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CUTTING OIL COMPOSITION

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The present invention relates to a cutting oil concentrate and to cutting oils containing such a concentrate characterized by a unique combination of superior cutting ability and absence of the formation of disagreeable odors when in use.

Oils containing sulfur or sulfur compounds dissolved therein have been suggested heretofore as cutting oils. Such oils also frequently contain chlorine and a fat such as lard oil. It is believed that the sulfur, to be effective in improving the cutting ability of a cutting oil, must be in an active form, i. e., not bound to a carbon atom, which can react with metal being machined under the conditions existing at the point of contact between the tool and the machined metal. Heretofore, the amount of active sulfur which could be incorporated into an oil has been dependent largely upon the solubility of free sulfur in the oil and upon the amount of sulfur which can be chemically combined but not bound to a carbon atom.

A great many suggestions have been made heretofore for increasing the active sulfur content of cutting oils. In some instances, these suggestions have resulted in oils having excellent cutting ability but poor odor characteristics and/or stability. In other instances, they have resulted in oils of acceptable odor but of poor cutting ability. In still other instances, the oils developed have been found to be too dark or opaque to permit adequate viewing of the work during a machining operation. Generally it has been found that some compromise must be made among odor, stability, optical density and cutting ability factors, the cutting ability being improved with an increase in sulfur content at the expense, however, of odor, stability and opacity. To the best of applicants' knowledge, no cutting oil having optimum cutting ability and odor has ever been developed heretofore.

The odor characteristics of a cutting oil are not necessarily apparent upon an examination of the oil as produced or supplied to a user. In many instances, odors are developed only when the oil is used in machining operations. This is apparently due to the formation of odoriferous compounds under the conditions prevailing at the cutting tool. If the cutting oil develops an unpleasant or obnoxious odor during use, it makes no difference how good the oil is from the standpoint of cutting ability, i. e., the number of pieces that can be machined with a given tool, the wear on the tool and the surface finish of the metal being machined because machinists will refuse to work with it.

Hydrocarbon polysulfides containing one or more alkyl or aryl radicals have been suggested as being suitable for addition to lubricating oils in order to produce cutting oils. Tests have shown that some of this class of hydrocarbon polysulfides, when added to lubricating oils, form compositions having acceptable cutting abilities. Unfortunately, however, most of these hydrocarbon polysulfides, as such and when diluted or blended with a mineral

cutting oil base, have obnoxious odors that immediately disqualify them as cutting oil concentrates. Thus, for example, diamyl tetrasulfide has an exceedingly unpleasant odor that is characteristic of the polysulfide itself rather than of a mercaptan impurity, since it cannot be altered by caustic washing, and diallyl tetrasulfide which, because of its light yellow color, ready miscibility with mineral oil and very high sulfur content appeared very promising, was found to have a nauseating garlic odor. The few polysulfides in this group that do not possess such odors initially, almost without exception develop odors, upon use, that are or soon become so obnoxious as to cause machinists to walk off the job. In some instances the odors developed during use do not seem too unpleasant at first but for some reason become intolerable upon continued use due to a subtle accumulation of odors in high speed, automatic, multi-spindle machines, because of the large volumes of oil they require and the heat, violent agitation and splashing to which the oil is subjected.

The surprising discovery has now been made that dimethylbenzyl polysulfide, having the formula

$$[(CH_3)_2C_6H_3—CH_2]_2S_x$$

wherein x is a number greater than 2, has both cutting and odor properties that meet the extremely rigorous requirements long sought to be satisfied in a cutting oil. It meets these requirements whether it is used as such, in a highly concentrated form, or as an additive to a sulfurized mineral cutting oil base. It forms cutting oils that are superior to any available on the market and which, unlike cutting oils containing other hydrocarbon polysulfides, develop no disagreeable or offensive odors in use under even the most trying conditions.

