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PROCESS FOR PREPARING SULFURIZED LUBRICATING COMPOSITION

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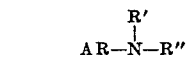
3 Claims. (Cl. 260—399)

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My invention relates to a process for preparing sulfurized lubricating compositions and has to do particularly with procedure for preparing sulfurized lubricating oil compositions adapted for use as cutting oils, extreme pressure lubricants and the like.

Cutting oil is used to cool and lubricate the tools and work pieces in the various metal forming operations which encounter extreme pressure, such as cutting, drilling and the like. Criteria by which cutting oils are judged include color, stability of oil with retention of quality with repeated re-use, odor particularly when heated and increased life of cutting tool. The most effective oils are those containing sulfur, and heretofore fatty oils have been heated in the presence of sulfur at very high temperatures and for such a length of time that various disagreeable characteristics have been imparted to the oil.

My invention is particularly applicable to the preparation of sulfurized oils and fats for use in the manufacture of cutting oils and lubricants; these sulfurized oils and fats are comparatively free from obnoxious odors and have relatively light transparent colors and hence are far superior to sulfurized oils and fats on the market. I obtain this unexpected result by using a sulfurized oil which has been sulfurized in the presence of a catalyst, having the general formula



wherein R′ and R″ represent substituents selected from the class consisting of hydrogen, lower aliphatic radicals and monocyclic aromatic radicals and AR is a benzene ring with a lower alkoxy or monocyclic aryloxy group attached to the ortho or para position. Among the catalysts used are

p-Anisidine
N-benzyl-p-anisidine
N-dibenzyl-p-anisidine
o-Anisidine
N-benzyl-o-anisidine
p-Phenetidine
N-benzyl-p-phenetidine
N-dibenzyl-p-phenetidine
o-Phenetidine
N-benzyl-o-phenetidine
N-ethyl-p-phenetidine
N-n-propyl-p-phenetidine
p-Phenoxyaniline

Furthermore, the process of my invention may be employed for sulfurizing mineral oils thus obviating the necessity of preparing sulfurized fatty bases which are added to mineral oils in order to obtain sulfurized cutting oils. Cutting oils and lubricants prepared from the sulfurized products of my invention are at least as effective as similar products prepared from present commercially available sulfurized substances, and are superior to such products in that they do not possess objectionable odors and colors, are substantially non-corrosive, and are stable products. The light colored product so obtained permits easy observance of the thread cutting or machining operations. Many of the prior art oils had to be subjected to treatment with acid and clay to improve the color, usually without permanent improvements, but such treatment is not necessary in my invention.

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In practicing my invention, an oil, sulfur and a catalyst having the general formula set forth in the paragraph above are reacted in suitable proportions and under proper conditions to obtain a sulfur base, and then this sulfur base is dispersed in correct proportions in a lubricating oil of predetermined viscosity to obtain a lubricating composition having the desired characteristics for the purpose intended. This invention is particularly applicable to the preparation of sulfur-type cutting oils and for this purpose a light lubricating oil is used. Other lubricating compositions such as extreme pressure base lubricating oils and extreme pressure gear lubricants, hypoid gear lubricants, etc., may be obtained by using mineral lubricating oils of high viscosity. In the preparation of sulfur-type cutting oils, a light lubricating oil of about 60 to 150 viscosity range at 100° F. is employed.

The following examples illustrate certain advantageous embodiments of my invention.

Example 1.—This example illustrates one advantageous method of preparing sulfurized oils in accordance with this invention; an excellent sulfurized fatty oil being prepared from the following materials:

	Pounds
Lard oil	89
Sulfur	10
N-benzyl-p-phenetidine	1

The lard oil employed was a commercial lard oil known as extra winter-strained lard oil.

In preparing this catalytically sulfurized fatty oil, or sulfur base, the lard oil was heated to 300° F. and then the sulfurization catalyst, namely, N-benzyl-p-phenetidine was added with stirring. Then, while continuing the stirring the sulfur was added to the hot mixture. The reaction mixture

was then held at a temperature between 310° and 320° F. for ¾ hour to complete the reaction. The sulfurized oil was then cooled to room temperature.

The catalytically sulfurized lard oil or sulfur base so obtained was a stable, clear, transparent liquid. It was substantially non-corrosive and free from obnoxious odors. This liquid sulfurized base was readily soluble in a wide variety of mineral lubricating oils. When incorporated in mineral oils in amounts of from 1 to 25 per cent by weight based on the mineral oil, improved lubricants are readily obtained. For instance, improved cutting oils and other lubricants may be readily prepared in this way. The following example illustrates the preparation of one such improved cutting oil.

Example 2.—In preparing this improved cutting oil, 22 pounds of the catalytically sulfurized lard oil obtained in Example 1 were dissolved in 78 pounds of a commercial mineral lubricating oil, namely, a paraffin oil of viscosity 100 S. U. S. and of color 2 on the N. P. A. scale. The improved cutting oil so obtained when subjected to standard lathe test gave outstanding performance, as compared to the mineral oil itself and a commercial cutting oil containing a small amount of sulfurized lard oil made without a catalyst. The lathe results obtained are given in the following table wherein "F. P. M." designates feet per minute.

Description	Cutting Speed, F.P.M. for Tool Life of—		Tool Life, min. at Cutting Speed of 200 F. P. M.
	10 min.	100 min.	
Dry Cutting	225	170	27
Paraffin Oil	252	190	73
Improved Cutting Oil of Example 2	285	247	2,500
Prior Commercial Cutting Oil	285	240	1,300

The prior commercial cutting oil employed in the above test was a composition comprising 78 per cent of a light paraffinic neutral oil blend and 22 per cent of sulfurized lard oil (non-catalytic); the parts being parts by weight. Non-catalytic sulfurized lard oil in this commercial cutting oil was prepared by heating lard oil to 275°–300° F., adding 10 per cent by weight of sulfur to the heated oil over a period of 2 hours, and then further heating this reaction mixture at 330°–340° F. for ½ hour. As shown by the test data given in the above table, the improved cutting oil prepared in Example 2 ante, is markedly superior to the prior commercial cutting oil as well as to the plain mineral oil employed in these comparative tests.

