

1

2,964,475

LUBRICANTS CONTAINING METAL CARBOXYLATE AND METAL PHOSPHATE**Arnold J. Morway, Clark, N.J., assignor to Esso Research and Engineering Company, a corporation of Delaware****No Drawing. Filed May 26, 1958, Ser. No. 737,491****9 Claims. (Cl. 252—18)**

This invention relates to lubricating oil compositions. Particularly, it relates to lubricants containing an alkaline earth metal salt of a low molecular weight carboxylic acid and a metal phosphate.

Alkaline earth metal salts of C_2 to C_5 fatty acids are useful in lubricating oil compositions for improving the extreme pressure properties and anti-wear properties of the lubricant. Also certain metal phosphates (e.g. trisodium phosphate) are very valuable materials for addition to lubricating compositions as they also serve as extreme pressure and anti-wear agents. In addition, many of these phosphates serve to prolong the effectiveness of conventional antioxidants by neutralizing the acidic products which result from oxidation degradation of the oil, thereby preventing these acidic materials from further catalyzing the decomposition of the oil. While both the alkaline earth metal carboxylates and the phosphates are valuable addition agents for oil, they are not dispersible per se in the oil to form stable lubricating oil compositions. In order to effectively use these materials it has been the practice to incorporate other agents to suspend these salts in oil. Thus, various soap and salt thickeners and so-called soap-salt complex thickeners have been used to stably incorporate the low molecular weight carboxylates into oil. In other instances, certain surfactants have been used, or the carboxylate is added in the form of a hydrate. In the case of the phosphates, heretofore it has only been possible to effectively use them in the form of greases. Thus, a sufficient amount of another thickener such as a soap or perhaps a polymeric thickener has been necessary to impart enough body to the oil to make a solid grease and thereby suspend the phosphate.

It has now been found that these low molecular weight carboxylates and the metal phosphates, when used together in oil, tend to suspend each other to thereby form a stable oil dispersion. Thus, stable fluid lubricants can be made containing the low molecular weight carboxylate and the phosphate. Based upon this discovery, very large quantities of these two materials may be incorporated into the oil without undue thickening of the oil or the necessity of forming a stable grease. As another advantage, the need for more expensive higher molecular weight soaps or the necessity for forming mixed soap-salt complexes as well as the attendant expense has been eliminated.

The low molecular weight carboxylates useful in this invention are those alkaline earth metal (e.g. calcium strontium, magnesium or barium) salts of a C_2 to a C_5 fatty acid (e.g. acetic, propionic, butyric acid, etc.). These metal carboxylates are best prepared in situ in the lubricating oil by neutralization of the appropriate acid or acid anhydride with a metal base, e.g. calcium hydroxide. By this in situ formation a very fine degree of dispersion of the carboxylate in the lubricating oil is obtained. Of course, the carboxylate in a powdered form may be directly dispersed in the lubricating oil or

2

it may be added to the lubricating oil in a water solution or dispersion and the excess water removed by heating.

The preferred carboxylate is calcium acetate, in either an anhydrous form or in the form of a monohydrate. If the calcium acetate is formed by neutralizing acetic acid with calcium hydroxide at a temperature between about 200 to 300° F. the monohydrate will result. Heating above 300° F. results in anhydrous calcium acetate.

The phosphate materials which may be used in preparing the compositions of the invention include the alkali metal (e.g. sodium and lithium) and the alkaline earth metal (e.g. calcium, barium, magnesium) salts of phosphoric acid, i.e. ortho, meta and pyrophosphoric acid. Preferred salts are those of orthophosphoric acid such as tricalcium orthophosphate, monocalcium dihydrogen orthophosphate, trisodium orthophosphate and disodium hydrogen orthophosphate.

The lubricating oil used in the compositions of the invention may be a mineral lubricating oil, a synthetic lubricating oil, or mixtures thereof. Synthetic lubricating oils which may be used include esters of dicarboxylic acids (e.g. di-2-ethylhexyl sebacate); esters of glycols (e.g. C_{13} Oxo acid diester of tetraethylene glycol); complex esters (e.g. the complex ester formed by reacting one mole of sebacic acid with two moles of tetraethylene glycol and two moles of 2-ethyl-hexanoic acid); formals, silicones, carbonates, polyglycols and other synthetic lubricating oils known in the art.

