obtained from all sources, such as petroleum wax fractions, the Fischer-Tropsch synthesis, etc., are contemplated herein.

Accordingly, when used hereinafter the expression "wax," unless otherwise qualified, denotes the desired reactant: crystalline paraffin wax. This term will also apply to the wax added to the synthetic lubricant prepared as described herein, together with a pour depressant similarly added.

With regard to the degree of halogenation of

wax which must be followed, it may first be said that all halogens may be used. In view of the relative cost, availability, etc., however, chlorine is preferred and, for these reasons, the following description will be confined to the chlorinated waxes. In addition, inasmuch as the degree of halogenation of wax is closely allied with the aromatic hydrocarbon used in our method, these two factors will be described together. When benzene is used as the aromatic hydrocarbon reactant, the chlorinated wax with which it is reacted should contain from 19 to 21.5 per cent chlorine. Similarly, with toluene, the chlorinated wax should contain from 12 to 26 per cent chlorine and, with a xylene, or a mixture of xylenes, the chlorinated wax should contain from 12 to 27 per cent chlorine. When chlorine concentrations in excess of the maxima recited above are present in the chlorinated wax reactant, the products obtained therewith are characterized by an undesirably high viscosity, as well as little or no response to small amounts of a pour point depressant and wax. In the same regard, when chlorinated waxes containing less chlorine than the minima recited above are used, the products so obtained contain excessive amounts of wax and are marked by little or no response to small amounts of the aforesaid added materials.

It should be understood that a 12 per cent chlorinated wax is one prepared by introducing chlorine into a crystalline paraffin wax, as defined above, until the weight of the wax has increased about 12 per cent. Obviously, then, the product is predominantly a mono-chlorinated wax containing substantial quantities of unchlorinated and polychlorinated wax. Correspondingly, a 20 per cent chlorinated wax is predominately dichlorinated wax with substantial quantities of unchlorinated wax, mono-chloro wax and more highly chlorinated wax.

It will be clear from the foregoing that the aromatic hydrocarbon reactants contemplated herein are benzene, toluene and xylenes. It will also be understood, of course, that when other halogens are used in place of chlorine in the aliphatic wax reactant, the foregoing percentages naturally will be subject to change in proportion to the relative atomic weights of the individual halogens.

Of primary importance are the relative amounts or proportions of the reactants, namely, chlorinated wax and aromatic hydrocarbon. In order to obtain the desired products, at least one mole of one of the aforesaid aromatic hydrocarbons is reacted with an amount of chlorinated wax which contains one atomic weight of chlorine. In short, at least one mole of an aromatic hydrocarbon, as defined above, is used for each atomic weight of chlorine in the chlorinated wax reactant. Accordingly, it will be clear that the reaction products obtained with such proportions are predominantly monowax aromatics. If less than one mole of aromatic hydrocarbon for each atomic weight of chlorine in a chlorowax is used, products of less desirable character are obtained. The latter products, unlike those contemplated herein, suffer from their failure to respond to small amounts of a pour point depressant and wax, and some suffer also from relatively high pour points.

The reaction temperatures useful in the present method are those normally referred to in the art as Friedel-Crafts condensation temperatures. While temperatures from about 25° C. to about 80° C. may be used satisfactorily when benzene is the aromatic reactant, the preferred reaction temperature is about 80° C., the boiling point of benzene. Similarly, when toluene and xylenes are used, the temperature may range from 25° C. to 108° C. and from 25° C. to 145° C., respectively, while the preferred temperatures are 105° C. and about 135–145° C., respectively.

The amount of catalyst used in the condensation should be relatively small, that is, a catalytic amount. Generally, amounts from about 1.0 per cent to about 10 per cent by weight of the chlorowax reactant are satisfactory, and amounts of the order of 3 per cent are preferred. Catalysts such as aluminum chloride, zinc chloride, ferric chloride, boron trifluoride, hydrogen fluoride, etc., serve the purposes of this invention; and for this reason, the catalysts are defined broadly herein as those capable of effecting a Friedel-Crafts type condensation. Particularly preferred, however, is aluminum chloride.

In order to more fully illustrate the method by which our synthetic lubricants are obtained the following typical procedure is detailed hereinbelow.

