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N-SUBSTITUTED FATTY ACID AMIDE LUBRICANTS

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ABSTRACT OF THE DISCLOSURE

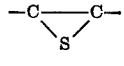
This invention relates to N-acylmorpholines and N-mono and N,N-disubstituted fatty acid amides and to similar derivatives of epithioamides which are useful as base and extreme pressure lubricants and additives.

This application is a continuation-in-part of Ser. No. 176,734 filed Aug. 31, 1971.

A non-exclusive, irrevocable, royalty-free license in the invention herein described, throughout the world for all purposes of the United States Government, with the power to grant sub-licenses for such purposes, is hereby granted to the Government of the United States of America.

This invention relates to certain compounds which are N-fatty acyl derivatives of primary and second amine and N-substituted acyl derivatives of primary and secondary amines which have exhibited utility as base lubricants, extreme pressure lubricants or lube additives. More particularly, this invention relates to N- and N,N-disubstituted long chain aliphatic amides the acyl component of which is a normal, branched, or substituted alkenoic or alkanoic acyl group containing from 16 to 22 carbon atoms the amide nitrogen of which may be derived from an alkyl amine, dialkyl amine, alkyl-alkoxyalkylamine, dialkoxyalkylamine or nitrogen heteroalicylic. Typical amines are butylamines, dibutylamines, N-methyl-alkoxyethyl amine, di-ethoxyethyl amine, and morpholine. The acyl substituent referred to may be divalent sulfur or pentavalent phosphorous.

We have discovered that many of the simple N-alkyl and N,N-dialkyl or N-alkyl-N-alkoxyalkyl amines of the C_{16} to C_{22} alkeneoic or alkanoic fatty acids are good base lubricants. We have further discovered that the introduction of divalent sulfur into the fatty acid moiety imparts excellent extreme pressure lubricant characteristics to such compounds. In particular we have found the epithio group



to be most effective. We have also observed that the introduction of both hydroxy and alkyl phosphate groupings alpha to each other also imparts extreme pressure lubricant characteristics to the compound.

In addition it was noted that the above hydroxy-phosphato compound and its mercapto analog N-[9(10)-mercapto-(9)10-dibutylphosphato]stearamide were effective antiwear compounds and additives.

Included among the specific compounds operable as base lubricants are N-methyl-N-butyloleamide, N-ethoxyethoxyethoxy propyloleamide, N-ethoxyethoxy, propyloleamide, N-methoxyisopropyloleamide, N-methoxyethyloleamide. Compounds showing particular promise as extreme pressure lubricants or additives are N,N-dibutyl-9,10-epithiostearamide, N,N - dibutyl-9,10,12,13-diepithiostearamide, 9,10-epithiostearoyl morpholine, N-ethoxyethoxyethoxypropyl-9,10 - epithiostearamide, N-9,10,12, 13-diepithiostearoylmorpholine and N-[9(10) - hydroxy-9(10)-dibutylphosphato]stearamide.

Compounds showing particular promise as antiwear additives are N,N - dibutyl-[9(10)-hydroxy-(9)10-dibutylphosphato]stearamide and N,N-[9(10)mercapto-(9)10-dibutylphosphatostearoylmorpholine.

In addition to their utility as beneficial additives for petroleum and synthetic diester base lubricants, it has more recently been discovered that some of the epithioamide and dibutyl phosphate derivatives thereof and of epoxides are useful additives for silicone fluids. Silicones in certain applications such as brass-on-brass, brass-on-nickel, and bronze-on-nickel lubrication are considered good lubricants. However, in the most common metal combination, steel on steel, they are very poor. Because of their low-toxicity and non-corrosiveness and superior physical and chemical characteristics as base oils compared to petrochemicals and synthetic esters in areas such as thermal stability, viscosity characteristics and sludging it would be decidedly advantageous to upgrade their deficient load carrying and wear characteristics as lubricants for the forementioned metal surfaces.

Most additives normally employed for the improvement of extreme pressure and antiwear characteristics in petroleum and synthetic diester base oils are insoluble in silicones and therefore ineffectual. We have discovered that the amide derivatives herein described are soluble in some silicone fluids and contribute a significant improvement at the 5% level in both the extreme pressure and antiwear performance of the silicone base oil.

