

UNITED STATES PATENT OFFICE

2,570,719

FOAM SUPPRESSED LUBRICANT COMPOSITIONS

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6 Claims. (Cl. 252-49.6)

1

The present invention relates to foam suppressed lubricant compositions, and the like. It relates more particularly to lubricating compositions containing one or more ingredients which tend normally to promote foaming and containing a novel type foam inhibitor.

The formation of foam is distinctly disadvantageous under many circumstances, as, for example, in engine oils for internal combustion engines, particularly the heavy duty type of oils. Many internal combustion engines employ both pressure and splash systems of lubrication, whereas other motors use only one of the systems. In either case the formation of foam on top of the oil during the operation of the motor leads to serious consequences due to lack of lubrication. This is particularly so in pressure lubricating systems employing the so-called "dry sump" operation where the oil returning from the engine parts is picked up by a scavenging pump and returned to the lubricant reservoir. Since the scavenging pump normally has a capacity of several times the volume of the oil returning from the engine, a large amount of air is pumped along with the lubricant and as a result foam tends to build up in the oil reservoir to a point where some oil may be lost through the breather pipe or to a point where the pressure pump delivers foam instead of oil to the various moving parts of the engine with a resultant lack of lubrication. Many heavy duty lubricating oils for gasoline and diesel engines contain dissolved therein from 0.5-5% of various metallo-organic additives, such as metal phenates, phenol sulfides, carboxylates, sulfonates, alcoholates, metal salts of esters of phosphorus and thio phosphorus acids, etc., which as oil soluble detergents act to maintain bearings and the internal surfaces of the engines free of gummy or varnishlike deposits. Oils containing additives of the above type are especially prone to foam under many conditions. The addition to oils of certain non-metallic agents, such as phosphatides, organic esters, fatty compounds, thickeners, etc., also promotes foaming on occasion.

In the case of gear lubricants, such as those designed for the lubrication of automobile rear ends and transmissions, when such lubricants show a marked tendency to form foam during use the foam entraps the lubricant to such an extent that the oil level will fall below the gear train. These oils usually owe their ability to withstand high unit loads to the presence of additives containing sulfur, chlorine or phos-

2

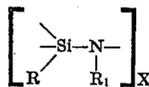
phorus or to the presence of lead soaps. Hydraulic and shock absorber oils likewise need protection against foaming.

Various suggestions have been made in the prior art to the effect that certain oxygen-containing materials are useful foam suppressors, especially in oils containing detergents, and the like, which tend to promote foaming. Thus, the patents to Borsoff et al., No. 2,430,857 and No. 2,430,858 suggest the use of alcohols, aldehydes, and the like. Other references, e. g., Larsen and Diamond, Patents Nos. 2,375,007 and 2,406,671, have suggested the use of certain oxygen-silicon compounds for this purpose, these materials being of the type described by Rochow, for example, in his Patent No. 2,258,220. Still other related materials such as cyclo-silicones have been suggested, e. g., in Hersh Patent No. 2,464,231.

The present invention is based upon the discovery that the reaction products of alkyl silicon halides with ammonia or with primary amines have excellent properties as foam suppressants in oils, especially mineral base oils, containing detergents or other foam promoters. These products, which are alkyl amino silanes, may be used in very small quantities, e. g., 0.0001 to 0.01%, for the purpose indicated. Proportions as small as 0.0001% by weight, based on the total lubricant are quite useful. Proportions of 0.00125% to 0.005% are specifically preferred.

In general, alkyl polysilamines useful for purposes of the present invention may be prepared as follows:

A lower alkyl silicon halide, for example, $(CH_3)_2SiCl_2$ is reacted with ammonia, or with a primary alkyl or aryl amine to form an amino silane of the following general structure



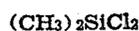
where R is a lower alkyl group, of C₁ to C₄, R₁ is hydrogen or alkyl or aryl, and X is an integer of such value that the polymer has a high molecular weight, e. g., 300 or more, up to several thousand. Where R is methyl or ethyl, as is preferred, the above materials are readily prepared by mere admixture.

The invention will be further understood by reference to the following example showing preparation of an amino-silane and its use in varying quantities in oils containing substantial amounts of foam promoting materials of the detergent type.

EXAMPLE

Method of preparation

14.8 g. of dimethyl silicon dichloride,



was added to 25 cc. of benzene in a 500 cc. Ehrlenmeyer flask. The flask and contents were cooled in a Dry Ice-acetone bath with a calcium chloride drying tube in the stopper to keep water condensate out. 31.5 g. of hexadecylamine, dissolved in 300 cc. of benzene was added to the cold $(\text{CH}_3)_2\text{SiCl}_2$. The contents of the flask were then allowed to warm up to room temperature. A reaction took place forming a gel. The benzene and any hydrogen chloride formed were removed by means of a water aspirator and the remaining solid was then air dried. The final product was a white, waxy powder.