Particularly desirable synthetic oils are those diester oils containing a total of 15 to 40, e.g. 20 to 35 carbon atoms and consisting of saturated hydrocarbon chains linked by two ester linkages. Such esters are prepared from C_6 to C_{20} alkanols reacted with C_6 to C_{12} alkanedioic acids; or C_2 to C_8 glycols reacted with C_6 to C_{12} alkanedioic acids. Preferred esters are those prepared from alkanedioic acids and Oxo alcohols. The Oxo alcohols are isomeric mixtures of branched chain primary alkanols produced by the reaction of an olefin with hydrogen and carbon monoxide in the presence of a cobalt catalyst to form a mixture of aldehydes, which aldehydes are then hydrogenated to form the Oxo alcohol. Such Oxo alcohols and their preparation are well known in the art. Specific examples of diesters of the above types will include: di- $(C_{10}$ Oxo) adipate; di(2-ethylhexyl) sebacate; di- $(C_8$ Oxo) sebacate; 1,6-hexamethylene glycol di-(2-ethylhexanoate) and 1,10-decamethylene glycol di-(caprylate).

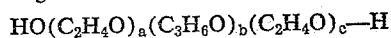
The compositions of the invention will contain a major amount of lubricating oil and about 1.0 to 50.0 e.g. 3.0 to 45.0 wt. percent of the carboxylate-phosphate combined additive. This additive combination in turn will generally comprise about 1.0 to 3.0, preferably 1.0 to 2.5 molar proportions of the carboxylate per molar proportion of the phosphate. In terms of weight proportions, this will roughly correspond to about 1.3 parts by weight of carboxylate per about one part by weight of phosphate.

The oil composition may also contain other additives. Examples of such additives include viscosity index improvers such as polyisobutylene; corrosion inhibitors such as sorbitan monooleate; pour depressants; dyes; detergents and the like.

As indicated above the phosphate and carboxylate stably suspend each other in oil so that no salt precipitation occurs. However, in certain instances if the oil-salt system is completely anhydrous, a small amount of the oil will tend to separate from the main mass upon prolonged storage. This is not a serious problem since upon mixing a homogeneous mass is readily obtained. However, it does represent a slight marketing deterrent. It has been further found that this can be inhibited by

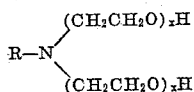
3

various surface active agents. One such type of surface active agents are copolymers of ethylene glycol and propylene glycol which are commercially available under the trade-name of "Pluronics." These materials have the following formula:

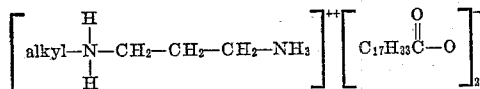


where a and c are integers of value 2 to 150, and b is an integer of 5 to 170. The molecular weight may vary considerably but generally is between 1,000 and 20,000, e.g. 1,000 to 10,000. Thus, Pluronic L-44, Pluronic L-62, and Pluronic F-68 appear to have molecular weights of about 2800, 2300 and 8700 respectively. The ratio of the ethylene oxide groups to the propylene oxide groups in the Pluronic materials vary between about 3/1 to 1/3. Thus, in Pluronics L-62, analysis showed a ratio of about 1.15 moles of ethylene oxide per mole of propylene oxide. However, the ratio of these two alkylene oxide groups is not critical for the present invention since operable materials will include ethylene or propylene polyglycols per se.

Another useful surface active agent includes N,N-polyalkyleneoxy disubstituted alkyl amine such as Ethomeen T/25 which has the formula:



where R is an alkyl group derived from tallow and the total number of ethylene oxide groups is about 15. Still another surface active agent which can be used is Duomeen T dioleate which has the structure:



wherein the alkyl group is derived from tallow. Other surface active agents which may be used are described in my copending patent application Ser. No. 692,041, filed on October 24, 1957. Generally, about 0.05 to about 2.0 wt. percent based on the weight of total composition of surface active agent will be sufficient to prevent oil separation or bleeding under even the worst adverse conditions.

The carboxylate-phosphate materials of the invention may also be incorporated into greases thickened with any conventional grease thickeners such as: soaps, soap-salt or mixed-salt complexes, polymeric thickeners (e.g., polymers of C_2 to C_4 monoolefins of 10,000 to 200,000 mol. wt. such as polyethylene), or inorganic thickeners (e.g., clay, carbon black, silica gel, etc.). The carboxylate and phosphate materials may be directly dispersed in said greases; or a lubricating oil dispersion of the carboxylate-phosphate materials may be added to the other greases; or other thickeners may be added or formed in the lubricating oil dispersion of the carboxylate and phosphate, etc. By using the carboxylate-phosphate combination of the invention with other grease making materials, not only does the combination impart extreme pressure, anti-wear and antioxidant properties to the composition, but the amount of other grease thickeners required to produce a grease of a given hardness is substantially reduced.

