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LUBRICANT COMPOSITION COMPRISING
ORGANIC SULPHONAMIDES

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This invention relates to methods of lubrication and lubricants and particularly to lubricant compositions comprising solutions or dispersions of sulphonamides. More particularly it relates to lubricants comprising aqueous solutions or dispersions of sulphonamides. Still more particularly it relates to metal-working lubricants comprising aqueous solutions or dispersions of sulphonamides which contain halogen atoms and to methods of lubricating involving the use of such solutions. In a more limited sense it relates to metal-working lubricants comprising aqueous solutions or dispersions of aliphatic sulphonamides preferably containing chlorine atoms and to methods of lubrication involving the use of such compositions.

A number of materials have been proposed for lubricant compositions, particularly aqueous lubricant compositions. Thus, chlorinated and sulphurized oils have been used in the drawing, stamping and cuttings of metals, but the former have the serious disadvantage of decomposing during use with the evolution of corrosive gases, and the latter are deficient as regards lubricating and cooling action. The beneficial cooling effect of water has long been made use of in many drawing and stamping operations by the use of aqueous emulsions of fatty oils, etc. However, in certain operations, such as the deep drawing of sheet steel and cutting of tough metals at high speeds, aqueous lubricants have been found to possess insufficient film strength to withstand the pressures developed. It has been necessary to turn to the more expensive straight oils for such work, and to operate at reduced rates of speed in order to dissipate the heat developed.

This invention has for an object the preparation of stable aqueous lubricants of high film strength which overcome the above disadvantages. A further object is to provide a material which will reduce the friction between a metal being formed and the metal or other parts which contact with it during the drawing or stamping operation. A still further object is to provide a highly stable lubricant which will not decompose or separate under the conditions of shaping, forming or machining metals. A still further object is to provide a metal-working lubricant combining the important features of efficient cooling and high film strength.

The above and other objects are accomplished and the described disadvantages obviated by the following invention which comprises the use in lubricant compositions, and particularly in aqueous lubricant compositions for metal-forming and machining operations of aliphatic, cycloaliphatic, or aromatic sulphonamides or mixtures thereof.

In a preferred embodiment of the invention

they are accomplished by the use as lubricants for metal-forming, shaping and machining operations, of aqueous solutions or dispersions of sulphonamides which contain combined halogen, preferably chlorine.

In a more preferred embodiment of the invention they are accomplished by the use as lubricants of petroleum sulphonamides, e. g. sulphonamides obtained from petroleum sulphonyl chlorides from non-gaseous petroleum fractions.

In a still more preferred embodiment they are accomplished by the use of sulphonamides obtained from the sulphonyl chloride reaction products obtained by reacting non-gaseous aliphatic or cycloaliphatic hydrocarbons, and especially saturated hydrocarbons with admixed gaseous chlorine and gaseous sulphur dioxide after the manner set forth in Reed U. S. Patent No. 2,046,090.

When a non-gaseous aliphatic or cycloaliphatic hydrocarbon or a mixture of such hydrocarbons or predominating in such hydrocarbons is reacted with gaseous sulphur dioxide and gaseous chlorine, especially at a temperature below the decomposition point of the formed organic sulphonyl chlorides in the manner set forth in U. S. Patent 2,046,090, and Fox, Henke, Lockwood & Tinker application, Serial No. 216,838, filed June 30, 1938, a rather complex mixture is formed, which usually contains in varying proportions hydrocarbon monosulphonyl chlorides, chlorohydrocarbon monosulphonyl chloride, hydrocarbon polysulphonyl chlorides, chlorohydrocarbon polysulphonyl chlorides and chlorohydrocarbons. The proportion of products in the mixture and the amount of chlorine substitution products can be varied somewhat by varying the proportions of sulphur dioxide to chlorine, the rate of flow, the intensity of the actinic light used to accelerate the reaction, pressure, and temperature conditions.

In the case where the saturated open-chain hydrocarbons and derivatives thereof, e. g. non-gaseous n-alkanes, iso-alkanes, chloroalkanes and mineral oil fractions are used as initial reactants the mixture are unusually complex since the sulphonyl chloride groups do not enter the same position in each molecule. Hence, the resulting sulphonamides are quite complex when the entire sulphonyl chloride reaction product or a portion or fraction thereof is used for the amide reaction.

The reaction products obtained from using saturated cycloaliphatic hydrocarbons, e. g. cyclohexane, menthane, camphane, etc. as initial reactants are not quite as complex and the sulphonamides prepared therefrom can in some instances be separated as pure compounds.

