

UNITED STATES PATENT OFFICE

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LUBRICANT

Mario S. Altamura, Brooklyn, N. Y., assignor to
Socony-Vacuum Oil Company, Incorporated,
a corporation of New York

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This invention has to do with lubricants. More particularly, the present invention has to do with lubricants which, in the presence of water, are likely to emulsify and which, in the presence of water and ferrous metal surfaces, generally fail to retard or prevent rusting of said surfaces.

As is well known to those familiar with the art, difficulties have been experienced during the operation of steam turbines, and this is particularly true during the initial operation of new steam turbines because of emulsion formation and rusting of the ferrous metal parts. During steam turbine operation, some moisture or water accumulates in the oil system which is designed to lubricate the bearings. When conditions become favorable, the oil and water emulsify, whereupon the high degree of lubricating efficiency required in turbine operation is seriously reduced or impaired.

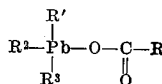
Additionally, the combination of fresh (not previously used) oil, clean new metal surfaces and moist conditions of operation of new steam turbines appears to be particularly conducive to rusting difficulties. Particles of rust deposited on or carried to bearing surfaces of small clearance such as the governor, for example, tend to cause sticking which renders them inoperative. In cases of severe rusting, bearings may be injured. This rusting phenomenon is evidently the result of improper or inadequate "wetting" of the metal surfaces with oil, thus permitting a preferential contact of the metal surfaces with the water which is present.

Numerous materials have been proposed for use in oils subject to the aforesaid shortcomings in order to counteract either the emulsification tendency or the inadequate "wetting" effect thereof. Generally, such materials as have been previously proposed may effectively reduce the emulsification tendency of the oil, but are usually ineffective in retarding rust formation or even accelerate the same. Corresponding materials proposed to correct the rusting action normally characteristic of turbine oils and the like either fail to retard emulsion formation or favor such formation. Further difficulty is introduced when other addition agents are incorporated in mineral oil for the purpose of lowering the pour point of the oil, increasing "oiliness," or increasing oxidation resistance of the oil. In some cases, particularly when pour point depressants are incorporated in the oil, the addition agents have proven undesirable in that they have contributed to emulsion formation.

The present invention is directed to an out-

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standing mineral oil composition which is substantially free from the aforesaid emulsion and rusting difficulties. The mineral oil composition of this invention comprises a mineral oil fraction having in admixture therewith a small amount of a trialkyl-plumbane salt of an aliphatic acid represented by the following general formula:



wherein R is an alkyl group having from about four to about ten carbon atoms, and R', R² and R³ are either the same or different alkyl groups, and are preferably ethyl groups. Closely related compounds are not characterized by both rust and emulsion inhibiting properties, as are those compounds which satisfy the aforesaid general formula. For example, triethyl lead acetate, wherein R is a methyl group, is unsatisfactory as a rust inhibitor; and corresponding compounds wherein R is a lauryl, myristyl, stearyl group or the like are unsatisfactory as emulsion retardants.

The trialkylplumbane salts of aliphatic acids contemplated herein may be prepared by any of the procedures set forth in the chemical literature, typical of which are the methods described by Klippel, J. prakt. Chem., (1) 81, 287, (1881), and by Browne and Reid, J. Am. Chem. Soc. 49, 830-8 (1927). The preparation of compounds of this invention are illustrated below in the example wherein the method of Browne and Reid, referred to above, was followed:

EXAMPLE

Triethyl lead caproate

One mol each of tetraethyl lead and of caproic acid were placed in an Erlenmeyer flask equipped with a reflux condenser. The flask was partially immersed in a water bath and the temperature of the bath was raised to 80° C. A small amount, about 0.1 gram, of silica gel was added to the reactants whereupon a slow evolution of ethane was noted. The temperature was then slowly raised to 90° C. and so maintained for 1½ hours. At the end of this period the liberation of ethane had practically ceased. The reaction mixture was a fluid which, upon cooling in an ice-water mixture, solidified to a white, crystalline mass. The latter material was filtered on a suction filter and thereafter pressed on a porous plate. The crude product was then taken up in boiling petroleum ether to which a small amount of decolorizing charcoal had been added. Silica gel con-

taminating the product was removed by filtration and the filtrate was then distilled to remove petroleum ether therefrom. The product, triethyl lead caproate, is a snow-white, crystalline solid, melting at 94.5-95.3° C. and requires no further purification. The product was immediately bottled and protected from air and light, inasmuch as decomposition occurs on exposure thereof to air and light.