The invention will be further understood by the following examples.

EXAMPLE I

A grease composition wherein all parts are by weight was prepared as follows:

17 parts of hydrated lime was mixed with 47 parts of a mineral lubricating oil having a viscosity of 80 SUS at 210° F. to form an intimate mixture. 25 parts of glacial acetic acid was then slowly added while stirring, the heat of reaction causing the temperature of the mixture to rise to a maximum of about 210° F. After all

4

the acetic acid was added, the mixture was then stirred for another ½ hr. Then, 10 parts of trisodium orthophosphate was stirred into the mixture, after which 1 part of phenyl α -naphthylamine was added. No external heating was applied. A solid grease material resulted, which was homogenized by passage through a Morehouse mill having a .003" opening.

EXAMPLES II AND III-A

The compositions of these examples were prepared by mixing additional amounts of mineral lubricating oil with the product of Example I.

EXAMPLE III-B

A sample of the product of III-A was heated to 320° F. in order to completely dehydrate the product and form anhydrous calcium acetate. The composition was then homogenized in a Gaulin homogenizer operating at 5000 p.s.i.

EXAMPLE IV

Example I was repeated except that 2 parts by weight of Pluronics-62 (described above) was initially mixed with the mineral oil and hydrated lime before the addition of the acetic acid. Also, 45 parts by weight of the mineral lubricating oil was used instead of the 47 parts by weight used in Example I.

EXAMPLE V

The product of Example IV was cut back with additional mineral lubricating oil after which the blended product was passed through a Gaulin homogenizer operating at 5,000 p.s.i.

EXAMPLE VI

A portion of the product of Example V was heated to a maximum temperature of 325° F. in order to dehydrate the composition and form anhydrous calcium acetate.

The compositions and properties of the products prepared above are summarized in Table I.

As seen from Table I, solid grease compositions may be prepared which contain only the calcium acetate and sodium phosphate as shown by Example I. The product of Example I was tested in the Anti-friction Bearing Manufacturers-National Lubricating Grease Institute's test machine and had a lubricating life in excess of 1800 hours. The grease composition of Example I can be readily cutback with varying amounts of lubricating oil to form either a softer grease as shown by Example II, or to form fluid products as illustrated by Examples III-A and III-B. Examples II, III-A and III-B further show that the resulting product had good wear characteristics as indicated by the 4-ball wear test and good load-carrying ability as indicated by the Almen test. Furthermore, such products were stable even when maintained at a high temperature for an extended length of time. Thus, in Examples III-A and III-B, there was no separation or settling of the solids after being maintained in an oven for 8 days at 350° F. Example III-B did show a slight separation of oil from the body of the material. However, this is not a serious disadvantage since upon stirring the original composition would be obtained. Also, no discoloration of the oil occurred during these oven tests, thus indicating that there was no serious oxidation degradation of the mineral oil. Examples IV, V and VI illustrate compositions further containing a minor amount of a polyglycol type surface active agent. Example VI did not show the slight oil separation that had occurred in Example III-B during the oven test. This improvement was due to the presence of the small amount of Pluronics L-62.

The above data further illustrates that the calcium acetate may be present either in a hydrous form, i.e. as a monohydrate as illustrated by Examples II and III-A or it may be present in the anhydrous form as illustrated by Example III-B.

