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LUBRICANTS

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This invention relates to lubricants and more particularly to sulfur-containing oils, such as cutting oils and the like.

The sulfurization of mineral lubricating oils to improve the character thereof, especially for uses wherein high pressures are encountered, as in certain gear lubrication, cutting tools, etc., is well known in the art. Sulfur, however, is difficultly incorporated into most mineral oils due to the low solubility therein and when the oil and sulfur are heated, under ordinary conditions, to high temperatures to effect chemical combination thereof, an unsatisfactory product is often obtained due to the poor physical appearance, darkened color, and the tendency toward sludge deposition of the finished product.

An important object of the present invention is to provide a simple and practical manner of introducing the desired amount of sulfur into any oil.

Another important object of the invention is to produce a high grade sulfur-containing lubricant which is stable and free from sludge-forming tendencies.

Another object of the invention is to produce a high grade sulfur cutting oil free from sludge and light in color.

A further object of the present invention is to provide an improved method of introducing sulfur into lubricants, such as mineral lubricating oils, by the use of cyclic unsaturated compounds or compounds of the nature of pine oil, such as terpenes, terpineols, pine oil, pine tars, etc.

The incorporation of sulfur into mineral oils through the agency of terpene compounds has been known heretofore and according to the prior art, so far as I am aware, this has been done by heating the terpenes and sulfur under atmospheric pressure to dissolve sulfur in the terpene to form a sulfur base which is then admixed with the lubricating oil. The sulfur base thus prepared is apparently a solution of sulfur in the terpene, stable only at higher temperatures and on cooling to room temperatures the sulfur will settle from a concentrated solution thereof; but, when the base is mixed with a mineral oil in small amounts, the terpene acts sufficiently as a solvent-aid to effect solution of the sulfur in the resulting mixture of base and lubricating oil. Sulfurized oils, however, prepared according to the above method, have the disadvantage of being relatively unstable due to the free sulfur content and the tendency, particularly at low temperatures or in service, to precipitate free sulfur

or sludge unless large amounts of terpenes are used.

In accordance with the present invention instead of incorporating the sulfur into the terpene-type of compound under atmospheric pressure I maintain a substantial superatmospheric pressure on the terpene and sulfur during the sulfurizing operation. I have found that by maintaining a superatmospheric pressure on the terpene and sulfur undergoing reaction, much higher temperatures, even above the boiling point of the sulfur or terpene, may be employed, and the time of digestion regulated to any time desired without substantial loss of the reagents. It has been found also that a much improved product may be obtained, according to my invention, in that the sulfur apparently exists essentially in a chemically combined state, and accordingly large quantities thereof may be combined with terpenes in a highly stable form. The sulfur base so prepared may be blended in a concentrated form and in any proportions with a mineral oil to give a sulfur-containing oil which is of good physical appearance, color, and highly stable.

More particularly, according to the invention, a suitable unsaturated cyclic compound such as terpene hydrocarbons or their derivatives, for example, terpenes, terpineols, pine tars, etc., are reacted with sulfur. Pine oil or a material of that nature is particularly suitable and I prefer to use, especially for the manufacture of light colored cutting oils, a light pine oil, such as steam distilled "Yarmor" pine oil, having an iodine value of around 100-125. The pine oil is mixed with about 5-30% of sulfur and the mixture heated to a temperature in the region of 300°-500° F. for about 1-5 hours under a superatmospheric pressure of around 50-500 lbs. per sq. in. For a light pine oil I have found that the optimum conditions are a temperature of about 400° F., a digestion period of about 1-2 hours and a superatmospheric pressure of around 100-150 lbs. per sq. in. It will be understood, of course, that these conditions may vary with different oils but in any case I prefer to use a temperature of at least 300° F. and a superatmospheric pressure of 50 lbs. per sq. in. or more.

The pressure may be conveniently generated by merely confining the vapors evolved. It has been found that a copious evolution of hydrogen sulfide occurs and by confining the gases and vapors a self-generated pressure of as much as 425 lbs. or more may be obtained at temperatures around 400° F., and even higher pressures are possible at more elevated temperatures. In case insufficient

pressure is generated, an extraneous gas pressure, say of natural gas, hydrogen sulfide, or nitrogen, may be applied.

5 The pine oil sulfur base, thus prepared, is a bright reddish oil, miscible with hydrocarbon oils in all proportions and having a sulfur content ranging from 5-25% or over. It may be admixed with the desired lubricant in suitable proportions. For a cutting oil I prefer to use a quantity sufficient to provide from 1-10% or more of sulfur
10 in the resulting mixture.

In case the sulfur base has an objectionable odor, for instance, due to dissolved hydrogen sulfide, a vacuum may be applied to the oil or a gas such as air bubbled therethrough to carry out the volatile materials of bad odor. The objectionable odor may also be removed, if desired, in substantially the same manner after the sulfur base has been blended with the lubricating oil.

20 The invention will now be described in connection with the practical application thereof to the preparation of a sulfur cutting oil, although the invention is not limited thereto.

25 Thirty pounds of steam distilled "Yarmor" pine oil having an iodine value of 110, an A. P. I. gravity of 19.5° and a sulfur content of 0.11% were heated with 10 lbs. of powdered sulfur in a fire-heated pressure kettle at 400° F. for 2 hours. Gases and vapors were released sufficiently to
30 maintain a gauge pressure of about 100 lbs. per sq. in. The materials were found to be completely reacted and neither free sulfur nor sludge was present. The resulting product was reddish brown in color, clear, and tested 22.1% sulfur and 35 1.1° A. P. I. gravity. One part of the resulting sulfur base was blended at room temperatures with three parts of a pale lubricating oil having a Saybolt viscosity of 70 seconds at 100° F. The blend tested 5.77% sulfur and was light red in color.
40 clear, free from sludge, exhibited extreme pressure lubrication characteristics and proved highly satisfactory as a cutting oil.

Instead of 70 viscosity pale oil other pale oils

from 100-200 seconds Saybolt universal at 100° F. may be used for preparing cutting oils. It is also contemplated that the sulfur may be added during the digestion operation or after the oil has reached a reaction temperature instead of first mixing the oil and sulfur and then heating the mixture.

While the above described specific embodiment of the invention has to do with cutting oils the invention is by no means so limited. It is contemplated that heavier oils, such as motor oils or gear lubricants with viscosities ranging from 200 seconds at 100° F. to 250 seconds at 210° F. Saybolt universal may be sulfurized by my method. In some cases the invention may also find valuable application for the incorporation of sulfur into greases.

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. A sulfur base, soluble in hydrocarbon oils, consisting essentially of pine oil and sulfur, the latter present in an amount ranging between 5 and 25% and incorporated in the oil under superatmospheric pressure, said base having an improved odor and precipitating less sulfur on storage and on cooling than a base containing sulfur incorporated therein under atmospheric pressure.

2. A sulfur base, soluble in mineral lubricating oils, consisting essentially of pine oil and sulfur, the latter present to the extent of about 20% and incorporated in the oil under superatmospheric pressure, said base having an improved odor and precipitating less sulfur on storage and cooling than a base containing sulfur incorporated therein under atmospheric pressure.

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