Various methods for the preparation of the preferred sulphonamides used according to this

invention are described in A. L. Fox application, Serial No. 246,903. In general, they comprise reacting ammonia or a sufficiently reactive organic primary or secondary amine with the reaction products obtained by reacting a saturated hydrocarbon or a derivative thereof with gaseous sulphur dioxide and gaseous chlorine; the first-mentioned reaction being carried out under sulphonamide-forming conditions.

The lubricants of this invention may be prepared by incorporating the desired sulphonamide or mixture of sulphonamides in a suitable carrying medium such as water or an aqueous solution to form emulsions or dispersions. Other aqueous lubricant constituents may be incorporated if desired with the sulphonamides.

The primary and secondary sulphonamides which represent the preferred embodiments of the invention may be easily incorporated into water or aqueous solutions in the form of their alkali metal, e. g. sodium and potassium, salts, thus providing a solution suitable for use in the machining or forming of metals. Tertiary sulphonamides may also be used in neutral and alkaline solutions and dispersions, but it is sometimes advisable to use emulsifying and dispersing agents.

The invention will be further illustrated, but is not intended to be limited by the following examples in which the parts stated are parts by weight.

Example I

A solution containing 45 parts of a sulphonamide prepared by reacting a No. 40 viscosity paraffin base white oil* substantially free from unsaturates with a gaseous mixture of chlorine and sulphur dioxide and a further reaction of the sulphonyl chloride product with liquid ammonia, 3.6 parts of sodium hydroxide and 131.4 parts of water was tested for film strength in a Cornell (Faville-Levally) friction testing machine. This machine consists of a small steel rod held in position by a soft brass pin and rotating at a speed of about 325 R. P. M. against the flat surfaces of two fixed cylindrical test blocks which are set in frames attached to two lever arms. Increasing load is applied uniformly and automatically through the arms to these test pieces by means of a ratchet and screw operated from an eccentric on the drive shaft. Torque and load are registered simultaneously. Failure of the lubricating film is indicated by seizure and consequent shearing of the brass pin, or by a "squealing" with a sharp and persistent rise in torque although the bearing continues to turn without actual seizure. The solution of the viscosity 40 white oil sulphonamide stood a Cornell load of 3750 pounds, with a torque reading at this load of 46 pound inches.

Example II

An aqueous solution containing 45 g. of the sulphonamide of Example I and about 5 g. of sodium hydroxide dissolved in 130 parts of water was prepared. The solution was used as a lubricant on a machine drawing cases for rifle cartridges. The machine operated at a speed of about 200 cups per minute, drawing annealed low-carbon deep-drawing steel through high-carbon steel dies. The lubricant was fed by positive forced feed circulation. With the customary lubricant, failure was usually apparent after

200-300 draws, but using the sulphonamide lubricant 800 cups were drawn before definite scratching was noticed, and even after 1200 draws there was not a severe pick-up of metal on the dies. The drawn cups were also found to be quite resistant to corrosion, samples examined after about 6 months showing only slight rusting, although they had been stored without removal of the lubricant.

Example III

A solution containing 10 parts of the sodium salt of cetanesulphonamide (prepared by treatment of the reaction product of cetane, sulphur dioxide and chlorine with liquid ammonia in the manner described above) carried a load of 3500 pounds on the Cornell machine without failure.

Example IV

An aqueous solution containing 100 parts of water, 2.5 parts sodium hydroxide, and 25 parts of the sulphonamide obtained from paraffin wax sulphonyl chloride and liquid ammonia prepared by a procedure similar to that described above, when tested in the Cornell machine carried a load of 4250 pounds without failure, with a torque value of 26 pound inches.

Example V

An aqueous solution containing 25 parts of propanesulphonamide derived from propanesulphonyl chloride and liquid ammonia, and 100 parts of water, failed when tested in a Cornell machine after the manner of Example I at a load of 750 pounds, with a torque reading of 42 pound inches at this load.

Example VI

An aqueous solution containing 3 parts of sodium hydroxide, 100 parts of water, and 25 parts of the sulphonamide prepared from hexanesulphonyl chloride prepared according to U. S. Patent 2,046,090 and ammonia was tested on the Cornell machine after the manner set forth in Example I, when it was found to carry a load of 2000 pounds.

Example VII

An aqueous solution containing 10 parts of the sodium salt of cyclohexanesulphonamide and 100 parts of water was prepared. It withstood a Cornell load of 2750 pounds when tested in the manner set forth in Example I, but gave some evidence of rupture of the film at a load of about 1000 pounds.

Example VIII

An aqueous solution containing 5 parts of sodium hydroxide, 25 parts N-phenylcyclohexanesulphonamide, and 100 parts of water was prepared. It showed evidence of film rupture at 750 pounds load on the Cornell machine after the manner set forth in Example I, but a load of 1750 pounds was reached before complete failure was observed.

