ABSTRACT OF THE DISCLOSURE

Sulfur-containing oil-soluble extreme pressure additives for lubricants are produced by reacting one part by volume of sulfurized low molecular weight polybutenes and 0.5 to 1.5 parts by volume of liquid triglycerides susceptible to sulfuration at a temperature of about 250-400° F. for a period of at least 5 minutes.

This invention relates to an improved sulfur-containing lubricant additive suitable for use in preparing lubricating oil compositions having improved extreme pressure properties for metalworking purposes.

Mineral lubricating oils containing sulfurized additive agents have been extensively used as metalworking lubricants and especially as cutting oils. In recent years, it has been found that the mineral oil component of such lubricants contain constituents that may be toxic or carcinogenic in nature and therefore undesirable due to the potential harm that might occur when brought in contact with the human body. It has been found that the polyyclic aromatic constituents naturally present in most mineral lubricating oils are the undesirable constituents. Accordingly, it is necessary to remove these constituents from the oil in order to have a mineral lubricating oil suitable for the preparation of metalworking oils that are not harmful to the human body. The polyyclic aromatics are highly polar and can be removed by suitable solvent extraction methods well known to the art.

Naturally occurring paraffinic or naphthenic petroleum oils or solvent extracted oils contain less than about 1 percent carbon in aromatic bonding, as determined by the n-d-M Method used for determining the hydrocarbon type analysis of lubricating oils, have been found to be relatively free of toxicity and carcinogenicity characteristics. A description of the n-d-M Method may be found on p. 226 of "Physical Chemistry of Lubricating Oils" by A. Bondi, Reinhold Publishing Corp., New York, 1951. Such oils containing minor amounts of the polar aromatic constituents are relatively poor solvents for organic sulfur compounds. Such oils are now used as the principal component of commercial metalworking lubricants.

Conventional sulfurized liquid triglycerides, such as the naturally occurring fatty oils of animal, vegetable, or mineral origin, are well-known additives for use in the preparation of mineral oil-based metalworking lubricants. Due to the change of the mineral oil used as the base for making compounded metalworking lubricants and the lack of solvency characteristics thereof, it is not possible to form satisfactory oil blends of the mineral oil and the sulfurized fatty oils so as to incorporate sulfur in amounts greater than about one weight percent of sulfur. In other words, the prior art sulfurized fatty oils are substantially insoluble in the aforesaid mineral oils containing minor amounts of polar aromatic constituents. The use of the sulfurized fatty oils are desirable for imparting additional lubricity to the mineral oil metalworking fluids.

It has been found, in accordance with the present invention, that the desirable properties of the liquid triglycerides can be obtained by reacting the liquid triglyceride in volume proportions of about 0.5-1.5 parts by volume per volume of sulfurized low molecular weight polybutenes having a viscosity of about 60-150 SSU at 100° F., a sulfur content of about 12-20 weight percent, a minimum flash point, COC, of about 220° F., a maximum ASTM D-1500 color of about 8, and a gravity of about 18-28° API at temperatures in the range of about 250-400° F. for a period of at least 4 hours.

No reaction was found in contrast to similar blends containing a sulfur-containing reaction product that is soluble in mineral oils having poor solvency characteristics. While any liquid triglyceride susceptible to sulfuration can be used to form the desirable oil-soluble sulfur addition agent, lard oil is preferred.

The aforesaid reaction product provides sulfur that may be very active or relatively inactive so as to provide the desired sulfur activity in the blended oil for light or heavy duty applications. When sulfur is present in a form that readily stains copper, it is called active sulfur. The sulfur activity in the additive of this invention can be varied by the temperature and time of reaction. At lower temperatures and times the sulfur is in an extremely active form; and when the reaction is carried out at the higher temperatures and longer periods of time, the activity of the sulfur is reduced. Sulfur activity is determined by heating the blended oil in the presence of copper turnings at 350° F. for 20 hours. The residual sulfur content after the heating period is inactive sulfur, and the difference between the residual sulfur and the initial sulfur content represents active sulfur.

Preparation of the sulfurized polybutene reactant is effected in accordance with conventional sulfurizing techniques with elemental sulfur. Inasmuch as the sulfur content of the sulfurized product is desired so as to minimize darkening of the base oil to which it is added, it has been found that polybutenes having average molecular weight of 300 or less are preferable. The polybutenes are prepared by known polymerization techniques for polymerizing butene or isobutene feedstocks. The preferred polybutenes used in the present invention are a mixture of butene dimers, trimers, tetramers, or pentamers, i.e. mixture of butene polymers containing 8 to 20 carbon atoms in the polymer chain, obtained as by-product from the polymerization of butene for the preparation of higher molecular weight commercial polybutenes. After sulfurization reaction is completed, the reaction mixture is stripped to remove unreacted polybutenes and then filtered to remove unreacted sulfur.

It has been found that steam stripping of the reaction mixture of the polymer and liquid triglyceride substantially improves the color and odor properties of the sulfurized additive of this invention.

The reaction of a mixture containing seven percent sulfur formed by admixing 45 parts of the sulfurized polybutene and 55 parts (by volume) of lard oil at 325° F. for one hour, followed by stripping to remove volatile by-products formed a sulfurized additive containing about 50 percent active sulfur.

The effectiveness of the additive concentrate of this invention in blended metalworking oils, and to obtain the requisite load-bearing or extreme pressure properties of such blended oils, is determined by the active sulfur content of the additive. It has been found that at least one weight percent of the total sulfur in the additive should be in the active form.

A series of cutting oil blends containing solvent extracted SAE 10 grade mineral oil and various levels of the aforesaid sulfurized polyurethane-lard oil reaction product were prepared to provide sulfur contents ranging from one to three percent in the blends. These blends were subjected to temperature cycles between 0 and 40° F. to determine whether separation of the sulfurized material would occur. No separation was found in contrast to similar blends containing conventional sulfurized lard oil.
having equivalent sulfur content, which blends immediately separated. Accordingly, this invention provides a satisfactory sulfurized additive containing fatty oils that are completely soluble in solvent extracted mineral oils that can be used to form satisfactory metalworking lubricants.

We claim:

1. The oil-soluble sulfur-containing reaction product produced by the process of reacting (A) sulfurized low molecular weight polybutenes having a viscosity of about 60-150 SSU at 100°F, a sulfur content of about 12-20 weight percent, a minimum flash point, COC, of about 220°F, a maximum ASTM D-1500 color of about 8, and a gravity of about 18-28° API; and (B) from about 0.5 to about 1.5 parts by volume per part by volume of reactant A of liquid triglyceride susceptible to sulfurization at a temperature in the range of about 250-400°F, for a period of at least five minutes.

2. The product of claim 1 wherein reactant B is lard oil.
3. The product of claim 1 containing at least 1.0 weight percent active sulfur.
4. The product of claim 2 wherein said polybutenes are mixtures of butene polymers containing 8 to 20 carbon atoms in the polymer chain.

References Cited
UNITED STATES PATENTS
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