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INORGANIC GEL-THICKENED LUBRICANT HAVING GOOD TEMPERATURE SUSCEPTIBILITY AND DYNAMIC WATER STABILITY CHARACTERISTICS

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This invention relates to an aerogel-thickened lubricant of good temperature susceptibility and water resistance characteristics.

Mineral lubricating oils thickened with a calcium or sodium soap are the most commonly used lubricants. The so-called soda base greases have relatively good stability at reasonably elevated temperatures but deteriorate readily in the presence of water, presumably due to the high solubility of the soda base soap in water. Calcium soap greases have better resistance to water but tend to lose their consistency or thin out at elevated temperatures. Both types of soap base greases break down at temperatures of the order of 300 to 400° F. and this breakdown is accompanied by an irreversible change in the grease structure so that on cooling the grease is found to have lost its greaselike characteristics. The aerogel greases usually are far superior to the soap base greases in stability at high temperatures as the following table shows:

TABLE I

Soap or other thickener	Appearance at 400° F.	Appearance after cooling
Aluminum	Very gummy and soft.	Hard and lumpy.
Lithium	Liquid	Do.
Calcium	do	Hard—separation of soap.
Sodium	do	Solidifies hard mass.
Do	do	Soupy.
Sodium-calcium	do	Complete separation of oil plus soap.
Silica aerogel	Unchanged	Unchanged except somewhat thinner.
Do	do	Unchanged, upon stirring, breaks down into a heavy liquid.

However after heating at high temperatures some aerogel greases tend to thin out upon stirring and this is undesirable in field applications where a grease is agitated or worked while subject to a high temperature. Aerogel greases also reveal an unsatisfactory dynamic water resistance and this again is unsatisfactory in field applications where the grease is agitated or worked in the presence of water. The grease may emulsify with water to the point of inversion to an oil-in-water emulsion, at which point the grease characteristics are lost.

In accordance with the invention water-insoluble oil-miscible or -dispersible polyalkylene glycol ethers characterized by a viscosity at 100° F. within the range of 285 to 1145 SSU are employed in conjunction with an alkylol or amino imidazoline in thickened lubricant compositions comprising a lubricating oil thickened with a nonabrasive inorganic thickening or filling agent and particularly finely divided silica, a silica aerogel being illustrative. The thickened lubricants so prepared have excellent temperature susceptibility properties and excellent dynamic water resistance, attributable to the presence of the alkylol or amino imidazoline and the polyalkylene glycol ether.

The glycol ethers are characterized by the following general formula:

$$R-\left[O-CHR_1[CHR_2]_n-\right]_x OH \quad (1)$$

where n is an integer having the value of one or more, preferably one, two or three, x is an integer taken in sufficient number to produce an ether having the prescribed viscosity, R_1 and R_2 are hydrogen or lower alkyl groups, and R is a lower alkyl group. When n is one, the oxyalkylene chain has at least a small proportion of oxyalkylene units of at least three carbon atoms, so that at least one of R_1 and R_2 is a lower alkyl group, preferably methyl. The lower alkyl group can have from one to about five carbon atoms, such as methyl, ethyl, propyl, or amyl and can have either a straight or branched chain. Preferably, one of R_1 and R_2 is hydrogen and one is a methyl group, and R can be methyl, ethyl, or isopropyl.

Within th eabove Formula 1 and worthy of especial mention are the mixed ethers of the types:

$$R-\left[\left[OCHR_1[CHR_2]_n\right]_a\left[OCHR'_1[CHR'_2]_{n'}\right]_b\right]_x OH \quad (2)$$

$$R-\left[\left[OCHR_1[CHR_2]_n\right]_a\left[OCHR'_1[CHR'_2]_{n'}\right]_b\left[OCHR_1[CHR_2]_n\right]_a\right]_x OH \quad (3)$$

where a, b and x are integers taken in sufficient number to produce an ether having the prescribed viscosity, and n and n', R_1 and R_1' and R_2 and R'_2 can be the same or different and are defined as in Formula 1. Preferably, when the "a" units are —OCH_2CH_2— and the "b" units

$$-O-\underset{\displaystyle CH_3}{\underset{|}{CH}}-CH_2-$$

or

$$-OCH_2\underset{\displaystyle CH_3}{\underset{|}{CH}}-$$

the ratio of a:b is from 75 to 25 of "a" to 10 to 90 of "b."

These ethers are obtained by reaction of the alkylene oxide with water or with a glycol, desirably in the presence of a catalyst. The reaction which takes place seems to be a simple addition wherein the alkylene oxide is converted to the corresponding oxyalkylene groups or radicals. The glycol may itself be prepared by reaction of the alkylene oxide with water. Thus water can be regarded as the ultimate starting material. The molecular weight of the polymer obtained will depend upon the relative proportions of alkylene oxide and water or glycol.

