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LUBRICANT COMPOSITIONS CONTAINING ORGANO MERCAPTOALKYL BORATES

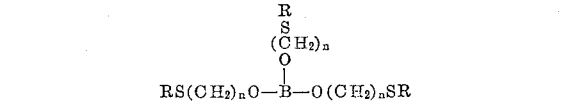
Frederick E. Scypinski, Monroeville, and Harold O. Strange, Penn Hills Township, Allegheny County, Pa., assignors to Gulf Research & Development Company, Pittsburgh, Pa., a corporation of Delaware

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This invention relates to an improved lubricant for use in internal combustion engines and more particularly to a lubricating composition having improved extreme pressure and antiwear characteristics.

In the design of modern engines, machinery, and the like, the engineer is constantly striving for improvements with respect to compactness, speed, power and acceleration. In achieving these improvements closer tolerances are being employed between adjacent moving surfaces. In order to prevent undue wear and perhaps even seizure of the adjacent surfaces, a lubricating composition is required which will form a lubricating film between the respective surfaces. The lubricating film must be able to withstand extremely high pressures without being squeezed from between the surfaces and without being decomposed.

We have discovered that a lubricating composition having improved antiwear and load-carrying characteristics can be obtained by incorporating into a lubricating oil a small amount of an organo mercaptoalkyl borate having the general formula



wherein R is selected from the group consisting of hydrogen, alkyl, aryl, alkaryl, aralkyl and cycloalkyl radicals containing 1 to 16 carbon atoms and *n* is an integer of 2 to 16, inclusive. The R substituents may be either alike or different. From the standpoint of ease in preparation, it is preferred to employ those compounds where the R groups are of the same kind.

Specific examples of the radicals which we intend to include with respect to "R" in the above formula are as follows: hydrogen; methyl; ethyl; propyl; isopropyl; n-butyl; sec-butyl; tertiary butyl; amyl; hexyl; heptyl; n-octyl; isooctyl; nonyl; decyl; undecyl; dodecyl; tridecyl; tetradecyl; pentadecyl; hexadecyl; phenyl; 1-naphthyl; 2-naphthyl; o-, m-, and p-tolyl; o-, m-, and p-xylyl; methylnaphthyl; dimethylnaphthyl; tri-, tetra-, penta-, and hexamethylnaphthyl; o-, m-, and p-ethylphenyl; o-, m-, and p-propylphenyl; o-, m-, and p-butylphenyl; o-, m-, and p-amylphenyl; o-, m-, and p-hexylphenyl; o-, m-, and p-heptylphenyl; o-, m-, and p-octylphenyl; o-, m-, and p-nonylphenyl; diethylphenyl; dipropylphenyl; dibutylphenyl; diamylphenyl; triethylphenyl; tripropylphenyl; tetraethylphenyl; pentaethylphenyl; benzyl; phenethyl; gamma-phenylpropyl; naphthylethyl; o-, m-, and p-methylbenzyl; o-, m-, and p-methylphenethyl; cyclopentyl; cyclohexyl; cyclooctyl; methylcyclohexyl; and the like.

The "organo" substituents of the organo mercaptoalkyl borates can be selected from a variety of the organic radicals mentioned above on the basis of their ability to impart oil-solubility to the compounds. Specific examples of some of the organo mercaptoalkyl borates represented by the above formula are

tri(mercaptoethyl)borate
tri(methylmercaptoethyl)borate
tri(ethylmercaptoethyl)borate
tri(propylmercaptoethyl)borate