EXAMPLE 1

N-methyl-N-butyloleamide.—100 grams (0.33 mole) of oleoyl chloride was added dropwise and with stirring to a mixture of 29 grams (0.33 mole) of N-methyl-N-butylamine and 27 grams (0.33 mole) of pyridine. Stirring was continued until the heat of reaction subsided. The solid pyridine hydrochloride was filtered off and the filtrate was washed successively with aqueous HCl and water until acid free. It was dried stripped, percolated through an activated alumina column, and the product removed from the percolate by stripping off the solvent. Analysis of the product: Percent C, 78.87 (theory 78.63); percent H, 13.02 (theory 12.82); percent N, 3.96 (theory 3.99).

EXAMPLE 2

N,N-dibutyloleamide.—This material was prepared by the procedure of Example 1 from 100 grams (0.33 mole) of oleoyl chloride 42.5 grams (0.33 moles) of di-n-butylamine and 27 grams (0.33 mole) pyridine. Analysis of the product: Percent C, 78.94 (theory 89.25); percent H, 13.16 (theory 13.06); percent N, 3.44 (theory 3.56) confirm the preparation.

EXAMPLE 3

Ethoxyethoxyethoxy propyloleamide.—This material was prepared by the procedure of Example 1 from 100 grams (0.33 mole) of oleoyl chloride, 73.0 grams (0.33 mole) ethoxyethoxyethoxypropylamine and 27.0 grams (0.33 mole) pyridine. Analysis of percent C, 71.84 (theory 72.04); H, 11.80 (theory 11.80), and N, 2.96 (theory 2.90) confirm the preparation.

EXAMPLE 4

Ethoxyethoxypropyloleamide.—This material was prepared by the procedure of Example 1 from 100 grams (0.33 mole) of oleoyl chloride, 58.3 grams (0.33 mole) ethoxyethoxypropylamine and 27.0 grams (0.33 mole) of pyridine. Analyses of percent C, 71.45 (theory 73.80); percent H, 11.87 (theory 12.07); and percent N, 2.92 (theory 3.18) confirm the preparation.

EXAMPLE 5

Methoxyisopropyloleamide.—This material was prepared by the procedure of Example 1 from 100 grams (0.33 mole) of oleoyl chloride, 29.3 grams (0.33 mole) methoxyisopropylamine and 27.0 grams (0.33 mole) pyridine. Analysis of percent C, 73.15 (theory 74.99); percent H, 12.01 (theory 11.93); and percent N, 3.67 (theory 3.97) confirm the preparation.

EXAMPLE 6

Methoxyethyloleamide.—This material was prepared by the procedure of Example 1 from 100 grams (0.33 mole) of oleoyl chloride, 24.4 gram (0.33 mole) of methoxyethylamine and 27.0 grams (0.33 mole) pyridine. Analysis of percent C, 73.08 (theory 74.55); percent H, 11.98 (theory 11.83); and percent N, 3.31 (theory 4.14) confirm the preparation.

EXAMPLE 7

Oleoylmorpholine.—This material was prepared by the procedure of Example 1 from 100 grams (0.33 mole) of oleoyl chloride, 28.7 grams (0.33 mole) of morpholine and 27.0 grams (0.33 mole) of pyridine.

EXAMPLE 8

N,N-dibutyloleamide.—This product was prepared following the exact procedure of Example 2 except for the substitution of oleoyl chloride by lineoleoyl chloride.

EXAMPLE 9

N,N-dibutyl-9,10-epithiostearamide.—120 grams (0.30 mole) of N,N-dibutyloleamide was epoxidized by the addition with stirring of 62.0 grams (0.36 mole) of meta-chloroperbenzoic acid in 630 ml. of chloroform. Reaction was continued for 1 hour beyond final addition and the excess peracid destroyed by a small amount of a 10% solution of Na_2SO_3. The meta-chlorobenzoic acid was removed by a $NaHCO_3$ wash, followed by water washing. The $CHCl_3$ solution was then dried with anhydrous Na_2SO_4 and the $CHCl_3$ stripped off to recover the product N,N-dibutyl-9,10-epoxystearamide, oxirane content 3.45% (theory 3.91%). 100 grams (0.24 mole) of this product was added to a well stirred slurry of 55.7 g. (0.75 moles) of thiourea and 89.5 g. (0.72 moles) of benzoic acid in acetone. Stirring was continued for 3 hours beyond the terminal addition whereupon the benzoic acid was washed out with 38.8 g. (0.36 mole) of Na_2CO_3 and the organic product extracted with hexane. The organic phase extract was dried and stripped to recover the product N,N-dibutyl-9,10-epithiostearamide containing 6.41% sulfur (theory 6.67%).