Example.—Preparation of wax-benzene (1–19)

Two moles of benzene (156 grams) and 187 grams of a 19 per cent chlorowax (126° F. A. S. T. M. melting point) are placed in a flask equipped with a thermometer, reflux condenser and an electrically driven stirrer. The chlorowax used contains 1 atomic weight of chlorine and, therefore, there is a molar excess of benzene. Aluminum chloride is slowly added to the chlorowax and benzene at a temperature of about 20–25° C. while agitating the same. The temperature is then raised to the reflux temperature of benzene, about 80° C., and maintained for about 1 hour to complete the condensation of chlorowax and benzene. The reaction mixture is then purified by filtering through a contact clay to remove aluminum chloride. The filtrate is then heated to remove excess benzene and the residue obtained thereby is a wax-substituted benzene containing some unreacted wax, about 10 per cent unreacted wax. The unreacted wax is removed by vacuum distillation (it may also be removed by conventional dewaxing procedures). The product is wax-benzene (1–19).

Parenthetical expressions (1–19) indicating that benzene is substituted with 1 wax group and that the chlorowax used in the preparation thereof contained 19 per cent chlorine are used hereinafter to so identify each product illustrative of the present invention, as well as related products which do not possess the superior characteristics of the lubricants prepared as outlined above.

It will be understood, of course, that any unreacted benzene, for example, can be used again in the preparation of additional product, and that unreacted wax can be further chlorinated for future use.

Although we have illustrated our synthetic lubricants by a product free of unreacted wax (removed by vacuum distillation, solvent dewaxing, etc.), it is to be understood that it is not essential that all of the unreacted wax be removed. Accordingly, both wax-free synthetic lubricants and synthetic lubricants containing relatively small amounts of unreacted wax are contemplated herein.

In order to illustrate the superior characteristics of the synthetic lubricants of the present invention, several typical examples are presented hereinbelow in Table I. There, the S. U. V. at

210° F., viscosity index and A. S. T. M. pour test of the above synthetic lubricants are shown; all of these characteristics and methods for determining the same are well known and need not be further described at this point. In addition the response of these synthetic lubricants with and without a pour point depressant and added wax is also demonstrated. Also present in Table I for comparison with the synthetic lubricants contemplated herein are several typical products which are much less desirable for use as lubricants for one reason or another. For convenience in identifying the latter products, they are preceded by an "*x*" in Table I.

Table I

Product	S. U. V. @ 210° F.	V. I.	ASTM Pour Test (°F.)	ASTM Pour Tests (°F.) With Pour Point Depressants, and With or Without Wax							
				½% A [1]	½% A [1] +½% Added Wax	¼% A [1]	¼% A [1] +½% Added Wax	½% B [2]	½% B [2] +½% Added Wax	½% C [3]	½% C [3] +½% Added Wax
x wax-benzene (1-12)	48.7	120.1	+25	+20	+10	+10	+15	+30	+30	+20	+20
x wax-benzene (1-15)	60.3	116.5	+30	+20	+15	+20	+15	+25	+30	+25	+15
wax-benzene (1-19)	91.0	121.4	+15	+20	−5			+20	0		
wax-benzene (1-20)	100.0	112.7	+15	+20	+5						
wax-benzene (1-21.5)	168.0	106.7	+15	+10	+5						
x wax-benzene (1-24)	296.7	105.0	+10	+15	+10			+15	+15		
wax-toluene (1-12)	47.5	119.8	+25	+15	+15						
wax-toluene (1-18)	61.1	110.0	+25	−5	−5	+5	0	+15	0	+5	0
wax-toluene (1-26)	128.2	71.1	+35	+10				+10	+10		
x wax-toluene (1-28)	187.0	69.5	+25	+25	+20			+20	+20		
wax-xylene (1-12)	44.4	119.0	+25	+15	+15			+15	+15		
wax-xylene (1-21)	63.6	89.5	+20	−5	−5			+15	+10		
wax-xylene (1-27)	124.0	49.7	+25	+10							
x wax-xylene (1-28)	127.2	47.3	+20	+15	+15			+15	+15		

[1] Pour Depressant A=Tetra-wax phenol phthalate.
[2] Pour Depressant B=Wax-naphthalene.
[3] Pour Depressant C=Stannous phenate-stannous carboxylate of wax phenol carboxylic acid disulfide.