tri(butylmercaptoethyl)borate
tri(hexylmercaptoethyl)borate
tri(octylmercaptoethyl)borate
tri(decylmercaptoethyl)borate
tri(dodecylmercaptoethyl)borate
tri(hexadecylmercaptoethyl)borate
tri(phenylmercaptoethyl)borate
tri(naphthylmercaptoethyl)borate
tri(tolylmercaptoethyl)borate
tri(ethylphenylmercaptoethyl)borate
tri(nonylphenylmercaptoethyl)borate
tri(benzylmercaptoethyl)borate
tri(cyclohexylmercaptoethyl)borate
di(mercaptoethyl) mono(methylmercaptoethyl)borate
di(mercaptoethyl) mono(ethylmercaptoethyl)borate
di(mercaptoethyl) mono(propylmercaptoethyl)borate
di(mercaptoethyl) mono(butylmercaptoethyl)borate
di(mercaptoethyl) mono(hexylmercaptoethyl)borate
di(mercaptoethyl) mono(octylmercaptoethyl)borate
di(mercaptoethyl) mono(dodecylmercaptoethyl)borate
di(mercaptoethyl) mono(hexadecylmercaptoethyl)borate
di(mercaptoethyl) mono(phenylmercaptoethyl)borate
di(mercaptoethyl) mono(tolylmercaptoethyl)borate
di(mercaptoethyl) mono(nonylphenylmercaptoethyl)-borate
di(mercaptoethyl) mono(benzylmercaptoethyl)borate
di(mercaptoethyl) mono(cyclohexylmercaptoethyl)-borate
mono(mercaptoethyl) di(methylmercaptoethyl)borate
mono(mercaptoethyl) di(ethylmercaptoethyl)borate
mono(mercaptoethyl) di(propylmercaptoethyl)borate
mono(mercaptoethyl) di(butylmercaptoethyl)borate
mono(mercaptoethyl) di(hexylmercaptoethyl)borate
mono(mercaptoethyl) di(octylmercaptoethyl)borate
mono(mercaptoethyl) di(dodecylmercaptoethyl)borate
mono(mercaptoethyl) di(hexadecylmercaptoethyl)borate
mono(mercaptoethyl) di(phenylmercaptoethyl)borate
mono(mercaptoethyl) di(tolylmercaptoethyl)borate
mono(mercaptoethyl) di(nonylphenylmercaptoethyl)-borate
mono(mercaptoethyl) di(benzylmercaptoethyl)borate
mono(mercaptoethyl) di(cyclohexylmercaptoethyl)-borate
tri(mercaptopropyl)borate
tri(methylmercaptopropyl)borate
tri(ethylmercaptopropyl)borate
tri(propylmercaptopropyl)borate
tri(butylmercaptopropyl)borate
tri(hexylmercaptopropyl)borate
tri(octylmercaptopropyl)borate
tri(decylmercaptopropyl)borate
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mono(mercaptobutyl) di(methylmercaptobutyl)borate
mono(mercaptobutyl) di(ethylmercaptobutyl)borate
mono(mercaptobutyl) di(propylmercaptobutyl)borate
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mono(mercaptobutyl) di(octylmercaptobutyl)borate
mono(mercaptobutyl) di(dodecylmercaptobutyl)borate
mono(mercaptobutyl) di(hexadecylmercaptobutyl)borate
mono(mercaptobutyl) di(phenylmercaptobutyl)borate
mono(mercaptobutyl) di(tolylmercaptobutyl)borate
mono(mercaptobutyl) di(nonylphenylmercaptobutyl) borate
mono(mercaptobutyl) di(benzylmercaptobutyl)borate
mono(mercaptobutyl) di(cyclohexylmercaptobutyl)borate
tri(mercaptoamyl)borate
tri(mercaptohexyl)borate
tri(mercaptoheptyl)borate
tri(mercaptooctyl)borate
tri(mercaptononyl)borate
tri(mercaptodecyl)borate
tri(mercaptoundecyl)borate
tri(mercaptododecyl)borate

tri(mercaptotridecyl)borate
tri(mercaptotetradecyl)borate
tri(mercaptopentadecyl)borate
tri(mercaptohexadecyl)borate

While all of the organo mercaptoalkyl borates mentioned hereinabove can be employed for improving the antiwear and load-carrying characteristics of an oil, it is not to be implied that all such compounds serve with equal efficiency. Those organo mercaptoalkyl borates having the general formula $B(OCH_2CH_2SR')_3$ wherein R' is hydrogen or an alkyl radical containing from 1 to 12 carbon atoms form a preferred group of compounds. Especially good results are obtained, for example, with tri(mercaptoethyl)borate and tri(dodecylmercaptoethyl) borate.