EXAMPLE 10

N-(9,10-epithiostearoyl)morpholine.—This product was prepared by the exact procedure described in Example 9 except for the substitution of oleoylmorpholine for the N,N-dibutyloleamide of Example 9. Analysis of product showed percent C, 71.22 (theory 69.34); percent H, 11.22 (theory 10.77); percent N, 3.66 (theory 3.68); percent S, 3.44 (theory 7.80).

EXAMPLE 11

N,N - dibutyl - 9,10,12,14 - diepithiostearamide.—This product was prepared following the procedure described in Example 9 except for the replacement of the N,N-dibutyloleamide of Example 9 by N,N-dibutyllinoleamide, and a doubling of the molar ratios of all reagents. The product analyzed as follows: percent C, 69.64 (theory 68.57); percent H, 10.93 (theory 10.76); percent N, 3.10 (theory 3.07); percent S, 11.7 (theory 14.06).

EXAMPLE 12

N - ethoxyethoxyethoxypropyl - 9,10 - epithiostearamide.—To 166 g. of N-ethoxyethoxyethoxypropyl oleamide, Example 3, was added dropwise and with stirring 73.8 g. of m-chloroperbenzoic acid in 800 ml. of $CHCl_3$.

The spent m-chloroperbenzoic acid was removed with 10% aqueous $NaHCO_3$ and the epoxide recovered by drying and stripping off the $CHCl_3$. 171 g. of this epoxide was added dropwise to a slurry of 56 g. thiourea in 1000 ml. of acetone and the concurrent addition of 43.4 g. of glacial acetic acid and the reaction mixture stirred for 3 hours. The glacial acetic acid was neutralized with Na_2CO_3 and the episulfide, N - ethoxyethoxyethoxypropyl - 9,10-epithiostearamide, extracted with Skellysolve B and washed, dried, and stripped. Its elemental analysis was C=67.1 (66.52), H=11.42 (10.88), N=2.67), 2.87 S=2.29 (6.57).

EXAMPLE 13

N - Ethoxyethoxyethoxypropyl - 9(10)mercaptostearamide.—173 g. of N-ethoxyethoxyethoxypropyloleamide in CCl_4 was treated in the cold with a CCl_4 solution containing 60.9 g. of bromine. Any unreacted bromine was removed by a thiosulfate wash. 140 g. of the resulting dibromo compound was added dropwise to a well stirred solution of 72 g. of $Na_2S \cdot 9H_2O$ in dimethylsulfoxide maintained at 80° C. Stirring was continued at 80° C. for 3 hours. Water was added and the reaction product was extracted with hexane; washed, dried, and stripped. Elemental analysis of the product was C=66.3 (66.5); H=11.1 (10.9), N=2.7 (2.9), S=4.4 (6.8).

EXAMPLE 14

N,N - Dibutyl - 9(10) [dibutylphosphono]stearamide.—100 g. of N,N-dibutyloleamide, Example 2, 148 g. of dibutyl phosphite, and 1.29 of benzoyl peroxide catalyst were heated at 115° C. for 3½ hours. Additional units of 1.29 g. of benzoyl peroxide was added after the first and second hours of reaction time. The excess dibutyl phosphite was then removed by distillation at reduced pressure, 0.45 mm. Hg. The stillpot contents showed strong adsorption bonds at 8, 9.3, and 9.7 microns characteristics of the phosphonate group. The elemental analysis was C=obs 71.46 (69.50), H=obs 12.42 (11.92), N=obs 2.20 (2.38), P=obs 4.24 (5.28) percent respectively.

EXAMPLE 15

N-9,10-12,13-diepithiostearoylmorpholine.—This product was prepared by the same procedure described in Example 9 except for the substitution of linoleoyl morpholine for the N,N-dibutyloleamide of Example 8 and the doubling of molar proportions of the m-chloroperbenzoic acid in the epoxidation step and the thiourea and benzoic acid in the "epthioation" step. The elemental analysis was C=obs 68.83 (63.92), H=obs 10.26 (9.44), N=3.29 (3.38), S=obs 4.00 (15.49) percent respectively.