From various analyses it has been determined that these polyalkylene glycol ethers are complex mixtures of compounds having polyoxyalkylene chains of different lengths and different internal configurations, and although the materials are characterized as monoethers, they may contain a small proportion of chains having hydroxyl groups at each end, i. e., diethers. If mixed alkylene oxides are employed as the starting material the chains will contain complex mixtures of the mixed oxyalkylene radicals in various combinations, depending upon the relative proportions of the alkylene oxides, and a given chain may have several oxyalkylene units of the same type together

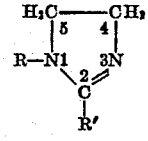
or interspersed with other oxyalkylene units in any order or combination. Thus the above formulae for the mixed ethers are merely suggestive of the types of combinations that may be found.

The important criterion from the standpoint of the invention is the viscosity of the ether, and this should be within the range from 285 to 1145 SSU at 100° F.

Thus it will be understood that there can be employed in accordance with the invention mixtures of these glycol monoethers with diethers and even dihydroxy compounds which are present in the mixture following condensation and/or etherification, but at least the major proportion and preferably upwards of 85% is the monoether. Reference is made to U. S. Patent No. 2,425,845, to Toussaint et al., dated August 19, 1947, which describes methods for the manufacture of the glycols and to U. S. Patent No. 2,425,755, to Roberts et al., dated August 19, 1947, which describes the preparation of the monoethers.

The polyethylene glycol ethers having one terminal hydroxyl group are water-soluble and would not be employed in the compositions of the invention to aid in imparting dynamic water resistance. They and related water-soluble compounds described in application Serial No. 253,984, filed October 30, 1951, now abandoned, are however effective in improving the temperature susceptibility characteristics of the thickened lubricant of the invention and can be used as a supplemental ingredient in the compositions of this invention, to take advantage of this property.

The imidazoline should be surface-active, oil-dispersible and water-insoluble and has the following structure:



Many of the compounds having the above general structure are also capable of improving the high temperature stability of lubricating oils thickened with inorganic thickening or gelling agents, such as silica aerogels.

In the above formula, R is a hydrophilic group. Especially satisfactory hydrophilic groups are alkylol groups and aliphatic, cycloaliphatic or mixed cycloaliphatic amino groups having at least one basic nitrogen atom.

The alkylol group can bear one or more hydroxyl groups.

The amino group preferably has at least two basic nitrogen atoms, which can be primary, secondary or tertiary, in any combination of the three. Where one or more of the nitrogens is primary, i. e., —NH_2, they can be attached at any position in an aliphatic chain or on a cyclic ring. Where one or more of the nitrogens is secondary or tertiary, i. e., —NH or

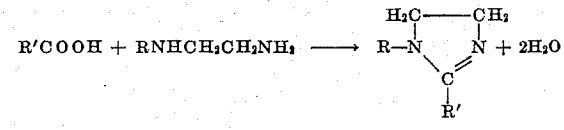
they can be substituted in a straight or branched aliphatic chain, or in a heterocyclic ring, which can itself bear alkyl, alkylene, hydroxyalkyl or hydroxyalkylene groups, desirably in the 1-position, as in the above imidazoline ring. See U. S. Patent No. 2,655,476, dated October 13, 1953, to Everett C. Hughes and Ernest C. Milberger.

Thus R can be a hydroxyethyl group, a mono-, di- or triethylene amino group, a 1-alkylene-imidazoline group, or a 1-alkylene amino imidazoline group.

R′ is an alkyl, hydroxyalkyl, alkylene or hydroxyalkylene group.

R′, which is derived from an acid, can have from eleven to twenty-one carbon atoms, such as undecyl, tridecyl, abietyl, pentadecyl, undecenyl, heptadecyl, ricinoleyl, and heptadecenyl.

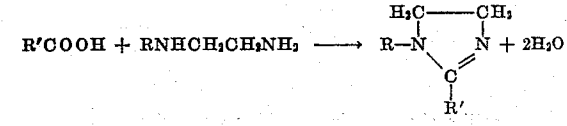
The alkylol imidazolines are prepared by reaction of aliphatic acids and hydroxy diamines followed by cyclization, in the following way:



Alkylol imidazolines in which R is from one to about six carbon atoms, such as hydroxyethyl and hydroxyisopropyl, are readily available and are preferred. The chain length for R is dependent upon the alcoholic (polar) character of the group; the larger the number of carbon atoms, the more the group takes on the character of a hydrocarbon and loses its alcoholic character. An upper limit of about eighteen carbon atoms for R is indicated by this requirement.

Amine "O," 1-β-hydroxyethyl-2-heptadecenyl imidazoline, and analogous compounds as described in application Serial No. 240,452, filed August 4, 1951, now U. S. Patent No. 2,711,393, patented June 21, 1955, have been found to be particularly effective alkylol imidazolines in the thickened lubricants of the invention.