Data on foam suppression

CRC L-12 foam tests were run on a blend of 2.5 weight per cent of the reaction product of P_2S_5 and the barium salt of tert.-octyl phenol sulfide in a heavy duty mineral base lubricating oil having a viscosity of 50 S. S. U. at 210° F. containing various amounts of the above polyalkyl silamine.

The base oil containing 2.5% by weight of the reaction product of P_2S_5 and the barium salt of tert.-octyl phenol sulfide, as above, showed the results set forth in the table.

TABLE
Results of CRC L-12 foam tests

Foam Depressant	Wt. Per Cent in Blends		c. c. Foam in Sequence		
			Test 1, 75° F.	Test 2, 200° F.	Test 3, 75° F.
None.....	0.000	(A)	260	440	250
		(B)	240	200	240
Polyalkyl silamine.....	0.00125	(A)	110	50	0
		(B)	100	0	0
Do.....	0.00250	(A)	0	140	0
		(B)	0	40	0
A Commercial Poly-silicone.....	0.00125	(A)	0	140	330
		(B)	0	0	270

For comparative purposes, commercial oxygen-containing polysilicones were also tested and were found unsatisfactory in the third phase of the foaming test.

The details of the foaming test CRC L-12 are as follows: In test 1, the sample, maintained at a temperature of 75° F. is air blown at a constant rate for five minutes, then allowed to settle for ten minutes. The volume of foam is measured at the end of both periods and recorded as (A) and (B) respectively. In tests 2 and 3, the identical test is repeated in sequence on a second sample at temperatures of 200° and 75° F., respectively.

While the amino silanes derived from dimethyl silicon dichloride are referred to specifically above, homologs such as diethyl disilicon dichloride are equally useful. The NR_1 radical may be derived from ammonia gas or from any alkyl or aryl primary amine having up to about 30 carbon atoms in the aryl or alkyl group.

Suitable primary amines are (1) straight chain aliphatic amines such as hexadecylamine, 1,4-diaminoheptane, triacontylamine, 5-amino octene-2; (2) branched chain aliphatic amines such as 3-ethyl-5-methyl hexamine, 4-ethyl-5-isopropyl octylamine-3; (3) aromatic amines such as aniline, leucaniline, xylydine, phenylene

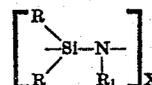
diamine, naphthylamine, di-phenyl butylamine, di-phenyl hydrazine; (4) alicyclic amines, such as cyclobutylamine, cyclooctylamine, vestrylamine; (5) heterocyclic amines such as histamine, aminopyridine, and the like. The reaction, being exothermic, may be conducted at any suitable temperature, for example from -20° F. to about 100° F. or up to 150° F.

The quantity of foaming suppressor to be used in any given lubricant will vary widely with the composition of the lubricant. Where large amounts of detergents or emulsifiers are present, the quantity of defoamer used should be increased, but in most circumstances a quantity of 0.01% by weight, based on the weight of the total lubricant, will be adequate.

In order to obtain satisfactory dispersion of the amino silane in the extremely small quantities employed, it is desirable first to disperse it by vigorous agitation in a small quantity of carrier liquid, for example a light lubricating oil which can be added to the larger mass to obtain a thorough mixing more easily.

What is claimed is:

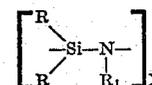
1. A lubricating composition consisting essentially of a mineral base oil of lubricating grade containing a detergent and, as a foaming suppressor, 0.0001 to 0.01% by weight, based on the total composition, of a material having the general formula:



wherein R is a lower alkyl group of 1 to 4 carbon atoms, R_1 is a member of the group consisting of hydrogen, alkyl and aryl, and X is an integer of such value that the polymer has a molecular weight of at least 300.

2. Composition as in claim 1 wherein the quantity of foaming suppressor is between 0.00125 and 0.005%.

3. A mineral base oil composition containing a minor proportion of a foam promoter and 0.0001 to 0.01% by weight, based on the total composition, of a polyamino-silane having a molecular weight of at least 300 of general formula



where R is a lower alkyl group of the C_1 to C_4 range, R_1 is selected from the group consisting of hydrogen, alkyl and aryl.

4. Composition according to claim 3 wherein the polymer is a primary amine substituted silane polymer.

5. Composition according to claim 1 wherein R_1 is an alkyl group.

6. Composition according to claim 1 wherein R_1 is a hexadecyl radical.

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REFERENCES CITED

The following references are of record in the file of this patent:

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2,462,635	Haber	Feb. 22, 1949
2,464,231	Hersch	Mar. 15, 1949