EXAMPLE 16

N,N-dibutyl - [9(10)-hydroxy-(9)10-dibutylphosphato] stearamide.—44.7 g. of dibutylhydrogen phosphate was added to 182.2 g. of N,N-dibutyl-9,10-epoxystearamide at 90–95° C. with stirring. Reaction conditions were continued for 3½ hours after termination of the addition. The reaction product was dissolved in commercial hexane and the unreacted dibutyl hydrogen phosphate neutralized and washed out with portions of aqueous 10° $NaHCO_3$ followed by several water washes. Acidulation of the hexane phase with diluted HCl followed by several water washes, drying and stripping, yielded the product. Elemental analysis was, C=obs 66.56 (65.91), H=obs 11.04 (11.30), N=obs 2.90 (2.26), P=obs 3.04 (4.20).

EXAMPLE 17

N-[9,(10)mercapto - (9)10 - dibutylphosphatostearoyl] morpholine.—12 g. of dibutylhydrogen phosphate was added dropwise with stirring to 51.0 g. of (9,10-epithiostearoyl)morpholine, the preparation of Example 10, at 85–90° C. and the heating and stirring continued for 3 hours beyond the terminal addition. The reaction product

was dissolved in commercial hexane and any excess or unreacted

$$HO-\overset{\overset{O}{\|}}{P}-(OC_4H_9)_2$$

was neutralized and washed out with aqueous 10% $NaHCO_3$. The hexane extract was washed several times with water dried and stripped. The elemental analysis of the product was C=obs 66.74 (69.36), H=obs 10.69 (11.05), N=obs 2.89 (3.32) S=obs 3.27 (2.17), P=obs 1.68 (2.10) percent respectively.

EXAMPLE 18

The compounds prepared in accordance to the procedures given in Examples 1 through 7 were evaluated as base lubricants in the Shell Four-Ball Wear tests following a modified procedure of ASTM D2266–67. The resulting scar diameter on the balls after running at 600 r.p.m. for 1 hour at 120° C. and 50 kg. load were compared with the scars obtained using commercial lubricants such as Aeroshell Mil-L-708 (di-2-ethylhexyl sebacate and additive), Gulfpride, single G, MS, multiviscosity, and 100 sec. paraffin oil. The results of these tests are reported in Table 1.

TABLE I.—ANTIWEAR LUBRICANT PROPERTIES OF FATTY ACID AMIDES

	Compound	Average wear scar, mm. Without additive	With 5% additive	Additive
1	N,N-bis (2-ethoxyethyl) oleamide.	0.597	0.735	N,N-di-butyl-9,10-epithiostearamide.
2	N,N-di-n-butyl-9,10-epithiostearamide.	0.842	----	None.
3	Oleoylmorpholine	0.623	0.658	Do.
4	N,N-dimethyloleamide	0.797	0.987	Do.
5	N,N-di-n-propyloleamide	0.908	0.885	Do.
6	N,N-di-n-hexyloleamide	0.893	0.898	Do.
7	N,N-di-n-butylerucamide	0.758	0.798	Do.
8	N,N-di-n-butyl amide of sel. hydrogenated cottonseed fatty acids.	0.798	0.958	Do.
9	N,N-bis(2-methoxyethyl) oleamide.	0.626	0.725	Do.
10	N,N-dibutyloleamide	0.710	----	
11	N-oleoyl-4-propyl-piperidine.	0.847	----	
12	Morpholide of sel. hydrogenated cottonseed fatty acids.	0.642	----	
13	N-methyl-N-butyl-oleamide.	0.607	----	
14	N-ethoxyethoxyethoxy-propyoleamide.	0.503	----	
15	N-ethoxyethoxypropyl-oleamide.	0.526	----	
16	N-methoxyisopropyl-oleamide.	0.705	----	
17	N-methoxyethyloleamide	0.420	----	
18	102 sec. paraffin oil	----	0.902	N-methoxyethyloleamide.
19	D.O.S	----	0.906	Do.
20	N,N-dibutyl-9(10)-carbobutoxyoctadecanamide.	0.610	----	
21	102 sec. paraffin oil	----	0.818	N-N,dibutyl-9(10)-carbobutoxyoctadecanamide.
22	D.O.S	----	0.945	Do.
23	N-[9(10) mercapto(9)10-dibutylphosphato-stearoyl]morpholine.	0.615	----	
24	102 sec. paraffin oil	----	0.552	N-[9(10)mercapto-(9)10-dibutylphosphatostearoyl] morpholine.
25	D.O.S	----	0.535	Do.
26	N,N-dibutyl-[9(10)-hydroxy-(9)10-dibutyl-phosphato]stearamide.	0.498	----	
27	102 sec. paraffin oil	----	0.498	N,N,dibutyl-[9(10)-hydroxy-(9)10-dibutylphosphatol stearamide.
28	D.O.S	----	0.498	
29	N,N-dibutyl-9(10)-[dibutylphosphono] stearamide.	0.530	----	
30	N-ethoxyethoxyethoxy-propyl-9,10-epithio-stearamide.	0.707	----	
31	N-ethoxyethoxyethoxy-propyl-9(10) mercapto-stearamide.	0.690	----	
32	N-(9,10,12,13-diepithio-stearoyl)morpholine.	0.665	----	
33	Bis(2-ethylhexyl)sebacate	0.872	----	
34	100 sec. paraffin oil	0.803	----	
35	Aero Shell (Mil-7808)	0.587	----	
36	Gulf Pride, Single G, MS, multiviscosity.	0.447	----	
37	100 sec. paraffin oil	----	0.813	N,N-dibutyl-9,10-12,13-diepithio-stearamide.
38	Bis(2-ethylhexyl)sebacate	----	0.836	Do.
39	100 sec. paraffin oil	----	0.723	N-9,10-epithio-stearoyl morpholine.
40	Bis(2-ethylhexyl)sebacate	----	0.915	Do.
41	100 sec. paraffin oil	----	0.917	N,N-dibutyl-9,10-dichlorostearamide.
42	Bis(2-ethylhexyl)sebacate	----	0.848	Do.