That is, Examples 1 and 2 ante are typical and illustrative of the improved results obtainable by the present invention. In the practice of this invention, other catalytically sulfurized oils may be prepared as illustrated in Example 1 and employed in making other improved cutting oils and lubricants as illustrated in Example 2. For instance, other fatty oils, such as corn, soybean oil, cottonseed oil, sperm oil and like fatty oils may be used in lieu of lard oil in Example 1 above. The preparation of a catalytically sulfurized sperm oil is illustrated below. The catalytically sulfurized sperm oil may likewise be incorporated in various mineral lubricating oils to produce improved cutting oils and other lubricants having extreme pressure characteristics. Such embodiments of this invention are illustrated in the following example:

Example 3.—Sperm oil was sulfurized according to the above procedure described for lard oil sulfurization. The product was blended with a 70 viscosity (S. U. S.) mineral oil (50:50) and the blend was free of objectionable odor. The 50:50 blend was added to an automotive lubricating oil (0.5 per cent by weight) to determine its ability to prevent corrosion. The tests were made at 325° F. in an Underwood oxidation test using General Motors truck Cu-Pb bearings. After 5 hours, the bearing actually was 5 mg. heavier instead of losing weight as when corrosion occurs. When the automotive lubricating oil was used without the addition of the inhibitor represented in the 50:50 mixture, there was a loss of 93 mg. after a 5 hour test period. Thus, a decided advantage is obtained when 0.5 per cent of the 50:50 mixture is employed.

Also, in the practice of the present invention various other fatty oils may be catalytically sulfurized by heating them with sulfur in the presence of a catalyst of the type described ante. Further, other sulfurized bases, useful in making improved cutting oils may be prepared from petroleum oils, such as a 200 S. U. S. Texas oil, or from olefinic hydrocarbons, such as octadecene and other olefinic hydrocarbons obtained by the cracking of mineral oils. The petroleum oils and the olefinic hydrocarbons readily react with sulfur and can be catalytically sulfurized with advantage using N-benzyl-p-phenetidine and similar catalysts. The following examples illustrate the catalytic sulfurization of petroleum oils and olefinic hydrocarbons in accordance with this invention.

Example 4.—In this example, octadecene-1 was sulfurized with 10 per cent by weight of sulfur using N-benzyl-p-phenetidine as the catalyst. In doing so, the octadecene-1 was heated to 300° F. and 1 per cent by weight of N-benzyl-p-phenetidine was added. Next 10 per cent by weight of sulfur was added to the hot mixture and the reaction mixture maintained between 310° and 320° F. for 1½ hours to complete the reaction.

The sulfurized base so obtained was stable and light in color. It had a good odor. On the other hand, if the catalyst was omitted in the foregoing procedure, the sulfurized product was very sour and very dark in color. Thus, it is evident that the small amount of N-benzyl-p-phenetidine employed as the catalyst in the foregoing example markedly alters the result of the reaction and facilitates the production of light-colored, sulfurized bases having improved properties for the present purposes.

Example 5.—In this example, a 200 S. U. S. Texas oil was sulfurized with 4 per cent by weight of sulfur using N-benzyl-p-phenetidine as the catalyst. In doing so, the Texas oil was heated to 350° F. and 1 per cent by weight of N-benzyl-p-phenetidine was added. Next 4 per cent by weight of sulfur was added to the hot mixture and the reaction mixture maintained between 340° F. and 350° F. for 4 hours.

The sulfurized base so obtained was stable and it had a good odor. However if the catalyst was omitted in the foregoing procedure, the sulfurized product had objectionable odor. The final sulfur content of this product was 2.5 per cent by weight.

For instance, the sulfurized base obtained in Examples 4 and 5 above is readily soluble in mineral lubricating oils and when incorporated therein in small amounts markedly improved their properties and utility as cutting oil.

In the foregoing examples, in lieu of N-benzyl-p-phenetidine, other catalysts of the type shown above may be employed and other improved sulfur bases and improved cutting oils prepared.

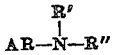
In general, by using catalysts in accordance with this invention, the sulfurization of fatty oils and mineral oils can be effected at relatively low temperatures and with shorter periods of heating; the sulfurized oils or bases so produced having a lighter color and other advantageous properties for the present purposes.

While various amounts of the catalysts may be used, it has been found most desirable in attaining all of the beneficial results of this invention to use from about 0.1 per cent to 2 per cent of the catalyst and most preferably 1 per cent.

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.

What I claim is:

1. A process of preparing a sulfurized oil which comprises reacting sulfur with oil in the presence of a catalyst having the general formula



wherein R′ and R″ represent substituents selected from the class consisting of hydrogen, lower aliphatic radicals and monocyclic aromatic radicals and AR is a benzene ring with a lower alkoxy or monocyclic aryloxy group attached to the ortho or para position.

2. As an improvement in preparing sulfurized oils, the improved process which comprises heating a fatty oil to approximately 300° F., adding from 0.1 to 2.0 per cent of N-benzyl-p-phenetidine to the hot oil, gradually adding sulfur to the hot mixture and maintaining the reaction mixture at a temperature between 310° and 320° F. until the sulfurization is complete.

3. The process of claim 2 wherein said fatty oil is lard oil.

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