Dimethylbenzyl polysulfide may be prepared in any one of a number of ways that may easily be developed by chemists skilled in the art. We have found that is is desirable to prepare the dimethylbenzyl polysulfide by mixing commercial grade dimethylbenzyl chloride, believed to be a mixture of the various isomers of

$$(CH_3)_2C_6H_3CH_2Cl$$

with an excess of sodium tetrasulfide in the form of an aqueous solution and then agitating the reactants at an elevated temperature of the order of 200° F. for several hours. Upon cooling and settling, the reaction mixture forms an upper organic phase and a lower aqueous phase. The lower aqueous phase is separated and the remainder, i. e., the upper organic phase, is washed with a dilute alkali hydroxide solution by stirring at room temperature. Upon separation of the alkali hydroxide solution, high yields of dimethylbenzyl polysulfide are obtained.

We have found that it is particularly desirable to carry out the reaction between dimethylbenzyl chloride and sodium tetrasulfide solution in the presence of a diluent having a low viscosity and a high flash point or to add such a diluent after the reaction has been completed. One such diluent that has been found particularly suitable is diesel fuel oil. The addition of this hydrocarbon, which does not react with either the reactants or the product, is preferred because a mixture thereof with dimethylbenzyl polysulfide has a lower viscosity than dimethylbenzyl polysulfide itself, facilitates handling of the product and aids in the separation of the aqueous phase after the reaction.

The treatment of the reaction product with dilute alkali hydroxide solution is carried out to wash out mercaptans, hydrogen sulfide and any remaining chlorides. It is desirable to use dilute rather than concentrated alkali hydroxide solutions to avoid extracting appreciable quantities of sulfur. Aqueous sodium hydroxide solu-

tions having concentration below about 10% are preferred.

It is also desirable to after-treat the washed reaction product with a mineral adsorbent such as Super Filtrol in order to brighten the product, and thereby make it more desirable from an aesthetic point of view.

The dimethylbenzyl polysulfide, diluted or not with a suitable diluent such as diesel fuel, may be blended with any mineral cutting oil base, preferably an acid-treated oil having a viscosity between about 75 and 300 SSU at 100° F. and containing free sulfur dissolved therein. The lower limit of viscosity specified is imposed largely by sulfur solubility. Oils having viscosities higher than 300 SSU at 100° F. are not preferred because of difficulties of handling and flowing. Blends containing as little as 1% by weight of dimethylbenzyl polysulfide have excellent cutting ability and concentrations of dimethylbenzyl polysulfide as high as 60% or higher in diesel fuel, without being blended with acid-treated oil, have superior cutting ability and excellent odor characteristics. For ordinary cutting operations, sulfurized oil blends containing between about 2 and 5% by weight of the concentrate are highly satisfactory.

It is to be understood that, while the products of this invention consist essentially of dimethylbenzyl polysulfide, preferably diluted with unsulfurized diesel fuel or blended with a mineral cutting oil base or both, conventional additives such as lard oil, graphite, chlorinated wax and the like may be added in amounts that do not materially alter the character of the products.

It is particularly surprising that cutting oils containing dimethylbenzyl polysulfide possess appreciably higher cutting ability and completely different odor forming characteristics than cutting oils containing such closely related compounds as benzyl polysulfide and monomethylbenzyl polysulfide. The advantages and utility of the cutting oil of this invention, particularly as compared with the properties of cutting oils containing benzyl polysulfide and monomethylbenzyl polysulfide, as well as with the best cutting oils on the market will become more apparent from the following detailed description in the examples.

EXAMPLE 1

27 parts by weight of dimethylbenzyl chloride were admixed with 52 parts by weight of a 40% aqueous solution of sodium tetrasulfide (approximately a 30% excess of the tetrasulfide to insure complete reaction of the chloride) and with 21 parts by weight of a diesel fuel having a viscosity of 34 SSU at 100° F. and a flash point of 180° F. The mixture was agitated at 200° F. for from 2 to 4 hours. During the reaction, the refractive index at 25° C. of the organic phase increased from a little below 1.50 to about 1.55 and the density of the organic phase increased from 0.94 to nearly 1.00. The mixture was then cooled and allowed to settle. The lower aqueous phase was separated and the remaining organic phase was treated with one part by volume of 3% aqueous sodium hydroxide solution per 5 parts by volume of organic phase and the mixture was stirred at room temperature for 30 minutes, the caustic then being separated. Thereupon 3% Super Filtrol was added and the mixture stirred for 10 minutes. The mixture was then filtered to yield 94 to 98% of the theoretical yield of dimethylbenzyl polysulfide in the diesel fuel.