TABLE I.—Continued

It can be seen from the data presented that the N-alkoxyalkyl and N,N-di(alkoxyalkyl)oleamides, without the assistance of additives, exhibited wear characteristics (i.e., low scar diameters) comparable to the commercial controls Aeroshell and Gulfpride, which do contain additives to improve performance. N,N-dibutyl-[9(10)-hydroxy-(9)10-dibutylphosphato]stearamide and N-[9(10)mercapto-(9)(10)-dibutylphosphatostearoyl]morpholine not only exhibit low wear characteristics in themselves but also impart as additives such characteristics to paraffin oil and D.O.S. base lubricants.

EXAMPLE 19

The various amides were evaluated as extreme pressure lubricants or admditives in a Shell 4-ball extreme pressure tester at 1440 PPM following ASTM Procedure D2595–67T. Loads were increased in increments of 20 kg. to weld point and the test ran for one minute or to weld whichever occurred first. Commercial hypoid fluid SAE #90 was employed as the control E. P. Lubricant. The performance of the amides tested is given in Tables II, III, and IV.

EXAMPLE 20

Selected amides were evaluated as additives to improve both the antiwear and extreme pressure performance of silicone fluids. The base silicone fluid employed was General Electric SF 1017 and the amides were employed at a 5% additive level. The results of the antiwear and extreme pressure tests are given in Table V.

TABLE II.—EXTREME PRESSURE TESTS (ASTM D2596–67T)

	Compound	Weld point, No additive	Weld point, With 5% #2
1	N,N-bis (2-ethoxyethyl) oleamide	120	170
2	N,N-di-n-butyl-9,10-epithiostearamide	300	----
3	Oleoylmorpholine	120	150
4	N,N-dimethyloleamide	120	140
5	N,N-di-n-propyloleamide	120	150
6	N,N-di-n-hexyloleamide	120	180
7	N,N-di-n-butylerucamide	120	200
8	N,N-di-n-butyl amide of hydrogenated cottonseed fatty acids.	120	170
9	N,N-bis(2-methoxyethyl)oleamide	120	160
10	N-methyl-N-butyloleamide	100	----
11	N,N-Dibutyl-9,10-epithiostearamide	300	----
12	N,N-dibutyl-9,10-12,13-diepithiostearamide	440	----
13	N-ethoxyethoxyethoxypropyloleamide	120	----
14	N-ethoxyethoxypropyloleamide	120	----
15	N-methoxyisopropyloleamide	100	----
16	N-methoxyethyloleamide	100	----
17	N-9,10-epithiostearoylmorpholine	380	----
18	Bis(2-ethylhexyl)sebacate	120	----
19	100 sec. paraffin oil	100	----
20	SAE #90 commercial hypoid fluid	280	----

TABLE III

Evaluation of N-dibutyl-9, 10-12, 13-di-epithiostearamide as an additive and as a base oil in extreme pressure tests. (ASTM D2596-67T)