65	First drop-----	* C.
	10 cc-----	262-265
	50 cc-----	270-272
	90 cc-----	276-278
	95 cc-----	287-290
	Dry-----	291-292
		306-314
70	Sulbolt Universal viscosity at 100° F.—36-37 sec.	
	Specific gravity 15.5°/15.5° C.—0.8030-0.8035	
	Refractive index 20° C.—1.4437-1.4442	

*No. 40 white oil has the following analytical characteristics: A. S. T. M. distillation range:

In place of the sulphonamides described in the previous examples may be substituted various other aliphatic, cycloaliphatic, aromatic or mixed sulphonamides, especially those having at least 6

carbon atoms. Primary and secondary sulphonamides have the most utility and the primary sulphonamides are preferred. Additional suitable sulphonamides include n-heptane-, n-octane-, n-dodecane-, n-tetradecane-, n-hexadecane-, n-octadecane-, 2-ethylhexane-, menthane-, pinane-, camphane-, tetrahydronaphthalene-, decahydronaphthalene-, p-toluene-, and 2-naphthalene-sulphonamides. These compounds may be substantially pure or in admixture with other sulphonamides. They may be substituted with halogen, amino-nitro, alkoxy, carboxy or ester groups. The sulphonamides may be substituted upon the nitrogen atom, e. g. N-octadecylparatoluenesulphonamide, N, N-dimethylhexadecanesulphonamide, N-methyl-N-phenyl-dodecanesulphonamide, etc.

The above products as previously stated may be obtained from pure sulphonic acids or sulphonyl chlorides or from the more complex sulphonyl chloride products obtained by reacting non-gaseous saturated hydrocarbons with gaseous chlorine and gaseous sulphur dioxide; by an appropriate amide forming reaction. The sulphonyl chlorides obtained by reacting hydrogenated olefine polymers, e. g., di-, tri- or tetra-isobutylene, hydrogenated hexene-3-polymers, hydrogenated ethylene polymers, etc. with gaseous chlorine and gaseous sulphur dioxide are also useful as intermediates. The petroleum sulphonyl chlorides described in Bruson et al. 2,142,934 are useful as intermediates for sulphonamides. The sulphonyl halides prepared by the method described in Johnson U. S. Pats. 2,146,744 and 2,147,346 may be converted to sulphonamides which are useful in carrying out the invention. Additional useful sulphonamides are described in the above-identified case of A. L. Fox.

Among the amines which may be used for the amine component are primary and secondary mono-, di- and poly-aliphatic, aromatic and heterocyclic amines. Suitable specific amines include primary amines such as methyl-, ethyl-, normal- and isopropyl-, iso and secondary butyl-, isoamyl-, 2-ethylhexyl-, octyl-, dodecyl-, octadecyl-, etc. amines, ethylene-, propylene-, hexamethylene-, etc. diamines, aniline, toluidine, xyldine, anisidine, phenetidine, etc. and ethanolamine; secondary amines such as dimethyl-, diethyl-, diethyl-, etc. amines, mixed alkyl amines such as methylethylamine, methyloctylamine, etc., mixed aryl alkyl amines such as N-methyl- and N-ethyl-aniline, heterocyclic amines such as morpholine, piperidine, C-methylpiperidine, hexamethylenimine, etc., and diethanolamine.

The sulphonamides may be dispersed or emulsified in water by means of a suitable emulsifying agent, e. g. Turkey red oil, water-soluble alkali metal and amine salts of long chain aliphatic carboxylic acids such as sodium oleate, sodium stearate, triethanolamine oleate, amine and alkali metal salts of higher aliphatic alcohol sulphates, mineral oil sulphonic acids, sulphonated oils, alkyl naphthalene sulphonates, casein, glue, starch, dextrin, pectin, gum Arabic, gum ghatti and polyvinyl alcohol. Combinations of the surface active type with colloidal dispersing agents may be used.

The aqueous solutions and dispersions described herein are suitable for lubrication per se in applications which will permit of a low-boiling lubricant, or they may be used for improving the lubricating qualities of other aqueous solutions or emulsions, e. g., of metal-working lubricants. The liquid sulphonamides may be used as special

purpose oils, e. g., in the machining of tough metals.

Solutions or dispersions containing about 25 to about 50% of the sulphonamides have been found to be generally useful, but up to 80% of the sulphonamide may be used with success. In the case of aqueous lubricants used for light drawing operations, for example, for drawing readily workable metal such as brass, tin, or copper, for shaping operations where the deformation is not great, for shallow draws on light gauge metal, or for the lubrication of textile fibers, concentrations of 0.5 to 10% are very effective.