The product was a 60/40 mixture of dimethylbenzyl polysulfide and diesel fuel having a total active sulfur content of 9 to 9.5% and a total sulfur content of 19 to 20%. Since the dimethylbenzyl polysulfide concentrate amounted to 60% of this product, the dimethylbenzyl polysulfide had an active sulfur content of 15 to 15.8% and a total sulfur content of 32 to 33%.

EXAMPLE 2

A cutting oil base was prepared by adding to Diamond Paraffin oil, an acid-treated lubricating oil stock, having an SSU of 100 at 100° F., 0.8% by weight elemental sulfur, i. e. the maximum amount of sulfur soluble in the oil at 0° C. The mixture was heated and stirred at about 200° F. until all of the sulfur was dissolved. This took approximately 1 to 2 hours.

Four cutting oils were prepared from this cutting oil base and compared with one another and with a commercial cutting oil for cutting ability in a thread cutting operation. One cutting oil, referred to in the table below as "Cutting Oil No. 1," was made by blending in 5% by weight of the 60/40 dimethylbenzyl polysulfide-diesel fuel concentrate prepared as described in Example 1; another, referred to in the table below as "Cutting Oil No. 2," was made by blending in 3% by weight of benzyl polysulfide and 0.5% by weight of "Anglamol 40," a chlorinated wax containing 40% chlorine; another, referred to in the table below as "Cutting Oil No. 3," was made by blending in 3% by weight of monomethylbenzyl polysulfide; and the fourth cutting oil, referred to in the table below as "Cutting Oil No. 4," was made by blending in 5% by weight of a 60/40 monoethylbenzyl polysulfide-diesel fuel concentrate. The commercial cutting oil, referred to as such in the table below, is a sulfurized mineral oil containing about 3% total sulfur and 4% lard oil. The commercial cutting oil, incidentally, is the best oil available on the market for ductile steel machining.

The monomethylbenzyl polysulfide for "Cutting Oil No. 3" was prepared by reacting 40 lbs. of methylbenzyl chloride with 78 lbs. of a 40% aqueous solution of sodium tetrasulfide under the conditions described in Example 1 with reference to the preparation of dimethylbenzyl polysulfide, no diesel fuel being added however because the viscosity of monomethylbenzyl polysulfide is low enough to permit facile separation of the organic and aqueous phases. The monoethylbenzyl polysulfide for "Cutting Oil No. 4" was prepared by reacting 46 lbs. of ethylbenzyl chloride with 82 lbs. of a 40% aqueous solution of sodium tetrasulfide by agitating the reactants for six hours at 190 to 200° F. After cooling, 25 lbs. of diesel fuel were added to reduce the viscosity of the product. After stirring for one-half hour, the product was allowed to settle for another half hour, the aqueous phase was removed and the organic phase was treated with caustic, washed and filtered.

The five cutting oils were subjected to tests on ductile steel forgings in a thread cutting operation, 11/16" diameter, 18 pitch, 2¼" long at 27 surface feet/minute. The finish was taken as the criterion of tool life, which was considered at an end when either tearing of the thread flang and crest or chip welding at the root of the chaser land occurred. The results are tabulated in Table I immediately below:

Table I

Oil	Percent Active Sulfur	Average No. of Pieces
No. 1 (Dimethylbenzyl)	1.3	3,016
No. 2 (Benzyl)	1.3	2,275
No. 3 (Monomethylbenzyl)	1.3	1,300
No. 4 (Monoethylbenzyl)	1.3	1,467
No. 5 (Commercial)	1.6	1,500

These results show that for ductile steel machining, the cutting oil of this invention (No. 1) is far superior, and in all but one instance more than twice as good as the remaining oils tested, including the best cutting oil (commercial) available on the market for such machining and the cutting oil containing monomethylbenzyl polysulfide (No. 3). The cutting oil containing benzyl polysufide as additive was found to be unsatisfactory because it developed an obnoxious and permeating odor during use. The character of this odor is more fully described in Example 4 of this application. These results are surprising because the lesser cutting ability of the monomethylbenzyl polysulfide-containing oil as compared with the cut-

ting ability of the benzyl polysulfide-containing oil would normally lead one to the conclusion that a cutting oil containing dimethylbenzyl polysulfide would have even less cutting ability than the monomethylbenzyl polysulfide-containing oil.