Base oil	Percent additive	Load, kg.	Wear scar, mm.
100 sec. paraffin oil	5	140	1.90
Do	5	200	2.35
Do	5	220	2.75
Do	5	240	(2)
Bis(2-ethylhexyl)sebacate	5	120	1.51
Do	5	180	1.59
Do	5	[1] 190	2.47
Do	5	200	[3]
Do	10	240	2.88
Do	10	260	3.55
Do	10	270	[4]
N,N-dibutyll-9, 10-12, 13-diepithiostearamide	None	+120	1.38
Do	None	160	1.61
Do	None	200	1.89
Do	None	280	+2.13
Do	None	320	2.47
Do	None	360	2.70
Do	None	400	2.88
Do	None	440	[5]

Extreme pressure tests on N-ethoxyethoxyethoxypropyl-9, 10-epithiostearamide (PC-0-No. 7682)

Base oil	Additive	Applied load	Average scar, mm.
7682	None	120	1.05
7682	None	160	1.75
7682	None	240	2.35
7682	None	300	2.19
7682	None	340	2.55
7682	None	360	2.62
7682	None	380	Weld
102 sec. paraffin oil	7682(5%)	120	2.28
Do	7682(5%)	140	2.46
Do	7682(5%)	150	Weld
D.O.S	7682(5%)	120	2.23
D.O.S	7682(5%)	140	+2.44
D.O.S	7682(5%)	150	Weld

Extreme pressure tests on N-ethoxyethoxyethoxypropyl-9(10)-mercaptostearamide (PC-0-No. 7683)

7683	None	200	1.54
7683	None	300	2.26
7683	None	400	2.25
7683	None	450	2.15
7683	None	500	[6] 2.15
102 sec. paraffin oil	7683(5%)	120	1.97
Do	7683(5%)	140	2.35
Do	7683(5%)	150	2.54
Do	7683(5%)	160	Weld
D.O.S	7683(5%)	120	1.68
D.O.S	7683(5%)	140	2.18
D.O.S	7683(5%)	160	2.59
D.O.S	7683(5%)	170	2.72
D.O.S	7683(5%)	180	Weld

Extreme pressure tests on N-(9,10-12,13-diepithiostearoyl)morpholine (PC-O-No. 7684)

7684	None	200	2.21
7684	None	260	2.38
7684	None	340	2.43
7684	None	400	2.75
7684	None	460	2.90
7684	None	500	[6] 2.98
102 sec. paraffin oil	7684 (5%)	100	2.23
Do	7684 (5%)	120	2.61
Do	7684 (5%)	130	2.74
Do	7684 (5%)	140	Weld
D.O.S	7684 (5%)	120	2.54
D.O.S	7684 (5%)	140	2.58
D.O.S	7684 (5%)	150	Weld
N-methoxyethyloleamide	7684 (5%)	140	2.53
Do	7684 (5%)	160	2.77
Do	7684 (5%)	170	Weld

Extreme pressure tests on N,N-dibutyl-9(10)-dibutylphosphono)stearamide (PC-O-No. 7685)

7685	None	120	3.05
7685	None	140	4.50
7685	None	150	Weld

Extreme pressure tests on N,N-dibutyl-[9(10)-hydroxy-9(10)-dibutylphosphato]stearamide (PC-O-No. 7686)

7686	None	200	0.887
7686	None	300	2.78
7686	None	340	4.32
7686	None	350	4.50
7686	None	360	Weld
100 sec. paraffin oil	7686 (5%)	120	2.28
Do	7686 (5%)	140	2.80
Do	7686 (5%)	150	3.01
Do	7686 (5%)	160	Weld
D.O.S	7686 (5%)	120	1.95
D.O.S	7686 (5%)	140	2.85
D.O.S	7686 (5%)	150	3.03
D.O.S	8676 (5%)	160	Weld

TABLE III.—Continued

Extreme pressure tests on N-[9(10)-mercapto-9(10)-dibutylphosphatostearoyl]morpholine (OC-O-No. 7687)

7687	None	120	0.608
7687	None	160	1.45
7687	None	200	1.40
7687	None	240	1.70
7687	None	280	[7] 3.36

Extreme pressure tests on N,N-dibutyl-9(10)-carbobutyoxyoctadecanamide (PC-O-No. 7688)

7688	None	80	1.92
7688	None	90	2.18
7688	None	100	2.45
7688	None	120	Weld

[1] Incipient seizure.
[2] 5 seconds.
[3] 4 seconds.
[4] 10 seconds.
[5] 46 seconds.
[6] Test stopped because of extreme decomposition. Did not weld.
[7] Test stopped because of extreme decomptosiion and noxious fumes. Did not weld.

