

esters of fatty acid and alcohol or by a mixture obtained from transesterification of vegetal or animal oils with polyethylene or polypropylene glycol or a higher alcohol, or by the synthetic products from the esterification of mono or polyalcohols with fatty acids or from the esterification of diacids with monoalcohols; and particularly a solution of a nitrogenous thiomolybdate in a diol, glycol, polyalcohol or polyethyleneglycol; the lubricants thus obtained having the property of decomposing at temperatures and pressures produced by friction.

Among others the following specific oils can be used as lubricants according to the invention:

Castor oil
Peanut oil
Olive oil
Ox-foot oil
Fish oil
Monooleates of polyethylene glycols
Monooleates of butylene glycols
Monooleates of propylene glycols
Monooleates of polytrimethylene glycols
Palmitates of polypropylene glycols
Dioleates of polyethylene glycols
Dioleates of butylene glycol
Dioleates of polypropylene glycols
Dioleates of polytrimethylene glycols
Bisheptyl sebacate
Bisheptyl adipate
Bisheptyl methyl adipate
Bisheptyl sebacate
Bisheptyl adipate
Bisheptyl methyl adipate
Bisocetyl sebacate
Bisocetyl adipate
Bisocetyl methyl adipate
Bisnonyl sebacate
Bisnonyl adipate
Bisnonyl methyl adipate
Bisdecyl sebacate
Bisdecyl adipate
Bisdecyl methyl adipate
Bisundecyl sebacate
Bisundecyl adipate
Bisundecyl methyl adipate
Bisdodecyl sebacate
Bisdodecyl adipate
Bisdodecyl methyl adipate
Decane diol
Octane diol
Hexane diol

Lubricants containing hydrocarbon-soluble molybdenum blue, as well as those obtained with the aid of a nitrogenous thiomolybdate, are disclosed and claimed in copending applications filed on even date herewith by Georges Hugel for "Hydrocarbon-Soluble Molybdenum Blues," Serial No. 480,612, filed January 7, 1955, now Patent No. 2,852,469 (corresponding to P.V. 661,055, filed in France on January 6, 1954) and for "Lubricants Containing Nitrogenous Thiomolybdates" (corresponding to P.V. 661,056, filed in France on January 6, 1954).

The present invention encompasses within the scope thereof lubricants produced with the aid of molybdc xanthates, and also those molybdc xanthates per se of the hereinbefore-given formula wherein R contains 6 or more carbon atoms, as for example molybdc decylxanthate, molybdc dodecylxanthate and molybdc hexylxanthate.

The principal results of tests using as lubricant, by way of example, an oil containing 2% by weight of molybdc decylxanthate, have been set forth above. The table which follows sets forth results obtained with the "4-Ball Machine," while insuring retention of the protective coating by means of an oil containing 1% by weight of molybdenum blue:

Tests on "Four-Ball Machine"
OIL WITH 1% OF MOLYBDENUM BLUE

5	Load Applied in kg.	Delay in Seizing in seconds	Coefficient of Friction			Diameter of Impression in mm.
			Before Seizing	Maximum	After Seizing	
BALLS SUBJECTED TO PRELIMINARY TREATMENT WITH MOLYBDIC XANTHATE						
10	280----- 350-----	>60 39	0.14 0.22	extremely small.	coalescence of balls in 57 sec.	0.82-----

This table shows that performances obtained while insuring retention of the protective coating by a lubricant with a hydrocarbon-soluble molybdenum blue base are remarkable since it is possible therewith to attain a pressure of 350 kg. with a seizing delay of 39 seconds.

It is also possible to use any one of the lubricants which come into consideration for the formation, and not only for the maintenance, of the said protective coating.

Comparable results are then attained provided that the increase in pressure is progressive and is effected with continuous lubrication. In fact, under the influence of the temperature attained by friction, a molybdenum sulfur compound is deposited in successive layers on metallic parts subject to friction, resulting simultaneously in the formation and in the maintenance of a protective coating.

The performances thus realized, with continuous lubrication by the progressive application of constantly increasing pressures, have already been set forth in connection with lubricants containing a nitrogenous thiomolybdate (thiomolybdate of an organic nitrogen base) in copending application, Ser. No. 480,611, filed January 7, 1955, now Patent No. 2,909,541 (corresponding to P.V. 661,056 in France). The table which follows sets forth results of tests obtained under the same conditions with a lubricant containing, for example, 2% by weight of molybdc decylxanthate:

Tests with "Four-Ball Machine"

[Mineral oil S.A.E. 30 (viscosity at 50° C.=54 centipoises) +2% by weight of molybdc decylxanthate (1 mm. at each load with the same balls, with progressively increasing pressures and without preliminary coating)]

Load Applied in kg.	Delay in Seizing in seconds	Coefficient of Friction			Diameter of Impression in mm.
		Before Seizing	Maximum	After Seizing	
130----- 150----- 180----- 200----- 240----- 260----- 280----- 300----- 320----- 340----- 360-----	>60 >60 >60 >60 >60 >60 >60 >60 >60 >60 >60	0.09 0.14 0.16 0.13 0.25 0.23 0.24 0.18 0.18 0.15 0.11	----- ----- ----- ----- ----- ----- ----- ----- ----- ----- -----	----- ----- ----- ----- ----- ----- ----- ----- ----- ----- -----	----- ----- ----- ----- ----- ----- ----- ----- ----- ----- 1.1

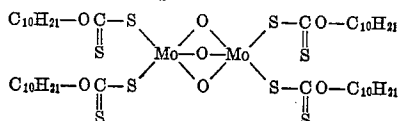
The following examples set forth representative embodiments of preparing molybdc xanthates according to this invention (percentages are by weight and yields are molar yields):

Example 1

One mol of anhydrous ammonium molybdate (196 grams) is dissolved in a liter of water admixed with 10 kilograms of crushed ice in a container which is exteriorly cooled by ice.