The amount of the organo mercaptoalkyl borate compound employed may vary over wide limits depending upon the particular compound employed, the type of base oil in which the compound is incorporated and the severity of the conditions to which the ultimate composition is subjected. In general, however, we use an amount between about 0.5 and 10 percent by weight based on the weight of the oil. Good results are obtained with about 1 to about 5 percent by weight of the organo mercaptoalkyl borate compound. In any event, the organo mercaptoalkyl borate is employed in an amount sufficient to improve the antiwear and extreme pressure characteristics of the oil to which the compound is added. When used in amounts of about 0.5 to about 10 percent by weight, the organo mercaptoalkyl borate also serves as an oxidation inhibitor.

The organo mercaptoalkyl borates can be prepared according to known chemical procedures. Neither the compounds per se nor their method of preparation constitutes any portion of this invention. The organo mercaptoalkyl borates, for example, can be prepared by reacting boric acid with one or more of the appropriate thioalcohols under reflux conditions in a suitable solvent such as a liquid hydrocrabon, e.g., benzene, toluene or the like. The reaction is continued until the theoretical amount of water is formed. Theoretically, complete reaction of the boric acid and alcohol requires 3 moles of the alcohol for each mole of boric acid. Thus, the reaction is theoretically complete when 3 moles of water are formed for each mole of boric acid used. When the theoretical amount of water is collected, the heating process is discontinued and the solvent evaporated from the product. No catalyst is required. The reaction of the boric acid and thioalcohol occurs readily at temperatures in the range of about 40° to about 200° C. The reaction is preferably conducted at atmospheric pressure. It will be recognized that when organo mercapotalkyl borates with the same organic radicals are desired, a single thioalcohol is employed. When organo mercaptoalkyl borates with different organic radicals are desired, two or more thioalcohols may be utilized separately or as mixtures thereof. In any event, the total amount of alcohol employed to effect esterification should be sufficient to replace the three hydrogen atoms of the boric acid. While we have disclosed a method of preparing the organo mercaptoalkyl borates by reacting boric acid with the appropriate thioalcohol or mixture of thioalcohols, other methods of preparing the organo mercaptoalkyl borates can be employed without deviating from the scope of the invention provided the resulting esters have the general formula disclosed hereinabove. Suitable thioalcohols for use as starting materials include organo mercaptoalkanols such as mercaptoethanol, methylmercaptoethanol, ethylmercaptoethanol, propylmercaptoethanol, butylmercaptoethanol, octylmercaptoethanol, dodecylmercaptoethanol, hexadecylmercaptoethanol, phenylmercaptoethanol, tolylmercaptoethanol, benzylmercaptoethanol, cyclohexylmercaptoethanol, mercaptopropanol, methylmercaptopropanol, mercaptobutanol, mercaptopentanol, mercaptohexanol, mercaptoheptanol,

mercaptooctanol, mercaptononanol, mercaptodecanol, mercaptododecanol, mercaptohexadecanol and the like.

The synthesis of organo mercaptoalkyl borates useful in compositions of the invention will be demonstrated by the following specific illustrative examples.