EXAMPLE 3

A cutting oil, referred to herein as "Cutting Oil No. 5," prepared by blending 2% by weight of the dimethylbenzyl polysulfide of Example 1 and 2% by weight of lard oil with the sulfurized base oil described in Example 2, was compared with sulfurized mineral oil containing 6% lard oil, referred to herein as "Commercial Oil," an oil available on the market and designed especially for use with semi-abrasive steels in a series of tests involving the manufacture of a differential side pinion gear blank from semi-abrasive steel (A3130). The machining was done at 110 surface feet/minute on an Acme Gridley 6-spindle automatic screw machine. The operation involved two rough and two finish formings, two drills, one counterbore, chamfering, and cut-off. The average lives of the wide-form roughforming tools, the most perishable tools in the operation, were compared. The results are tabulated below:

Table II

Oil	Average Tool Life
Commercial Oil	3,100
Cutting Oil No. 5	3,500

These results show that dimethylbenzyl polysulfide, even when present in concentrations as low as 2%, imparts, a cutting ability to a cutting oil base that compares favorably and is in fact superior to that of a commercial cutting oil designed specifically for use with semi-abrasive steels.

EXAMPLE 4

Diamond Paraffin oil containing 0.8% sulphur and 2% dimethylbenzyl polysulfide was compared with Diamond Paraffin oil containing 0.8% sulfur and 2% benzl polysulfide in a field test on a multi-spindle automatic screw machine, in forming, threading, drilling, chamfering, counterboring and cut-off operations. The dimethylbenzyl polysulfide-containing oil was in service for approximately 7 days, and the night and day shift operators as well as the foreman of the department and other adjacent machine tool operators reported that the odor was not objectionable.

The benzyl polysulfide-containing oil was used in the same machine and under the same conditions. After the third day, the oil could be smelled 150 feet away from the plant and the plant was completely permeated with the odor. Observations indicated that if all the machines in the plant had been operated with this oil, the personnel of the plant would have refused to work and the odor would have become intolerable in the general neighborhood of the plant.

EXAMPLE 5

The concentrate prepared as described in Example 1 was used full strength, i. e., 60% dimethylbenzyl polysulfide and the remainder unsulfurized diesel fuel, in the threading of ½" nuts of type 347 stainless steel. It was found possible to thread from 2000 to 8000 pieces, whereas none of the best conventional type cutting oils available on the market made it possible to thread more than 10.

This example shows that dimethylbenzyl polysulfide is suitable for use, especially where the work piece is almost as hard as the tool, in concentrations as high as 60%. Higher concentrations up to 100% are possible by omitting or reducing the amount of diluent. The only disadvantage of such an omission or reduction is that the product is more viscous and less easy to handle.

EXAMPLE 6

Field tests were carried out with the sulfurized cutting oil base of Example 2 containing 2 and 3% dimethylbenzyl polysulfide concentrate. In these tests, these oils were used in threading ½" galvanized steel pipe at a speed of 148 surface feet/minute on an automatic machine. It was found that the cutting oil containing a 2% concentration made it possible to thread 1710 pieces on the average and that the oil containing a 3% concentration permitted 1872 pieces on the average to be threaded.

Further field tests were carried out to compare the suitability of the sulfurized cutting oil base of Example 2 containing 3% dimethylbenzyl polysulfide concentrate with a commercial cutting oil that consists of a sulfurized base stock containing 1.8% sulfur and 3% "Anglamol 40." In these tests, the oils were used in threading ¾" galvanized steel pipe at 35 surface feet/minute on an automatic machine. It was found that an average of 8155 pieces could be threaded with the dimethylbenzyl polysulfide-containing oil and only an average of 4000 pieces could be threaded with the commercial oil. It was also noticed during the test that the work pieces remained cooler when the dimethylbenzyl polysulfide-containing oil was used and that there was very little chip welding.