The amino imidazolines are prepared by reaction of aliphatic acids and polyamines followed by cyclization, in the following way:



Amino imidazolines in which R has two basic nitrogen atoms and from four to about twenty carbon atoms, such as diethylene diamino and triethylene triamino, and the alkyl-substituted N-alkylene and N-alkylene-amino imidazolines (wherein R is attached to the imidazoline nucleus of the general formula through the N-alkylene or N-alkyleneamino group), are readily available and are preferred. The chain length for R is dependent upon the basic (polar) character of the group; the larger the number of carbon atoms, relative to the number of amino nitrogens, the more the group takes on the character of a hydrocarbon and loses its basic or amine character. An upper limit of about five carbon atoms per amino nitrogen is indicated by this requirement, with about a maximum of thirty carbon atoms for the R group.

The amino imidazolines described in U. S. Patent No. 2,655,476, dated October 13, 1953, have been found to be particularly effective in the thickened lubricants of the invention.

The presence of the polyalkylene glycol ether in an amount to obtain improved high temperature and dynamic water stability does not markedly effect the consistency of the thickened lubricant, i. e., the amount of the inorganic gelling agent to impart a given consistency to the thickened lubricant is not materially modified. Furthermore, the inclusion of the polyalkylene glycol ether will not effect a change in the consistency of the thickened lubricant upon storage.

Due to the inorganic nature of the gelling agent, the thickened lubricant has excellent storage stability. This is to be contrasted with the heat susceptibility and deterioration of fatty materials in soap-base greases.

The preparation of the grease is simple and readily adaptable to continuous operation, as contrasted with the

involved grease-making techniques which are often considered in the industry as an art.

The oil stock used in making the thickened lubricant may be widely varied, as contrasted with present grease-making requirements in which the oil in many cases must meet certain critical specifications.

In addition, the avoidance of the use of soap permits the manufacturer to be independent of the fat supply, which is important in periods in which fats and soaps are scarce and, many times, of pronounced nonuniformity.

The inorganic gelling agent to be used in making the thickened lubricant in accordance with this invention may be any inorganic material which forms a gel with a lubricating oil and which is so finely divided as to be non-abrasive. The preferred materials are the aerogels, which may be formed from any material not incompatible with oil, such as silica, alumina, and other gel-forming metal oxides.

A series of silica aerogels which can be used as the inorganic gelling agent of the invention are manufactured by Monsanto Chemical Company and marketed under the trade name "Santocel."

Santocel C is prepared from a sodium silicate solution in the following way: The solution is neutralized with sulfuric acid and then allowed to stand until the mixture sets to form a hydrogel. The by-product sodium sulfate is washed out by repeated washings with water. The continuous water phase in this hydrogel is then replaced by continued washing with alcohol until an alcogel is formed. In order to remove the liquid phase without a collapse of the gel structure, the alcogel is placed in an autoclave which is heated above the critical temperature of the alcohol and the pressure is allowed to increase to a point above the critical pressure of the alcohol. The vent valve is opened and the alcohol allowed to escape. Under these conditions, the silica gel structure remains practically undisturbed and the liquid phase of the gel is replaced with air. The material is then reduced in particle size by blowing it through a series of pipes containing sharp bends with jets of compressed air. Santocel C has a secondary agglomerate particle size of about 3 to 5 microns.

Santocel A is prepared as set forth for Santocel C up to the point of removal of the product from the autoclave. This material is run through a continuous heating chamber where it is heated for ½ hour to a temperature of about 1500° F. to eliminate the last traces of volatile material. It is then broken down in a reductionizer or micronizer to a particle size of about 1/16 inch in diameter. The solids content of the original hydrogel used in preparing Santocel C is approximately 25% higher than that of Santocel A.

AR is a modification of A, differing only in that the material is reductionized to about the same particle size as C, approximately 1 to 6 microns in diameter.

ARD is a modification of AR, differing only in that ARD is densified by extracting air under vacuum, and therefore has a smaller volume than AR.

AX is an A which has not been devolatilized.

CDv is a C which has been devolatilized as set forth for A. CDv is reductionized before being devolatilized.

CDvR differs slightly from CDv in that the CDvR has been devolatilized just after heating in the autoclave and then reductionized. It differs from CDv in that the latter is reductionized before being devolatilized.

The primary differences between the A's and the C's are as follows:

(1) The C's are prepared from a sodium silicate solution containing 25% more silica than the A's. Therefore, in general the A's are lighter and composed of smaller particles than the C's.

(2) The A's have undergone a devolatilization step in their preparation.

The following are the bulk densities of preferred silica aerogels:

	Density, grams per ml.
AR	0.029
ARD	0.056 to 0.064
C	0.082

In general, AR and ARD show superior gelling ability and the A's in general are better than the C's. Silica aerogels which have been devolatilized generally have a higher gelling efficiency than the undevolatilized aerogels.

Other types of inorganic gelling agents which may be used include a Fumed Silica marketed by B. F. Goodrich Company. It is finely divided and appears very much like an aerogel. It is made by a combustion or vaporization process, as a source of white "carbon black" for the rubber industry. The particles are several microns in size and porous in nature.

Another material is "Linde Silica Flour" marketed by Linde Air Products Co. It is very similar in physical appearance to the silica aerogel. The particle size of the silica is purported to be 0.01 to 0.5 micron and to be manufactured by burning silicon tetrachloride and collecting the combustion product on cool plates analogous to the production of carbon black. The particles are throught to be aggregates or clusters of particles rather than of sponge-like character.