Example I

A mixture of 6.2 grams (0.1 mole) of boric acid, 23.4 grams (0.3 mole) of 2-mercaptoethanol and 100 ml. of benzene was refluxed at about 80° C. in a flask connected to a reflux condenser by means of a Dean-Stark trap. The water which formed during the reaction was collected as a benzene azeotrope. When 5.2 ml. of water was collected (theory is 5.4 ml.), the reaction was terminated and the benzene was stripped off under reduced pressure. The product thus obtained was a clear liquid weighing 23.4 grams (96% yield). Elemental analysis of the product shows it to compare favorably with the theoretical analysis of tri(2-mercaptoethyl)borate as follows:

Percent by weight for $B(OCH_2CH_2SH)_3$, Found: Carbon, 28.0; hydrogen, 6.1; sulfur, 37.5; boron, 4.4. Theoretical: Carbon, 29.8; hydrogen, 6.2; sulfur, 39.8; boron, 4.5.

Example II

A mixture of 76.2 grams (2.34 moles) of 2-n-dodecylmercaptoethanol, 48.8 grams (0.78 mole) of boric acid and one liter of toluene was refluxed at about 110° C. in a flask connected to a reflux condenser by means of a Dean-Stark trap. The water which formed during the reaction was collected as a toluene azeotrope. When 43 ml. of water was collected (theory is 42.2 ml.), the reaction was terminated, the solution filtered and the toluene removed under reduced pressure. The product thus obtained was a clear liquid which solidified slowly to a wax on standing. The product weighed 582 grams (99+% yield). Elemental analysis of the product shows it to compare favorably with the theoretical analysis of tri(2-dodecylmercaptoethyl)borate.

Percent by weight for $B(OCH_2CH_2SC_{12}H_{25})_3$, Found: Carbon, 65.91; hydrogen, 10.80; boron, 1.47. Theoretical: Carbon, 67.5; hydrogen, 11.65; boron, 1.47.

The lubricating oil to which the organo mercaptoalkyl borate is added to form a lubricating composition having improved antiwear and extreme pressure characteristics can be any oil of lubricating viscosity such as refined or semirefined paraffinic, naphthenic or asphalt base oil. Hydrotreated mineral oils, because of their improved stability over untreated oils are especially prefered lubricating bases for preparing lubricants to be used under moderately elevated temperatures. When temperatures in the order of 400° F. and above are to be encountered, synthetic oils of lubricating viscosity form a preferred class of lubricating oil bases to which the organo mercaptoalkyl borate is added. Thus, synthetic lubricating oils such as polymerized olefins, organic esters such as di-2-ethylhexyl sebacate and di-2-ethylhexyl azelate and the like can also be used as the base oil to form a lubricating composition in accordance with the invention. If desired, a blend of oils of suitable viscosity can be employed instead of a single oil, by means of which any desired viscosity can be secured.

The lubricating composition of our invention can contain minor amounts of other agents normally added to lubricating oils for a specific purpose such as a dispersant, a detergent, a pour point depressant, a thickener, a corrosion inhibitor, a viscosity index improver, an antifoamant, a rust inhibitor, a dye and the like. The lubricating composition can also contain oiliness and other extreme pressure agents and antioxidants to further enhance the lubricating characteristics if desired.

To prepare an improved lubricating composition the additives disclosed herein can be added to the lubricating oil base directly or in the form of an oil concentrate in an amount to give the desired concentration in the final lubricating composition. Slight heating of the mixture may be advantageously employed to facilitate blending of the additives in the composition.

To demonstrate the effectiveness of the organo mercaptoalkyl borates of the type described hereinabove, we have conducted comparative tests, the results of which are listed in Table I, with a mineral lubricating oil alone and with the same oil blended with representative organo mercaptoalkyl borates. The mineral oil employed had the following typical inspection:

Gravity, ° API	32.2
Viscosity, SUS Sec.:	
100° F.	153.0
210° F.	44.0
Flash OC, ° F.	440
Fire OC, ° F.	490

In illustrating the improved antiwear and load-carrying characteristics, tests were made using the four-ball wear test. In conducting this test, a four ball wear test machine was employed. In this machine, three steel balls are securely fastened in contact with each other in a horizontal plane in a cup. A fourth steel ball in a rotating spindle is placed so that it is entirely supported by the other three balls forming a pyramid. The test cup is placed on a stage which can move vertically to facilitate loading. The stage rests on a calibrated fulcrum so that specific weights may be applied to force the three balls in the cup to contact the rotating fourth ball at a predetermined pressure. The cup holding the three balls also contains the test lubricant at a level of 2 mm. above the stationary balls, thus assuring an adequate supply of lubricant at the contact points. A fixed oil temperature is maintained by a relay system connected to a thermocouple in the cup and a heater in the stage. The fourth ball can be rotated from a motor drive at 600, 1200 or 1800 r.p.m. Each test is run with new steel balls.