It will be clear on inspection of the pour test results set forth in Table I above that wax-benzenes prepared from a chlorowax containing 12, 15 or 24 per cent chlorine are erratic in their response to a pour point depressant and are similarly responsive to the combination of a pour point depressant and added wax. None of these products consistently shows a pour point lowering of at least 10° F. when the aforesaid additives are incorporated therewith in small amounts, and some even have higher pour points when the said additives are incorporated therewith. Surprisingly, however, when the chlorowax reactant contains from 19 to 21.5 per cent chlorine, the wax-benzenes obtained therefrom are susceptible of at least a 10° F. pour point lowering when small amounts of a pour point depressant and wax are added thereto. Similarly, characterized are the wax-toluenes (1–12) to (1–26) and the wax-xylenes (1–12) to (1–27). It appears then that a pour point depressant and added wax cooperate in some manner, as yet undetermined, to substantially lower the pour point of our synthetic lubricants which by themselves have relatively low, and therefore, desirable pour points.

It will be further noted from inspection of the results provided in Table I that the wax-benzenes (1–19) to (1–21.5) have viscosities (S. U. V.) and viscosity indexes which make them extremely valuable lubricants. The wax-toluenes (1–12) to (1–26) and the wax-xylenes (1–12) to (1–27) are similarly characterized. These synthetic lubricants are useful both as automotive and aviation lubricants.

To illustrate the critical nature of the proportions of aromatic hydrocarbon and of the halogen wax reactants in obtaining the synthetic lubricants of this invention, and those related materials of somewhat inferior character, several typical examples of wax-toluenes are shown in Table II below. The data in this table are similar to those shown in Table I above and the products not contemplated herein are again identified by the letter "*x*" which precedes them, as "*x* wax-toluene (3–12)."

Table II

Product	S. U. V. @ 210° F.	V. I.	ASTM Pour Test (°F.)	ASTM Pour Tests (°F.) With Pour Point Depressants, and With or Without Wax							
				½% A [1]	½% A [1] +½% Added Wax	¼% A [1]	¼% A [1] +½% Added Wax	½% B [2]	½% B [2] +½% Added Wax	½% C [3]	½% C [3] +½% Added Wax
wax-toluene (1-12)	47.5	119.8	+25	+15	+15						
wax-toluene (1-18)	61.1	110.0	+25	−5	−5	+5	0	+15	0	+5	0
wax-toluene (1-26)	128.2	71.1	+35	+10				+10	+10		
x wax-toluene (3-12)	42.6	140	+25	+25	+25	+25	+20	+25	+25		
x wax-toluene (3-18)	64.9	119.6	+25	+25	+20	+20	+20	+25	+30		
x wax-toluene (3-24)	170.5	107.4	+15	+20	+10	+20	+10				
x wax-toluene (5-12)	42.6	141	+20	+20	+25	+25	+20	+25	+30		
x wax-toluene (5-18)	83.3	122.5	+25	+30	+30	+30	+25	+30	+30		
x wax-toluene (5-24)	342.4	114.1	+20	+25	+20	+25	+30				

[1] Pour Depressant A=Tetra-wax phenol phthalate.
[2] Pour Depressant B=Wax-naphthalene.
[3] Pour Depressant C=Stannous phenate-stannous carboxylate of wax phenol carboxylic acid disulfide.

It will be seen that the wax-toluenes (1–12) to (1–26) in Table II are responsive to substantial pour point lowering with a pour point depressant and added wax. Yet, wax-toluenes prepared with chlorowaxes containing the same percentages (12,

18 and 26) of chlorine as those used in the preparation of the aforesaid monowax synthetic lubricants, but prepared with more than one mole of toluene for each atomic weight of chlorine in the chlorowax, are not so responsive. For example, wax-toluene (3–12) has an A. S. T. M. pour point of +25° F., as has the combination of the same synthetic, a pour point depressant and added wax. In some cases, as with wax-toluene (3–18) and wax-toluene (5–24), the pour point of the synthetic with a pour point depressant and added wax is higher, rather than equal to or lower than, the pour point of the synthetic alone.

In Tables I and II above several typical pour point depressants were used to illustrate the broad class. It is to be understood, however, that all such additives are broadly contemplated. Preferred, however, are depressants of the wax-phenol phthalate class and of the metal organic class which are typified by depressants A and C in the tables. Particularly preferred of such materials are tetra-wax phenol phthalate (A) and stannous phenate, stannous carboxylate of wax phenol carboxylic acid disulfide (C). Although varying amounts of these materials may be used, satisfactory results are obtained with concentrations of from about ¼ per cent to about ½ per cent.