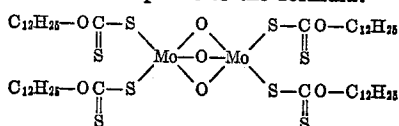
A solution of 2 mols (512 grams) of sodium decylxanthate is prepared in 2.5 liters of water. This second solution is slowly added, while stirring vigorously, into the first-prepared ammonium molybdate solution. There is

The solution in petroleum ether, after decantation, is dried over anhydrous magnesium sulfate, filtered, and freed of petroleum ether by evaporation under reduced pressure on a water-bath. There are obtained as residue 400 grams of molybdic decylxanthate in the form of a very dark red, almost black, oily mass (which represents a yield of 70% relative to the theoretical yield). Molybdic decylxanthate corresponds to the formula:



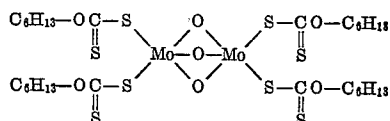
Example 2

The procedure being otherwise the same, there are obtained 450 grams of molybdc dodecylxanthate, which represents a yield of 71% of the theoretical. Molybdc dodecylxanthate corresponds to the formula:



Twenty parts by weight of ammonium molybdate are dissolved in 100 parts by volume of water. The resultant solution is poured into 1000 parts by weight of the solution in 250 parts by weight of water of 50 parts by weight of hexylxanthate.

Then there are gradually added 25 parts by volume of an aqueous acetic acid solution of 20% strength. The resultant organomolybdic compound is then extracted with benzene, the latter is eliminated from the extract by boiling on a water-bath, first under atmospheric pressure and finally under a pressure of 10 mm. of Hg. There are thus obtained 37 parts by weight of molybdic hexylxanthate, which corresponds to a yield of 76.4% of the theoretical. Molybdic hexylxanthate corresponds to the formula:

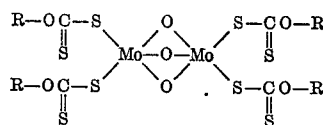


5

According to the first of the aforementioned copending applications, a hydrocarbon-soluble molybdenum blue can be prepared from a molybdenum blue which is soluble e.g. in isobutyl alcohol, by dissolving the last-mentioned blue in isobutyl alcohol to which has been added an organic liquid, such as a higher alcohol, which is simultaneously soluble in isobutyl alcohol and in hydrocarbons, then expelling the isobutyl alcohol, and finally concentrating the remainder down to a suitable concentration whereby excess solvent is eliminated. The starting molybdenum blue can also be extracted from its aqueous solution by means of a phosphorus compound, such as diethylamido-di-decylphosphate, which is soluble in oil but not in water.

20 According to the second of the aforementioned co-
pending applications, a thiomolybdate of an organic ni-
trogen base, as for example triethylamine, is prepared
from a soluble thiomolybdate and a soluble salt, e.g.
the hydrochloride of the base, whereby the desired organic
25 base thiomolybdate is precipitated. These thiomolybdates
are soluble in lubricant bases, including polyalcohols
and the like, and are thermally decomposable to yield
sulfur compounds of molybdenum for coating parts which
are subject to friction.

1. A lubricant consisting essentially of a mixture of about 90 to 99.5% by weight of a lubricating mineral oil and about 0.5 to 10% by weight of a molybdenic xanthate having the formula



2. A lubricant according to claim 1 wherein the molyb-
dic xanthate is molybdc decylxanthate.

45 3. A lubricant according to claim 1 consisting essentially of about 98% by weight of the mineral oil and about 2% by weight of the molybdc xanthate.

4. A lubricant according to claim 1 wherein the molyb-
dic xanthate is molybdc dodecylxanthate.

50 5. A lubricant according to claim 1 wherein the molyb-
dic xanthate is molybdc hexylxanthate.

6. A lubricant according to claim 1 consisting essentially of about 92% by weight of the mineral oil and about 8% by weight of the molybdc xanthate.

UNITED STATES PATENTS

60	1,740,809	Cronshaw et al. -----	Dec. 24, 1929
	2,173,383	Carter -----	Sept. 19, 1939
	2,346,155	Denison et al. -----	Apr. 11, 1944
	2,363,884	McCleary et al. -----	Nov. 28, 1944
	2,394,954	Watkins -----	Feb. 12, 1946
65	2,436,051	Mixon -----	Feb. 17, 1948
	2,758,089	Hoff et al. -----	Aug. 7, 1956