Still another inorganic gelling agent known is "Ludox" silica from Du Pont which is known as a silica sol, and silica derivatives thereof. It has a particle size of the order of 0.01 to 0.03 micron.

In preparing thickened lubricants it is necessary to remove the water from the sol and replace it with an oil. This is possible by formulating the lubricant and removing the water by flash distillation or azeotropic distillation.

No attempt is made to enumerate all of the inorganic gelling agents which will be suitable, nor to present examples of all of them since the novel aspects of the invention reside in water-proofing the lubricant rather than the use of novel gelling agents, per se.

The lubricating oil to be used in the process may have any lubricating viscosity. It may be raw oil, acid-refined, or solvent-refined, as required for the particular lubricating need.

The nature of the base oil has been found to make little difference in the relative consistencies of the thickened lubricants and conventionally (acid) refined oils produce slightly thicker lubricants than solvent refined oils. Excellent working stability is obtained regardless of the type of the base oil. An increase in the viscosity of the base oil, as might be expected, brings increased viscosity to the thickened lubricant and minimizes bleeding. The change is relatively small and fairly linear. The viscosity of the oil does not affect the working stability of the lubricant.

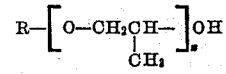
The amount of the inorganic gelling agent, as might be expected, affects the consistency of the thickened lubricant in that an increase in its concentration brings a corresponding increase in consistency. The range is fairly linear and the amount of the gelling agent can be selected with relation to the consistency desired in view of the information in the following examples. While the difference is slight, the lubricants made with lower concentrations of gelling agent possess better working stability, while lubricants with larger amounts of gelling agent show slightly improved temperature susceptibility characteristics. The bleeding tendencies are decreased by increasing concentrations of the gelling agent.

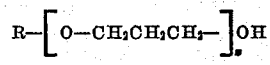
In general, the properties of the thickened lubricants are remarkably independent of the composition variables and are not critical. The relative concentration of the gelling agent effects the most significant alteration, particularly with regard to the final consistency of the product. This permits the manufacture of thickened lubricants having a wide variety of consistencies.

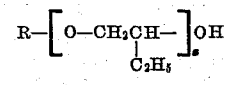
A wide variety of water-insoluble polyalkylene glycol

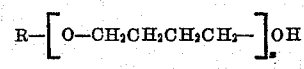
ethers can be employed in accordance with the invention. The molecular weight and chain length of the ether are not critical except as they affect viscosity. Any polyalkylene glycol ethers having a viscosity at 100° F. within the range of 285 to 1145 SSU can be employed in the composition of the invention.

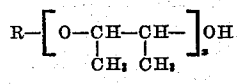
The following exemplify several preferred polyalkylene glycol ethers in accordance with the invention:

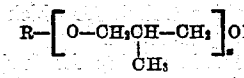
(1) 

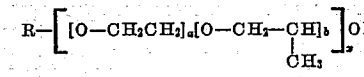
(2) 

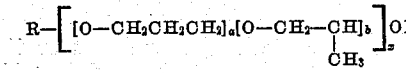
(3) 

(4) 

(5) 

(6) 

(7) 

(8) 

R in the above formulae can be methyl, ethyl, or isopropyl, and x is an integer taken in sufficient number to produce an ether having the prescribed viscosity.

The above polyalkylene glycol ethers can be mixtures of ethers in which x is variable to produce a material having the prescribed viscosity. The mixed polyethylene-1,2-propylene glycol ethers are preferred.

Exemplifying specific monoethers within the scope of the invention are the Ucon LB and LB-X series of polyalkylene glycols. These are mixed polyethylene-1,2-propylene glycol monomethyl ethers, some of which have the following properties:

TABLE II

	LB-285	LB-385	LB-525	LB-625	LB-1145
Viscosity:					
Saybolt Seconds at—					
210° F	62.7	75.1	93.1	106	177
100° F	285	385	525	625	1,145
0° F	14,200	21,600	33,000	41,000	92,000
Centistokes at—					
210° F	11.0	14.3	18.8	21.9	37.8
100° F	61.7	83.3	114	135	248
0° F	3,100	4,700	7,200	8,900	20,000
−20° F	14,000	23,000	38,000	50,000	----------
−40° F					
Viscosity Index (A. S. T. M. D-567-41)	146	144	143	141	137
Pour Point, ° F. (A. S. T. M. D-97-39)	−40	−35	−30	−25	−20
Flash Point, Open Cup, ° F. (A. S. T. M. D-92-45)	425	430	430	430	430
Do	490	500	505	510	510
Refractive Index, $N_D^{20°}$	1.448	1.449	1.450	1.450	1.451
Density, g./cc. at—					
210° F	0.930	0.935	0.938	0.939	0.942
100° F	0.975	0.980	0.983	0.984	0.987
60° F	0.992	0.996	1.000	1.001	1.003
Coefficient of Expansion per ° F. at 68° F	.00042	.00042	.00042	.00042	.00042
Specific Gravity, 20/20° C	0.991	0.995	0.999	1.000	1.002
Pounds per Gallon, 60° F	8.27	8.30	8.34	8.35	8.36
Water, percent	(1)	(1)	(1)	(1)	(1)
Ash, percent (A. S. T. M. D-482-43)	(2)	(2)	(2)	(2)	(2)
Carbon Residue	(3)	(3)	(3)	(3)	(3)