TABLE IV

Evaluation of N-9,10-epithiostearoyl morpholine as an additive and as a base oil in extreme pressure tests (ASTM D 2596-67T)

Base oil	Percent additive	Load, kg.	Wear scar, mm.
100 sec. paraffin oil	5	120	2.00
Do	5	140	2.80
Do	5	160	3.25
Do	5	180	[1] Weld
Bis(2-ethylhexyl)sebacate	5	80	1.57
Do	5	120	2.03
Do	5	140	2.41
Do	5	160	2.94
Do	5	180	3.00
Do	5	200	[2] Weld
N-9,10-epithiostearoylmorpholine	None	120	0.70
Do	None	200	2.13
Do	None	240	2.43
Do	None	300	2.68
Do	None	380	[3] Weld

[1] 10 sec. [2] 5 sec. [3] 50 sec.

TABLE V

Antiwear and extreme pressure performance of some amide additives in silicone base lubricants

Base oil	Additive[1] 5%	Load, kg.	Average wear scar, mm.	E.P. weld pt., kg.
General Electric SF 1017	None	5	2.36	130
Do	None	50	4.16	190
Do	7641	5	1.38	
Do	7641	50	4.32	230
Do	7642	5	0.410	
Do	7642	50	3.07	180
Do	7684	5	1.62	
Do	7684	50	3.36	
Do	7686	5	1.41	160
Do	7686	50	3.54	
Do	7687	5	1.34	200
Do	7687	50	3.52	

[1] Additives: 7641—N-9,10-epithiostearoylmorpholine; 7642—N,N-dibutyl-9,10,12,13-diepithiostearamide; 7684—N-(9,10,12,13-diepithiostearoyl)morpholine; 7686—N,N-dibutyl-[9(10)-hydroxy-(9)10-dibutyphosphato]stearamide; 7687—N,N-[9(10)mercapto-(9)10-dibutylphosphatostearoyl]morpholine.

NOTE.—At 5 kg. load, sample 7642 reduced the wear scar of SF 1017 from 2.36 mm. (no additive) to 0.41. The other samples also exhibited antiwear additive properties; however, these did not reduce the scar diameters to acceptable levels. Samples 7642 and 7687 also have good E.P. additive properties in SF 1017. Samples 7641 and 7684 were not as effective.

We claim:

1. A composition of matter useful as a base and extreme pressure lubricant, said matter consisting of a silicone base lubricant and 5–10 weight percent of an N,N-disubstituted epithioamide selected from the group consisting of N,N-dibutyl-9,10-12,13-diepithiostearamide and N,N-dibutyl-[9(10)-hydroxy - (9)10 - dibutylphosphato]-stearamide.

2. The process of claim 1 wherein the N,N-disubstituted epithioamide is N,N-dibutyl - 9,10 - 12,13-diepithiostearamide.

3. The process of claim 1 wherein the N,N-disubstituted epithioamide is N,N-dibutyl-[9(10)-hydroxy-(9)10-dibutylphosphato]stearimide.

4. A composition of matter useful as a base and extreme pressure lubricant, said matter consisting of a silicone base lubricant and 5–10 weight percent of an N-epithioacyl

morpholine selected from the group consisting of N,N-[9(10)mercapto - (9)10 - dibutylphosphatostearoyl]morpholine, N-9,10-epithiostearoyl morpholine and N-(9,10-12,13-diepithiostearoyl)morpholine.

5. The process of claim 4 wherein the N-epithioacyl morpholine is N,N - [9(10)mercapto-(9)10-dibutylphosphatostearoyl]morpholine.

6. The process of claim 4 wherein the N-epithioacyl morpholine is N-9,10-epithiostearoyl morpholine.

7. The process of claim 4 wherein the N-epithioacyl morpholine is N - (9,10-12,13-diepithiostearoyl)morpholine.

References Cited

UNITED STATES PATENTS

2,722,518	11/1955	Watson	252—46.3
3,278,649	10/1966	Barnas	252—46.7
3,317,426	5/1967	Lowe	252—46.7
3,321,401	5/1967	Ford et al.	252—46.7

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