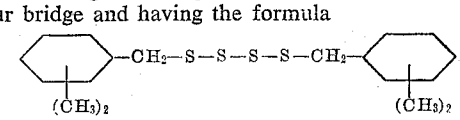
This example is believed to show that dimethylbenzyl polysulfide is not only suitable for use in concentrations as low as 2% and 3% or lower, but also that blends with a sulfurized cutting oil base containing such low concentrations are superior in thread cutting ability to one of the best commercial oils available on the market for that purpose.

Sodium tetrasulfide, used in the preparation of dimethylbenzyl polysulfide as described specifically in Example 1, is not a stable compound in the solid state. It is ordinarily available in the form of an aqueous solution prepared by adding to a sodium sulfide solution sufficient sulfur stoichiometrically to form sodium tetrasulfide. It is quite possible that the solute may actually be a mixture of sodium tri-, tetra-, and pentasulfides. The invention is applicable to the use of sodium polysulfides having various amounts of sulfur dissolved in sodium sulfide in excess the amount to form sodium disulfide. The term "dimethylbenzyl polysulfide" as used herein is intended to include any one, or a mixture, of dimethylbenzyl tri-, tetra- and pentasulfides.

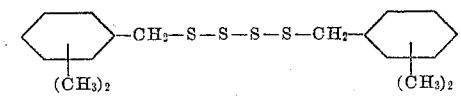
It is to be understood that all variations and modifications which will readily become apparent to those skilled in the art upon reading this description are intended to be included in the scope of this invention as defined in the accompanying claims.

We claim:

1. A cutting oil consisting essentially of an oil-soluble sulfurized mineral cutting oil base containing between 1 and about 60% by weight of dimethylbenzyl tetrasulfide containing an average of about 4 sulfur atoms in the sulfur bridge and having the formula

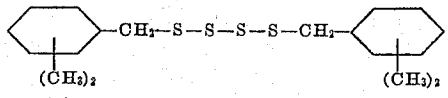


2. A cutting oil concentrate consisting essentially of a solution in an unsulfurized hydrocarbon diluent of at least about 60% by weight of dimethylbenzyl polysulfide containing an average of about 4 sulfur atoms in the sulfur bridge and having the formula

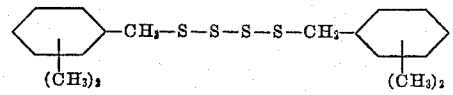


3. A cutting oil concentrate consisting essentially of a solution in an unsulfurized diesel fuel of at least about 60% by weight of dimethylbenzyl polysulfide contain-

ing an average of about 4 sulfur atoms in the sulfur bridge and having the formula

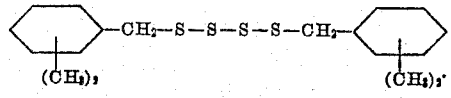


4. A cutting oil concentrate consisting essentially of about 60% by weight of dimethylbenzyl polysulfide containing an average of about 4 sulfur atoms in the sulfur bridge and having the formula

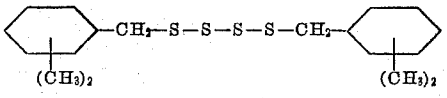


and 40% by weight of diesel fuel.

5. A cutting oil consisting essentially of a mineral cutting oil base containing up to about 0.8% free sulfur dissolved therein and from about 2 to about 5% by weight of dimethyl polysulfide containing an average of about 4 sulfur atoms in the sulfur bridge and having the following formula:



6. A cutting oil consisting essentially of a sulfurized mineral cutting oil base having a viscosity at 100° F. between about 75 and about 300 SSU and from about 2 to 5% by weight of dimethylbenzyl polysulfide containing an average of about 4 sulfur atoms in the sulfur bridge and having the following formula:



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