	LB-300-X	LB-400-X	LB-550-X	LB-650-X
Viscosity:				
Saybolt Seconds at—				
210° F	62.7	74.3	93.1	106
100° F	300	400	550	650
0° F	18,380	26,600	40,400	50,500
Centistokes at—				
210° F	11.0	14.1	18.8	21.9
100° F	65.0	86.6	119	141
0° F	4,000	5,800	8,800	11,000
−20° F	18,500	29,000	47,000	----------
−40° F				
Viscosity Index (A. S. T. M. D-567-41)	142	141	141	140
Pour Point, ° F. (A. S. T. M. D-97-39)	−40	−30	−25	−20
Flash Point, Open Cup, ° F. (A. S. T. M. D-92-45)	490	490	490	490
Fire Point, Open Cup, ° F. (A. S. T. M. D-92-45)	585	595	600	605
Refractive Index, $N_D^{20°}$	1.452	1.453	1.454	1.454
Density, g./cc. at—				
210° F	0.933	0.936	0.938	0.939
100° F	0.979	0.981	0.984	0.985
Coefficient of Expansion Per ° F. at 68° F.	.00042	.00042	.00042	.00042
Specific Gravity, 60/60° F	0.997	0.999	1.001	1.002
Gravity, ° API, 60° F	10.5	10.1	9.9	9.8
Pounds per Gallon, 60° F	8.30	8.32	8.33	8.34
Water, percent	(1)	(1)	(1)	(1)
Ash, percent (A. S. T. M. D-482-43)	(2)	(2)	(2)	(2)
Carbon Residue	(3)	(3)	(3)	(3)

[1] Less than 0.25%.
[2] Less than 0.01%.
[3] Less than 0.01%.

The polyalkylene glycol ether should be oil-dispersible. The polyalkylene glycol ether is incorporated in the thickened lubricant in an amount to impart high temperature stability to the grease. Ordinarily, a concentration of polyalkylene glycol ether ranging from 0.25 to about 2.5% by weight of the thickened lubricant gives satisfactory results. There is no reason to employ more polyalkylene glycol ether than is necessary, but excessive amounts do no harm, and amounts up to 5% or even higher have been successfully employed.

In general, the concentration of the imidazoline should be at least about 2.5% by weight of the aerogel but will vary depending upon the water stabilizing effect desired, the nature of the particular compound selected, the amount and nature of the gelling agent used, and the economics involved. Both static and dynamic water resistance tend to increase with time, so that an amount of imidazoline may be inadequate to impart the desired water resistance at once, yet after the grease has aged for a few weeks or months it may display excellent water resistance. Preferably, from 4 to 14% imidazoline by weight of the aerogel is used, as such amounts usually impart excellent water resistance at once. A cheap compound, of course, can be used in much larger quantities than can expensive compound, at the same total cost for the lubricant.

In some instances, the thickened lubricant may not display a long life when used continuously at high temperatures. A breakdown in high temperature stability at high temperatures if it appears is due to a decomposition, through oxidation, and in such circumstances, it is desirable to include an antioxidant in the composition. Conventional amine antioxidants which are more readily oxidized than the components of the lubricant can be employed for this purpose. Tetramethyldiaminodiphenylmethane, available under the trade name "Calco MB," is a particularly desirable antioxidant. Only small quantities are required, and ordinarily an amount ranging from 0.1 to about 1% by weight of the thickened lubricant is ample. There is no reason to employ more antioxidant than is necessary to produce the desired effect, but excessive amounts do no harm and amounts up to 5% can be used, if desired.

The composition is made simply by mixing the inorganic gelling agent, the oil, the polyalkylene glycol ether, the cationic water stabilizer and the antioxidant in any order or manner.

In one embodiment, the polyalkylene glycol ether and, desirably, the cationic water stabilizer and antioxidant, can be incorporated with the inorganic gelling agent either by mixing directly, or if desired, by dissolving them in a volatile hydrocarbon solvent, such as pentane, adding oil, mixing the solution with the inorganic gelling agent, and then evaporating the solvent.

Generally, the polyalkylene glycol ether and, optionally, cationic water stabilizer and antioxidant are dispersed in the oil and the inorganic gelling agent added thereto and mixed therewith. Any simple mixing technique can be employed, and, if desired, the mixture can be homogenized in a colloid mill, although this is not necessary.

The mixing temperature is not critical, but is preferably in the range of 100° F. to about 250° F. At higher temperatures grease yield is affected. Mixing temperatures of 140° to 160° F. are preferred.