A test is run on a lubricant at a specific load, temperature, speed and time. Lubricating properties are evaluated from an examination of (1) the scars on the clean surfaces of the three stationary balls, the two maximum right angle diameters of each wear scar being measured to the closest 0.01 millimeter and averaged and (2) the load in kilograms at which seizure occurs. A more complete description of the machine and test method are given in the Naval Research Laboratory Report entitled "A Study of the Four Ball Wear Machine," by W. C. Clinton, NRL Report 3709, September 1950.

In illustrating the improved antiwear and load-carrying characteristics of compositions of the invention by the four ball wear test, the movable ball was rotated at 1800 r.p.m. for one hour while maintaining the test lubricant at 250° F. Separate one hour tests were conducted using new steel balls at loads of 10 to 50 kilograms unless seizure was encountered. At the end of each test, wear scars were measured. The advantageous wear properties of the oil containing an additive of the invention as compared with the base oil are illustrated by the data set forth in Table I.

TABLE I

Composition	Percent by Weight		
	A	B	C
SAE 10 Mineral oil	100	99.0	99.0
Tri(mercaptoethyl)borate (Example I)		1.0	
Tri(dodecylmercaptoethyl)borate (Example II)			1.0
Four-Ball Wear Test—Wear Scar Diameter, mm.:			
Load, kg.:			
10	0.67	0.46	0.50
30	[1] 1.75	0.60	0.62
40			0.73
50		0.8	0.77

[1] Seizure.

It is apparent from the data in Table I that a lubricating composition containing an organo mercaptoalkyl borate in accordance with the invention has substantially improved antiwear and load carrying characteristics as determined by the four-ball wear tests.

and acid number. The compositions of the invention (Compositions E and F) also show improved viscosity indices after the 48 hour test.

Specific examples of other compositions within the scope of the invention are set forth in Table III.

TABLE III

Composition	Percent by Weight					
	G	H	I	J	K	L
Lubricating Oil:						
Diisooctyl azelate	99.5			97		
Di(2-ethylhexyl)sebacate		99			95	
Mineral oil			98			90
Organo mercaptoalkyl borate:						
Tri(methylmercaptoethyl)borate	0.5					
Tri(nonylphenylmercaptoethyl)-borate		1				
Di(mercaptoethyl) mono(methyl-mercaptoethyl) borate			2			
Tri(phenylmercaptobutyl)borate				3		
Mono(mercaptoethyl) di(ethyl-mercaptoethyl)borate					5	
Tri(mercaptohexadecyl)borate						10

In addition to improved antiwear and load carrying characteristics, lubricating compositions of the invention also have improved resistance to oxidation. In order to demonstrate the effectiveness of the organomercaptoalkyl borates of the type described hereinabove as antioxidants in lubricating oils, we have conducted comparative oxidation tests with representative lubricating oils alone and with the same lubricating oils blended with representative organo mrcaptoalkyl borates. The oxidation test was conducted by bubbling air at a rate of one liter per hour through approximately 20 milliliters of test lubricant contained in a glass tube inserted into an aluminum block heated to 347° F. (175° C.). The duration of the test was 48 hours. The viscosity of the test lubricant and its acid number were determined at the beginning and the conclusion of each test. The percent increase in viscosity and the increase in acid number of the lubricant during the test is indicative of the oxidation stability of the test lubricant at a temperature of 347° F. under accelerated oxidizing conditions.