Table I

Components (Weight Percent)	Examples						
	I	II	III-A	III-B	IV	V	VI
Glacial Acetic Acid	25.0	12.5	5.0	5.0	25.0	5.0	5.0
Hydrated Lime	17.0	8.5	3.4	3.4	17.0	3.4	3.4
Sodium Phosphate (Na_3PO_4)	10.0	5.0	2.0	2.0	10.0	2.0	2.0
Phenyl α -naphthylamine	1.0	0.5	0.2	0.2	1.0	0.2	0.2
Pluronic L-62					2.0	0.4	0.4
Mineral lubricating oil (80 SUS at 210° F.)	47.0	23.5	89.4	89.4	45.0	89.0	89.0
Mineral lubricating oil (55 SUS at 210° F.)		50.0					
Form of Calcium Acetate	mono-hydrate.	mono-hydrate.	mono-hydrate.	anhydrous	mono-hydrate.	mono-hydrate.	anhydrous.
Properties:							
Appearance	(1)	(1)	(1)	(1)	(1)	(1)	(1)
Dropping Point, ° F.	500+				500+		
Penetration 77° F. mm./10:							
Unworked	175	340			180		
Worked 60 strokes	195	365			200		
Worked 10,000 strokes		355			210		
Viscosity at 100° F. (SUS)			1,925	1,575		semi-fluid	semi-fluid.
Viscosity at 210° F. (SUS)			135	110		205	145
4-Ball Wear Test (1800 r.p.m.-10 Kg. Load-75° C.-1 hour).		0.31	0.29	0.28		0.41	0.32
Almen Test (Wgts. carried):							
Shock Loading						13	
Gradual Loading		15	15	15		15	
Stability (8 days at 350° F.)			(2)	(2)			(3)

1 Smooth, uniform product.

2 Excellent, no salt separation.

3 Excellent, no salt or oil separation.

EXAMPLE VII

0.8 part by weight of polyethylene of 250,000 molecular weight was dispersed in 88.2 parts by weight of a mineral lubricating oil having a viscosity of 80 SUS at 210° F. by stirring the mixture while heating to 330° F. The oil-polyethylene dispersion was then cooled to 100° F. and 0.4 part by weight of Pluronic L-62 was added along with 3.4 parts by weight of hydrated lime. After blending in these last two named components into the composition, 5.0 parts by weight of glacial acetic acid was then slowly added during which the temperature rose to a maximum of about 210° F. The composition was then stirred for an additional ½ hour after which 2 parts by weight of trisodium orthophosphate was added, followed by an additional 30 minutes' stirring. The resulting composition was then homogenized by passage through a Morehouse mill having a .003" clearance.

EXAMPLE VIII-A

Mineral oil, Ethomeen T/25 (previously described) and hydrated lime were intimately mixed without ex-

ternal heating. Glacial acetic acid was slowly added while stirring and the temperature of the mixture rose to 210° F. Mixing for another half-hour was continued and disodium acid orthophosphate was added and stirred in, followed by the addition of phenyl α -naphthylamine. A solid grease resulted which was passed through a Morehouse mill having a 0.003" opening.

EXAMPLE VIII-B

The product of Example VIII-A was cutback with additional mineral oil and homogenized at 4,000 p.s.i.

EXAMPLE VIII-C

The product was similar to that of VIII-B except that the base material was cutback with a different mineral oil in a different amount.

The composition and properties of the products of Examples VII through VIII-C are summarized in the following table:

Examples VII, VIII-A, B and C all contained the calcium acetate in the form of the monohydrate. These examples illustrated the use of both trisodium orthophos-

Table II

Components (Weight Percent)	Examples			
	VII	VIII-A	VIII-B	VIII-C
Hydrated Lime	3.4	14.0	7.0	2.8
Glacial Acetic Acid	5.0	20.0	10.0	4.0
Trisodium Phosphate (Na_3PO_4)	2.0			
Disodium Acid Phosphate		20.0	10.0	4.0
Pluronic L-62	0.4			
Ethomeen T/25		2.0	1.0	0.4
Polyethylene (250,000 m.w.)	0.8			
Phenyl alpha-Naphthylamine	0.2	1.0	0.5	0.2
Mineral Lubricating Oil (80 SUS at 210° F.)	88.2	43.0	21.5	88.6
Mineral Lubricating Oil (55 SUS at 210° F.)			50.0	
Properties:				
Appearance	Smooth fluid	Solid grease	Solid grease	
Penetration 77° F. mm./10 (ASTM):				
Unworked		210	295	
Worked 60 strokes		220	310	
Worked 10,000 strokes			325	
Dropping Point, ° F.		500+		
Consistency:				
Vis./100° F.	SUS 2,500			1,945
Vis./210° F.	SUS 2,000			165
4 Ball Wear Test (1800 r.p.m.-10 Kg. Load-75° C.-1 hour).	0.29		0.29	0.32
Almen Test (Wgts. carried):				
Gradual Loading	15		15	15
Shock Loading	11		15	10
Pin Condition	Excellent			
Oven Test:				
8 Days at 210° F.	(1)			(4)
Lubrication Life-Hours-(250° F.-10,000 r.p.m.)			1,710 hours	

1 No separation or discoloration.

phate and disodium acid orthophosphate to form both solid greases and fluid lubricants, depending upon the relative amounts of salt material used.