Mixing is continued until the components are thoroughly dispersed in the oil and the consistency has attained the desired level. Any type of mixing is satisfactory. High shear supplemental mixing at a high temperature is useful.

The composition of the invention is not limited to the oil, gelling agent, imidazoline and polyalkylene glycol ether. Any of the materials conventionally added to lubricants and greases can be included. The expression "consisting essentially of" as used herein is intended to refer to the components which are essential to the composition, namely, the oil, the inorganic gelling agent, the imidazoline, and the polyalkylene glycol ether, and the expression does not exclude other components from the composition which do not render it unsuitable for lubrication, such materials being, for instance, the antioxidant, high polymers to modify viscosity or viscosity index, materials to impart tackiness, lubricating solids such as graphite, antioxidant additives, corrosion inhibitors of various types, sulfur, additives to render the lubricant suitable for use in gears, for cutting, grinding, etc.

The following examples illustrate preferred embodiments of the invention.

In the examples which follow the high temperature stability of the grease was determined by measurement of micropenetrations before and after heating to 400° F. in the "block" test. The grease was prepared for the determination of high temperature stability by placing approximately 100 cc. of grease in a 150 ml. beaker. The beaker was heated to the test temperature in an aluminum block furnace. This furnace consisted of a solid block of aluminum heated by internal electrical heaters. Six holes, each large enough to accommodate a 150 cc. beaker, were drilled in the top of the block, together with a thermocouple, so that a measure of the temperature of the block could be obtained. In this manner, six beakers could be heated simultaneously. The beakers containing the grease were placed in the aluminum furnace and held there until the equilibrium temperature of the grease reached 400° F. The samples were stirred at five-minute intervals during heating. After this the grease was allowed to cool to room temperature overnight and then was stirred vigorously with a spatula. Micropenetration measurements were obtained on the grease before and after the test procedure. The cycle was repeated as many times as desired and the results are expressed by plotting the actual penetration against the number of cycles.

The static water stability was determined as follows: A 2 x 2 inch stainless steel plate was coated with a uniform layer of the grease, following which the coated plate was immersed in a beaker filled with tap water and boiled for thirty minutes.

The dynamic water resistance of the grease was determined as follows: One hundred and fifty grams of grease was weighed into the ASTM grease worker cup. The grease then was worked 300 strokes after which a Shell penetration was obtained. Fifty percent water by weight of the grease was added, followed by a second 300 stroke working cycle. A Shell penetration was again obtained and an observation made of the type of emulsion formed. If free water remained unemulsified this was noted. Whether the emulsion was an oil-in-water or water-in-oil type was readily apparent, for the water-in-oil emulsion was shiny in appearance while the oil-in-water emulsion had a dull matte surface. If the 50 weight percent of water had been completely emulsified and no emulsion inversion had occurred an additional 50% of water on the original weight of the grease was added, the grease worked an additional 300 strokes, in the presence now of 100% water, and another Shell penetration obtained. The grease was considered to have good dynamic water resistance if, at the conclusion of the second 300 stroke working cycle, in the presence of 50% water, the emulsion had not inverted, i. e., the emulsion remained of the water-in-oil type, whether or not the water added had been completely emulsified. Dynamic water resistance was considered excellent if inversion of the emulsion had no occurred at the end of the third 300 stroke working cycle, in the presence of 100% water.

The following results are typical of the application of this test to conventional greases:

TABLE III

Base grease	Shell penetrations			Comments
	Initial	50% H_2O	100% H_2O	
Lithium hydroxystearate.	129	140	146	At 50%, all water emulsion with free water in drops. W/O.[1] Free water at 100% W/O.[1]
Aluminum	155	166	------	Considerable free water. W/O.[1]
Lime	182	206	------	Free water. W/O.[1]
Barium	141	191	------	Do.[1]
Soda	120	77	------	Do.[1]
Aerogel (Santocel ARD).	145	142	------	Emulsion inverted to O/W.[2]

[1] W/O means water-in-oil emulsion.
[2] O/W means oil-in-water emulsion.

These results show that the soap base greases tested were resistant to water, but the aerogel grease was not. Accordingly, an aerogel base grease that passes the test can be regarded as the equal of a soap base grease in dynamic water resistance under these test conditions, which closely approximate field conditions.

The ability of the grease to emulsify with free water to some extent is desirable, and therefore in evaluating the test results it is not necessary that the grease not emulsify with water, but merely that it not emulsify with water to the point where inversion to an oil-in-water emulsion occurs.

The working or mechanical stability of the greases was evaluated by 10,000 strokes' working at room temperature in the ASTM grease worker and by four hours at room temperature in the grease working assembly wherein the grease sample was subjected to the severe working afforded by close intermeshing gears in an enclosed space. This approximates many field conditions for general ball and roller bearing and transmission greases. The measurements include a Sohio micropenetration of the grease before working, a Sohio micropenetration after working, plus a Sohio micropenetration after the grease has been allowed to stand for approximately twenty-four hours, and another after the setup in grease body has been disturbed by vigorous stirring with a spatula. The last two measurements offer further data on the ability of the greases to resist mechanical breakdown.