The paraffin wax which is added to our synthetic lubricants together with a pour point depressant is a microcrystalline paraffin wax having a melting point not greater than about 140° F., that is, of the same type of wax as that used in the halogenated wax reactant. While about 0.5 per cent is preferred for use, amounts from about 0.25 per cent to about 1.0 per cent may also be used. It will be understood, of course, that the amounts of added wax and of the pour point depressant will be determined to some extent by the degree of pour point lowering of the synthetic lubricant required for a particular use.

We claim:

1. The method of preparing a synthetic lubricant of low pour point and susceptible of substantial pour point lowering, from a crystalline paraffin wax having a melting point not greater than about 140° F. and a xylene, which comprises: partially halogenating the paraffin wax to form a mixture of halogenated wax and substantially unhalogenated wax, said mixture being selected from the group consisting of a mixture containing an amount of halogen corresponding to from 12 to 27 percent chlorine; mixing the halogenated wax and unhalogenated wax with said xylene in the proportion of at least one mol of xylene to each atom of halogen in the halogenated wax mixture; adding thereto a catalyst capable of effecting a Friedel-Crafts type condensation; heating the mixture thus obtained at a Friedel-Crafts condensation temperature until substantially all of the halogenated wax has reacted with said xylene; and separating the Friedel-Crafts catalyst, unreacted xylene and unhalogenated wax from the reaction mixture, so obtained, to obtain said synthetic lubricant.

2. The method of preparing a synthetic lubricant of low pour point and susceptible of substantial pour point lowering, from a crystalline paraffin wax having a melting point not greater than about 140° F. and a xylene, which comprises: partially chlorinating the paraffin wax to form a mixture of chlorinated wax and substantially unchlorinated wax containing from 12 to 27 per cent chlorine; mixing the chlorinated wax and unchlorinated wax with said xylene in the proportion of at least one mol of xylene to each atom of chlorine in the chlorinated wax mixture; adding a Friedel-Crafts catalyst thereto; heating the mixture thus obtained under refluxing conditions to the refluxing temperature of said xylene until substantially all of the chlorinated wax has reacted with said xylene; and separating the Friedel-Crafts catalyst, unreacted xylene and unchlorinated wax from the reaction mixture, so obtained, to obtain said synthetic lubricant.

3. The method of preparing a synthetic lubricant of low pour point and susceptible of substantial pour point lowering, from a crystalline paraffin wax having a melting point not greater than about 140° F. and a xylene which comprises: partially chlorinating said paraffin wax to form a mixture of chlorinated wax and substantially unchlorinated wax containing from 12 to 27 per cent chlorine; mixing said chlorinated wax and unchlorinated wax with a xylene in the proportion of at least one mole of xylene to each atom of chlorine in the chlorinated wax mixture; adding anhydrous aluminum chloride catalyst thereto; heating the mixture thus obtained under refluxing conditions to the refluxing temperature of said xylene until substantially all of said chlorinated wax has reacted with said xylene; and separating said aluminum chloride, unreacted xylene and unchlorinated wax from the reaction mixture, so obtained, to obtain said synthetic lubricant.

4. The method of preparing a synthetic lubricant of low pour point from a crystalline paraffin wax having a melting point not greater than about 140° F. and a xylene, which comprises: partially halogenating the paraffin wax to form a mixture of halogenated wax and substantially unhalogenated wax containing an amount of halogen corresponding to from 12 to 27 per cent chlorine for reaction with a xylene; mixing the halogenated wax and unhalogenated wax with said xylene in the proportion of at least one mole of xylene to each atom of halogen in the halogenated wax mixture; adding thereto a catalyst capable of effecting a Friedel-Crafts type condensation; heating the mixture thus obtained at a Friedel-Crafts condensation temperature until substantially all of the halogenated wax has reacted with said xylene; separating the Friedel-Crafts catalyst, unreacted xylene and unhalogenated wax from the reaction mixture, so obtained; and adding thereto small amounts of a pour point depressant and a crystalline paraffin wax having a melting point not greater than about 140° F.