EXAMPLE IX

In this composition 30 parts by weight of mineral lubricating oil having a viscosity of 80 SUS at 210° F., 20 parts by weight of a di-(C₁₀ Oxo) adipate and 24 parts by weight of hydrated lime were intimately mixed to form a smooth dispersion. The C₁₀ Oxo-alcohol used in preparing the ester was obtained by subjecting tripropylene to the previously described Oxo process. Then a mixture consisting of 15 parts by weight of glacial acetic acid and 10 parts by weight of an 85 wt. percent concentrate of orthophosphoric acid was added to the main mixture while stirring and without external heating. As the acid mixture was added, the heat of reaction caused the composition to reach a maximum temperature of 220° F. The composition was then externally heated to attain a temperature of 320° F. in order to dehydrate the product. It was noted that as long as water was present the mixture appeared as a smooth grease-like material. However, as the water was removed by continued heating the product became more fluid and grainy. The resulting hot anhydrous fluid dispersion was then cooled to 200° F., where 1 part by weight of phenyl α -naphthylamine was added, following which the mixture was cooled to 150° F. At this point the product was passed through a Morehouse mill. An excellent fluid product having a very slight degree of graininess resulted.

EXAMPLE X

The product of Example IX was cutback with additional di-(C₁₀ Oxo) adipate, then passed through a Gaulin homogenizer operating at 3,000 p.s.i.

EXAMPLE XI

A grease was prepared in the general manner of Example I. However, a different proportion of materials was used, tricalcium orthophosphate was used in place of the trisodium orthophosphate and a small amount of Pluronics L-62 was added before the addition of the acetic acid.

EXAMPLE XII

A portion of the product of Example XI was mixed with additional mineral oil and the resulting mixture

was heated to 440° F. while stirring. The mixture was then cooled to 120° F. and milled in a Morehouse mill at a 0.003" opening and then filtered. Nothing filtered out.

EXAMPLE XIII

Di-(C₁₀ Oxo) adipate, lime and mineral oil were charged to a steam heated kettle and intimately mixed with no external heating. Duomeen T dioleate (previously described) was added and blended into the mass. While continuing to mix, a mixture consisting of 15 parts by weight of glacial acetic acid and 10 parts by weight of a 85 wt. percent orthophosphoric acid solution was slowly added, the temperature rising to about 200° F. After all the acid mixture was added, the mixture was stirred for another half hour. Then external heating was initiated and the temperature raised to 320° F. At this point, heating was discontinued and the dispersion was rapidly cooled to 150° F. by passing cold water through the kettle jacket, and phenyl α -naphthylamine was added. The resulting grease was Gaulin homogenized at 6,000 p.s.i.

EXAMPLE XIV

A portion of the product of Example XIII was cutback with additional lubricating oil.

The properties and composition of the preceding lubricants in terms of their original ingredients are summarized in Table III.

Examples IX, X, XIII and XIV demonstrate the direct use of orthophosphoric acid to form an orthophosphate in situ. Examples XI and XII illustrate the use of tricalcium orthophosphate. Also, the above examples illustrate the formation of both fluid and grease compositions as well as demonstrating the use of a diester oil and various additive materials.

While the preceding working examples have been directed to the use of orthophosphates, the meta and pyrophosphates may also be used. In fact, depending upon the temperatures involved and whether or not water is present, the form of the phosphate may change. Thus, sodium diacid orthophosphate, upon heating, will be converted into the metaphosphate of sodium by losing water. However, the reaction is reversible as upon the addition of water, the orthophosphate is again formed. The above is to illustrate the fact that even during use, the composition of the lubricant might change. However,