The results of comparative tests on a lubricating oil alone, and the same lubricating oil with an organo mercaptoalkyl borate are shown in Table II.

TABLE II

Composition	Percent by Weight		
	D	E	F
Diisooctyl azelate	100	99.0	99.0
Tri(mercaptoethyl)borate (Example I)		1.0	
Tri(dodecylmercaptoethyl)borate (Example II)			1.0
Oxidation Test (Air flow, 1 l/hr.):			
Viscosity, cs. at 100° F.:			
Initial	12.66	12.66	12.28
After 48-hour test	[1] 22.79	[1] 13.00	[1] 12.55
Increase	10.13	0.34	0.27
Percent Increase in Viscosity	80	2.7	2.2
Viscosity Index:			
Initial	166	153	163
After 48-hour test	[1] 142	[1] 168	[1] 165
Change in viscosity index	−24	+15	+2
Acid Number (ASTM D 974):			
Initial	0.7	0.07	0.06
After 48-hour test	[1] 28.68	[1] 1.82	[1] 1.12
Increase	28.61	1.75	1.06

[1] Average of 2 tests.

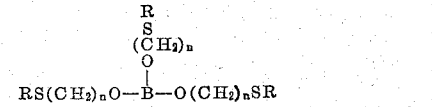
The data in Table II clearly show that a lubricating oil normally tending to undergo oxidational changes at an elevated temperature has improved resistance to oxidational changes when a small amount of an organo mercaptoalkyl borate is present. The lubricating oils containing the tri(mercaptoethyl)borate and tri(dodecylmercaptoethyl)borate have very little increase in viscosity

Although the invention has been described particularly with reference to lubricating oil compositions, it will be recognized that other lubricating compositions including oils thickened to the consistency of a grease are included by the invention.

While our invention has been described with reference to various specific examples and embodiments, it will be understood that the invention is not limited to such examples and embodiments and may be variously practiced within the scope of the claims hereinafter made.

We claim:

1. A lubricating composition comprising a major amount of a lubricating oil and a small amount, sufficient to improve the antiwear and load-carrying characteristics of the oil, of an organo mercaptoalkyl borate having the general formula



wherein R is selected from the group consisting of hydrogen, alkyl, aryl, alkaryl, aralkyl and cycloalkyl radicals containing 1 to 16 carbon atoms and n is an integer of 2 to 16.

2. The lubricating composition of claim 1 wherein the organo mercaptoalkyl borate is present in an amount of about 0.5 to 10 percent by weight.

3. A lubricating composition comprising a major amount of a lubricating oil and a small amount, sufficient to improve the antiwear and load-carrying characteristics of the oil, of an organo mercaptoethyl borate having the general formula

$$B(OCH_2CH_2SR')_3$$

wherein R' is selected from the group consisting of hydrogen and an alkyl radical containing 1 to 12 carbon atoms.

4. The lubricating composition of claim 3 wherein the organo mercaptoethyl borate is tri(mercaptoethyl)borate.

5. The lubricating composition of claim 3 wherein the organo mercaptoethyl borate is tri(dodecylmercaptoethyl) borate.

6. A lubricating composition comprising a major amount of a mineral lubricating oil and about 0.5 to about 10 percent by weight of tri(mercaptoethyl)borate.

7. A lubricating composition comprising a major amount of a mineral lubricating oil and about 0.5 to about 10 percent by weight of tri(dodecylmercaptoethyl) borate.

8. A lubricating composition comprising a major amount of diisooctyl azelate and about 0.5 to about 10 percent by weight of tri(mercaptoethyl)borate.

9. A lubricating composition comprising a major amount of diisooctyl azelate and about 0.5 to about 10 percent by weight of tri(dodecylmercaptoethyl)borate.

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DANIEL E. WYMAN, *Primary Examiner.*

L. G. XIARHOS, *Assistant Examiner.*