The lubricants described in the present invention are stable aqueous solutions or emulsions which show little tendency to separate in service. The solutions obtained from the primary and secondary sulphonamides are especially useful. The lubricants described possess excellent cooling action by virtue of their water content and in addition have greater film strength than it has heretofore been possible to obtain in such aqueous lubricants.

The compounds which contain chlorine possess high film strength. Since the compounds are preferably used in the form of their salts, or in an alkaline solution, they will not tend to become corrosive, as has been the case with chlorinated oils, which constitutes a distinct advantage. These materials possess the further advantage of a stably bound sulphur atom which cannot separate or form corrosive substances such as are often encountered in the use of sulphurized oils. Another advantage of the materials of the present invention is that they do not corrode metal, but in fact tend to prevent corrosion, and therefore aid in obtaining and maintaining bright finishes.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that we do not limit ourselves to the specific embodiments hereof except as defined by the appended claims.

I claim:

1. A lubricant composition comprising an aqueous carrying medium containing from 10% to 80% of a sulphonamide having an organic radical attached to the sulphur atoms.

2. A lubricant composition comprising an aqueous alkaline solution containing a sulphonamide having an organic radical attached to the sulphur atom.

3. A lubricant composition comprising an aqueous carrying medium containing from about 25% to 80% of a sulphonamide having an organic radical of at least 6 carbon atoms attached to the sulphur atom.

4. A lubricant composition comprising an aqueous alkaline solution containing a sulphonamide having an organic radical of at least 6 carbon atoms attached to the sulphur atom.

5. A metal-working lubricant composition comprising an aqueous carrying medium containing from 10% to 80% of a sulphonamide having a hydrocarbon radical of at least 6 carbon atoms attached to the sulphur atoms, said sulphonamide having at least one hydrogen atom attached to the nitrogen atom thereof.

6. A metal-working lubricant composition comprising an aqueous alkaline solution containing a sulphonamide having an organic radical of at least 6 carbon atoms attached to the sulphur atom.

7. A metal-working lubricant composition comprising an aqueous alkaline solution contain-

ing a sulphonamide having a chlorine-containing hydrocarbon radical of at least 6 carbon atoms attached to the sulphur atom.

8. A metal-working lubricant composition comprising an aqueous alkaline solution containing an aliphatic primary sulphonamide having a saturated hydrocarbon radical of at least six carbon atoms attached to the sulphur atom.

9. A metal-working lubricant composition comprising an aqueous carrying medium containing an aliphatic primary sulphonamide having at least six carbon atoms and containing combined chlorine attached to carbon.

10. A metal-working lubricant composition comprising an aqueous alkaline solution containing a sulphonamide obtained from the sulphonyl chlorides produced by reacting a non-gaseous saturated hydrocarbon with a gaseous mixture of chlorine and sulphur dioxide.

11. A metal-working lubricant composition comprising an alkaline aqueous solution containing a sulphonamide obtained from the sulphonyl chlorides produced by reacting a non-gaseous petroleum hydrocarbon with a gaseous mixture of chlorine and sulphur dioxide.

12. A lubricant composition comprising an aqueous alkali metal hydroxide solution containing 0.5 to 80% by weight of a cetane sulphonamide.

13. A lubricant composition comprising an aqueous alkali metal hydroxide solution containing 0.5 to 80% by weight of a mixture of aliphatic sulphonamides wherein the hydrocarbon radicals attached to the sulphur atoms correspond to the hydrocarbons present in petroleum white oils,

said hydrocarbon radicals containing chlorine attached to carbon atoms.

14. A lubricant composition comprising an aqueous alkali metal hydroxide solution containing 0.5 to 80% by weight of a mixture of aliphatic sulphonamides wherein the hydrocarbon radicals attached to the sulphur atoms correspond to the hydrocarbons present in a paraffin wax, said hydrocarbon radicals containing chlorine attached to carbon atoms.

15. A lubricant composition comprising an aqueous carrying medium containing from 10% to 80% of a sulphonamide having a hydrocarbon radical attached to the sulphur atom and an emulsifying agent.

16. A lubricant composition comprising an aqueous carrying medium containing as an essential lubricant thereof a sulphonamide having an organic radical attached to the sulphur atom and being further characterized in that the groups attached to the nitrogen contain hydrogen and carbon atoms only.

17. A lubricant composition comprising an aqueous carrying medium containing as an essential lubricant thereof an aliphatic sulphonamide having an aliphatic hydrocarbon radical of at least 6 carbon atoms attached to the sulphur atom.

18. A lubricant composition comprising an aqueous carrying medium containing as an essential lubricant thereof an aliphatic primary sulphonamide having an aliphatic hydrocarbon radical of at least 6 carbon atoms attached to the sulphur atom.

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