Table III

Components (Weight Percent)	Examples					
	IX	X	XI	XII	XIII	XIV
Glacial Acetic Acid.....	15.0	4.5	25.0	5.0	15.0	4.5
Hydrated Lime.....	24.0	7.2	17.0	3.4	22.0	6.6
Tricalcium Phosphate.....			10.0	2.0		
Ortho Phosphoric Acid (85%).....	10.0	3.0			10.0	3.0
Duomeen T Dioleate.....					1.0	0.3
Pluronics L-62.....			2.0	0.4		
Pheny α -naphthylamine.....	1.0	0.3	1.0	0.2	1.0	0.3
Di-(C ₁₀ Oxo Adipate).....	20.0	76.0			10.0	3.0
Mineral Lubricating Oil (80 SUS at 210° F.).....	30.0	9.0	45.0	89.0	41.0	82.3
Properties:						
Appearance.....	Heavy fluid.	Smooth fluid	Smooth grease.	Smooth fluid	Smooth grease.	Smooth fluid.
Penetration 77° F. mm./10 (ASTM):						
Unworked.....			180		370	
Worked 60 strokes.....			210		390	
Dropping Point, ° F.....			500+		500+	
Consistency:						
Vis./100° F. (SUS).....				1,728		1,295.9
Vis./210° F. (SUS).....				155		119.3
4-Ball Wear Test (1,800 r.p.m.-10 Kg. Load-75° C.-1 hour) Scar dia. mm.	0.20	0.28		0.32		0.32
Almen Test (Wgts. carried):						
Gradual Loading.....	15	15		15		
Shock Loading.....	15	15		13		
Pin Condition.....	Excellent	Slight scratch.				
Oven Test:						
14 days at 210° F.....				(1)		

¹ No separation or discoloration.

any of the forms noted above are considered as within the scope of this invention.

In summary, this invention is based upon the discovery that certain metal salts of low molecular weight fatty acids and metal salts of phosphoric acid tend to suspend each other in oil when used in certain critical ratios. Thus, either greases or fluids may be prepared in which these two salts are the sole salt components of the oil, e.g. the need for salts of higher molecular weight fatty acids as suspending or thickening agents, such as calcium stearate can be avoided.

What is claimed is:

1. A lubricating composition comprising a major proportion of a lubricating oil, an alkaline earth metal salt of a C_2 to C_5 fatty acid and a metal phosphate, wherein the metal component of said phosphate is selected from the group consisting of alkali metals and alkaline earth metals, wherein there is about 1 to 3 parts by weight of said fatty acid salt per 1 part by weight of phosphate, wherein the amount of said fatty acid salt and said phosphate is about 1 to 50.0 wt. percent of the total composition and wherein said metal salt of said C_2 to C_5 fatty acid is the sole fatty acid salt present in said composition.

2. A lubricating composition according to claim 1, wherein said phosphate is selected from the group consisting of ortho, meta and pyrophosphates.

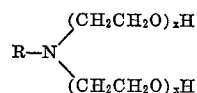
3. A lubricating composition according to claim 1, wherein said lubricating oil is selected from the group consisting of mineral lubricating oils and synthetic diester lubricating oils.

4. A lubricating composition according to claim 1, which also includes about 0.05 to 2.0 wt. percent, based on the total weight of the composition, of a material selected from the group consisting of: copolymers of ethylene glycol and propylene glycol having the formula:

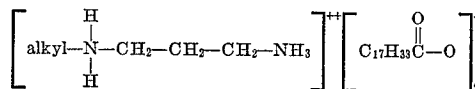


where a and c are integers of value 2 to 150, and b is an

integer of 5 to 170; N, N polyalkyleneoxy disubstituted alkyl amines having the formula:



where R is an alkyl group derived from tallow and the total of both x 's is about 15 and material of the formula:



wherein the alkyl group is derived from tallow.

5. A lubricating oil composition according to claim 1, wherein said metal salt of said fatty acid is calcium acetate and said metal phosphate is an orthophosphate.

6. A lubricating composition according to claim 5, wherein said metal phosphate is selected from the group consisting of tricalcium orthophosphate, trisodium orthophosphate, and disodium acid phosphate.

7. A lubricating composition according to claim 5, wherein said calcium acetate is anhydrous.

8. A lubricating composition according to claim 5, wherein said calcium acetate is calcium acetate monohydrate.

9. A method of lubricating relatively moving contacting surfaces which comprises maintaining between said surfaces the composition of claim 1.

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UNITED STATES PATENT OFFICE
CERTIFICATION OF CORRECTION

Patent No. 2,964,475

December 13, 1960

Arnold J. Morway

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 57, for "1.3" read -- 1 to 3 --.

Signed and sealed this 30th day of May 1961.

(SEAL)

Attest:

ERNEST W. SWIDER

Attesting Officer

DAVID L. LADD

Commissioner of Patents