5. The method of preparing a synthetic lubricant of low pour point from a crystalline paraffin wax having a melting point not greater than about 140° F. and a xylene, which comprises: partially chlorinating said paraffin wax to form a mixture of chlorinated wax and substantially unchlorinated wax containing from 12 to 27 per cent chlorine; mixing said chlorinated wax and unchlorinated wax with a xylene in the proportion of at least one mole of a xylene to each atom of chlorine in the chlorinated wax mixture; adding anhydrous aluminum chloride catalyst thereto; heating the mixture thus obtained under refluxing conditions to the refluxing temperature of said xylene until substantially all of said chlorinated wax has reacted with said xylene; separating said aluminum chloride, unreacted xylene and unchlorinated wax from the reaction mixture, so obtained; and adding small amounts of a pour point depressant and a crystalline paraffin wax of the type defined above thereto.

6. A lubricant of low pour point comprising a synthetic lubricant having incorporated therewith small amounts of a pour point depressant and a crystalline paraffin wax having a melting point not greater than about 140° F., said synthetic lubricant being prepared from a crystalline paraffin wax of the type defined above and a xylene, by: partially halogenating the paraffin wax to form a mixture of halogenated wax and substantially unhalogenated wax containing an amount of halogen corresponding to from 12 to 27 per cent chlorine for reaction with a xylene; mixing the halogenated wax and unhalogenated wax with said xylene in the proportion of at least one mole of xylene to each atom of halogen in the halogenated wax mixture; adding thereto a catalyst capable of effecting a Friedel-Crafts type condensation; heating the mixture thus obtained at a Friedel-Crafts condensation temperature until substantially all of the halogenated wax has reacted with said xylene; and separating the Friedel-Crafts catalyst, unreacted xylene and unhalogenated wax from the reaction mixture, so obtained, to obtain said synthetic lubricant.

7. A lubricant of low pour point comprising a synthetic lubricant having incorporated therewith small amounts of a pour point depressant and a crystalline paraffin wax having a melting point not greater than about 140° F., said synthetic lubricant being prepared from a crystalline paraffin wax of the type defined above and a xylene by: partially chlorinating said paraffin wax to form a mixture of chlorinated wax and substantially unchlorinated wax, said mixture containing from 12 to 27 per cent chlorine; mixing the chlorinated wax and unchlorinated wax with a xylene in the proportion of at least one mole of said xylene to each atom of chlorine in the chlorinated wax mixture; adding anhydrous aluminum chloride catalyst thereto; heating the mixture thus obtained under refluxing conditions to the refluxing temperature of said xylene until substantially all of the chlorinated wax has reacted with said xylene; and separating said aluminum chloride, unreacted xylene and unchlorinated wax from the reaction mixture, so obtained, to obtain said synthetic lubricant.

8. The method of preparing a synthetic lubricant of low pour point and susceptible of substantial pour point lowering from a crystalline paraffin wax having a melting point not greater than about 140° F. and a xylene, which comprises: partially halogenating the paraffin wax to form a mixture of halogenated wax and substantially unhalogenated wax, said mixture being selected from the group consisting of a mixture containing an amount of halogen corresponding to from 12 to 27 per cent chlorine; mixing the halogenated wax and unhalogenated wax with said xylene in the proportion of at least one mol of xylene to each atom of halogen in the halogenated wax mixture; adding thereto a catalyst capable of effecting a Friedel-Crafts type condensation; heating the mixture thus obtained at a Friedel-Crafts condensation temperature until substantially all of the halogenated wax has reacted with said xylene; and separating the Friedel-Crafts catalyst, unreacted xylene and substantially all unhalogenated wax from the reaction mixture, so obtained, to obtain said synthetic lubricant.

9. The method of preparing a synthetic lubricant of low pour point and susceptible of substantial pour point lowering, from a crystalline paraffin wax having a melting point not greater than about 140° F. and a xylene, which comprises: halogenating the paraffin wax to form a halogenated wax selected from the group consisting of a halogenated wax containing an amount of halogen corresponding to from 12 to 27 per cent chlorine; mixing the halogenated wax and said xylene in the proportion of at least one mol of xylene to each atom of halogen in the halogenated wax; adding thereto a catalyst capable of effecting a Friedel-Crafts type condensation; heating the mixture thus obtained at a Friedel-Crafts condensation temperature until substantially all of the halogenated wax has reacted with said xylene; and separating the Friedel-Crafts catalyst and any unreacted xylene from the reaction mixture, so obtained, to obtain said synthetic lubricant.

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