To demonstrate the outstanding character of the oil compositions contemplated herein, typical emulsion and rust tests were carried out with a mineral oil and with blends of the mineral oil and small amounts of salts represented by the aforesaid general formula. The emulsion tests used were the Herschel demulsibility test at 130° F. described in the Federal Standard Stock Catalogue, VV-L-791a, Method 320.32, F-6z; and the Steam emulsion test described in the Tag Manual for Inspectors of Petroleum, Test No. 28, A. S. T. M. (Serial D157-36). These tests are not comparable, since frequently an oil which is quite acceptable in one test will not be acceptable in the other. Accordingly, an oil which is to be used in steam turbines, for example, should pass both of these tests. The Herschel demulsibility test is designed to measure the resistance of an oil to the formation of semi-permanent emulsions with water, and is represented in terms of the demulsibility number, D. N.; improved emulsive properties being indicated by larger demulsibility numbers. The steam emulsion test is represented in terms of the steam emulsion number, S. E. N. Lower S. E. N. values are indicative of improved emulsive properties inasmuch as the ability of the oil to resist emulsification varies inversely with the S. E. N.

In demonstrating the effectiveness of the salts of this invention, an S. A. E. 20 lubricating oil (S. U. V. of 56 seconds at 210° F.) alone or containing 0.1 per cent of a commercial pour depressant (an esterified alkyl-substituted phenol) having an adverse effect on emulsive properties, was used as the base oil. The results of these tests are presented in Table I following:

Table I

Base Oil	Conc. (Wt. Per Cent) of Triethyllead Caproate	D. N.	S. E. N. (Secs.)
S. A. E. 20.....	-----	1,080	230
S. A. E. 20+Depressant.....	-----	810	300+
S. A. E. 20+Depressant.....	0.012	1,260	200

The rust test used demonstrates the relative ability of an oil to resist rust formation on ferrous metal surfaces of steam turbines and simulates steam turbine operation. The test consists of inserting a clean steel specimen in 300 ccs. of the test oil containing 30 ccs. of distilled

water, and stirring the oil at 138-142° F. for test periods of the order of two days. The rust formed is noted at the end of the test period. The oil used in the test is a turbine oil having a Saybolt Universal viscosity (S. U. V.) of 150 seconds at 100° F. The results of these tests are set forth in Table II below.

Table II

Improving Agent	Conc. (Wt. Percent)	Rust Test
None.....	-----	Very severe rust in ½ hr.
Triethyllead caproate.....	0.075	Mild rust in 24 hrs.
Triethyllead caproate.....	0.1	No rust in 72 hrs.

The salts of this invention are generally used in oils in amounts of from about 0.01 per cent to about 1.0 per cent, although preferred concentrations are of the border of 0.01 per cent to about 0.1 per cent. As indicated above, these salts may be used alone in oils and may also be used in oils containing one or more additive agents. For example, they may be used in oils with pour point depressants, oxidation inhibitors, film strength improving agents, oiliness agents, etc. Typical of such addition agents are the well-known phenols, amines, sulfides, organic compounds containing phosphorus, organic compounds containing halogen, and the like.

It is to be understood that although the invention has been illustrated hereinabove by certain preferred salts and oil blends of the same, it is not to be restricted thereto and it is to be broadly construed in the light of the scope of the appended claim.

I claim:

A mineral oil composition comprising a mineral oil fraction normally permitting rusting of ferrous metal surfaces in contact therewith in the presence of water, and normally susceptible to emulsion formation in the presence of water, having in admixture therewith a minor proportion, from about 0.01 per cent to about 1.0 per cent of triethyllead caproate.

MARIO S. ALTAMURA.

REFERENCES CITED

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

UNITED STATES PATENTS

Number	Name	Date
1,857,761	McCabe	May 10, 1932
2,236,910	Lincoln et al.	Apr. 1, 1941
2,257,750	Lincoln et al.	Oct. 7, 1941
2,261,888	Rocchini	Nov. 4, 1941
2,288,288	Lincoln et al.	June 30, 1942
2,290,413	De Groote	July 21, 1942
2,363,510	Farrington	Nov. 28, 1944
2,367,470	Neely et al.	Jan. 16, 1945