Example 1

A grease was prepared of the following formulation at a process temperature of 265° F.:

	Percent
Santocel ARD	10.0
Ucon lubricant LB–300–X	2.0
Amine "O" [1] (2.5% by weight of the Santocel)	0.25
Paratac [2]	1.0
Paraflow [3]	0.5
Calco MB [4]	0.5
Solvent-extracted bright stock (78 SSU at 210° F.)	85.75

[1] 1-β-hydroxyethyl-2-heptadecenyl-imidazoline.
[2] An isobutylene polymer sold by the Enjay Company, Inc. and commonly used in compounding greases.
[3] A Friedel-Crafts reaction product, useful as a pour point depressant and sold by the Enjay Company, Inc.
[4] Tetramethyldiaminodiphenylmethane.

The grease was subjected to the static water stability test and rated as poor.

The storage stability of the grease was determined by allowing it to age for six months, whereupon it was tested for dynamic water resistance with the following results:

Original Shell penetration (300 strokes)	178.
Shell penetration after second 300 stroke cycle (50% water)	136.
Type of emulsion	Water-in-oil (free water remaining).
Shell penetration after third 300 stroke cycle (100% water)	130.
Type of emulsion	Water-in-oil (free water remaining unemulsified).

The aged grease had excellent dynamic water resistance. This shows that the amount of imidazoline was too low to impart dynamic water resistance at once, but was adequate after aging. Storage stability obviously was excellent.

Examples 2 to 12

A group of greases was prepared having the following formulation:

	Percent
Santocel ARD	9.0
Ucon lubricant—LB 625	2.0
Amine "O" [1]	Amount indicated in table below.
Paratac [2]	1.0
Paraflow [3]	0.5
Ortholeum 300 [4]	0.5
Solvent extracted bright stock (2000 SSU at 100° F.) [5]	89.2

[1] 1-β-hydroxyethyl-2-heptadecenyl-imidazoline.
[2] An isobutylene polymer sold by the Enjay Company, Inc. and commonly used in compounding greases.
[3] A Friedel-Crafts reaction product, useful as a pour point depressant and sold by the Enjay Company, Inc.
[4] An antioxidant for mineral lubricating oils.
[5] 46.5 volume percent solvent-extracted bright stock, 78 SSU at 210° F., and 53.5 volume percent solvent-extracted bright stock, 250 SSU at 210° F.

The oil-soluble additives, i. e., the Paratac, Paraflow and Ortholeum 300, were dissolved in the oil. The oil was brought to 95° F., the Amine "O" and Ucon added with thorough mixing and then the Santocel was blended in as rapidly as it was absorbed in the mixture. The total mixing time was sixty minutes at the temperature indicated in the table.

TABLE IV

Example No.	Percent Amine "O" [1]	Ucon LB—625, percent	Temp., ° F.		Final grease temp., ° F.	Initial Shell pen.	24 hr. Shell	ASTM penetration	
			Initial oil bath	Final oil blend				0 strokes	60 strokes
2	0	2	95	98	110	115	114	264	271
3	4	2	95	93	108	124	133	282	290
4	8	2	94	95	104	124	137	276	298
5	10	2	99	95	107	139	154	306	312
6	11	2	95	97	110	125	142	302	315
7	12	2	96	100	96	100	99	239	248
8	12	2	95	95	108	107	117	272	295
9	13	2	97	92	106	125	152	302	321
10	14	2	97	98	110	159	169	325	328

[1] Based on weight of Santocel ARD.

The table shows that in many cases the grease yield decreases as the amount of Amine "O" increases and this is a linear decrease as is evident when the results are plotted on a graph. It would be concluded from this data that the smallest amount of Amine "O" required to give the desired water resistance should be employed.

The static and dynamic water resistance properties of these greases was evaluated by the tests described above and are summarized below:

TABLE V

Example No.	Percent Amine "O" [1]	Static 30-minute boiling water test	Dynamic water resistance		
			Shell pens.		Comments
			Orig.	100% H_2O	
2	0	Fair—considerable bleeding and sep. Santocel.	114	282	Oil-in-water emulsion—inversion; complete breakdown of grease.
3	4	Good—surface slightly whitened.	133	124	Water-in-oil emulsion; free unemul. H_2O.
4	8	do	150	113	Do.
5	10	do	150	113	Do.
7	12	do	99	104	Do.
10	14	Very good	169	140	Do.

[1] Based on weight of Santocel ARD.

It is evident that as little as 4% Amine "O" by weight of the Santocel was sufficient to impart good water resistance. The results for Examples 3, 4, 5, 7 and 10 are to be contrasted with Example 2 which did not contain Amine "O" but did contain 2% of the Ucon lubricant. This shows that the Ucon lubricant alone, even at a concentration of 2%, is not able to impart the necessary dynamic water resistance.

As an additional comparison two greases were prepared containing 5 and 15% Amine "O," respectively, by weight of the Santocel, and no Ucon oil. These greases had the following composition.

	Example 11	
	A	B
	Percent	*Percent*
Santocel ARD	10.0	10.0
Amine "O"	0.5	1.5
Paratac	1.0	1.0
Paraflow	0.5	0.5
Ortholeum 300	0.5	0.5
Solvent extracted bright stock (78 SSU at 210° F.)	87.5	86.5

These greases were subjected to the static and dynamic water resistance and the high temperature stability tests. Example A had borderline static stability and very poor dynamic water resistance. Example B had excellent static and dynamic water resistance. Both these greases showed very poor high temperature stability. This shows that very large amounts of Amine "O" in the absence of Ucon oil are required to give an adequate dynamic water resistance, but that when such large amounts of Amine "O" are used, the grease has very poor temperature susceptibility properties.

In contrast, Examples 2 to 10, inclusive, passed the high temperature stability test.

The working stability of Examples 2 to 10 was determined by the four hour test in the gear grease worker. The results obtained appear in the following table:

TABLE VI

Example No.	Sohio micropenetration				Gain in penetration	
	Original	Wk'd. 4 hrs.	At rest	24 hrs. stirred	Wk'd. 4 hrs.	24 hrs stirred
2	72	97	88	128	25	56
3	88	127	103	166	39	78
4	101	115	99	169	14	68
5	119	123	100	170	4	51
6	124	116	99	170	−8	46
7	62	114	94	180	52	118
8	105	115	94	184	10	79
9	131	112	98	183	−19	52
10	134	121	106	204	−13	70

The changes of pentration shown in the table are of an order of magnitude indicating satisfactory mechanical stability. In fact, several of the greases exhibited a stiffening tendency, as exhibited by the negative penetration change after the four hour test period.

These results show that greases containing Amine "O" alone even in amounts as high as 15% by weight of the Santocel or a Ucon lubricant (mixed polyethylene-1,2-propylene glycol monomethyl ether) alone have either satisfactory dynamic water resistance or satisfactory high temperature stability, but not both. On the other hand, greases containing both the Amine "O" and the Ucon oil have excellent static and dynamic water resistance as well as good high temperature susceptibility properties even though the amount of Amine "O" is as low as 4% by weight of the Santocel and the Ucon oil is in the same amount as when used alone. Evidently in the presence of the Ucon oil the Amine "O" is more effective in smaller amounts to impart dynamic water resistance while not affecting high temperature stability. The same effect is not obtained when diethylene glycol monoethyl ether (Carbitol) is substituted for the Ucon oil, showing that this effect can be uniquely attributed to the polyalkylene glycols of higher molecular weight. This is shown by the following:

Six greases were prepared having the following formulation:

Example 12

	Percent
Santocel ARD	9.0.
Carbitol (diethylene glycol monoethyl ether)	2.0.
Amine "O"	Amount indicated in table below.
Paratac	1.0.
Paraflow	0.5.
Ortholeum 300	0.5.
Mineral lubricating oil [1] (2000 SSU at 100° F.)	85.92.

[1] 46.5 volume percent solvent-extracted bright stock (78 SSU at 210° F.), 53.5 volume percent solvent-extracted bright stock (250 SSU at 210° F.).

The Paratac, Paraflow and Ortholeum 300 were dissolved in the oil. The Santocel, Amine "O" and Carbitol then were blended and the preparation mixed at 95° F. for sixty minutes overall, including the time necessary to add the Santocel.

Shell penetrations were taken on all of the grease samples at the conclusion of the mixing cycle and a second Shell penetration obtained after the grease had stood overnight. The standard ASTM 0 and 60 stroke penetrations also were taken after the grease had stood overnight. The results are given in the following table:

TABLE VII

Example No.	Amine "O" [1]	Initial Shell penetration	24 hr. Shell penetration	24 hr. ASTM penetrations	
				0 strokes	60 strokes
	Percent				
C	6	123	120	269	274
D	8	137	136	285	290
E	10	127	125	283	291
F	12	152	159	303	316
G	14	184	190	334	344
H	16	>282	>282	400	416

[1] Based on the weight of the Santocel.

The greases were tested for high temperature stability, and showed satisfactory high temperature stability characteristics after five cycles. Furthermore, those greases containing the higher amounts of Amine "O" which had given low grease yields stiffened with the heating during the test, compensating for the low yield therein.

The water resistance characteristics of the greases were checked by a static water test and the dynamic water test in the ASTM worker. The results are shown in the following table:

TABLE VIII

Example No.	Percent Amine "O" [1]	Static 30-minute boiling water test	Dynamic water resistance		
			Shell pens.		Results
			Orig.	100% H_2O	
C	6	Very poor—considerable emulsion—loses grease characteristics.			
D	8	Poor—considerable emulsion—loses grease-like characteristics.			
E	10	Good			
F	12	do	168	218	Oil-in-water emulsion-inverted.
G	14	do	208	120	Water-in-oil free H_2O—still adhesive.
H	16	Very good	>280	108	Water-in-oil emulsion small amount free H_2O—still adhesive.

[1] Based on weight of Santocel ARD.

If the grease did not pass the static test, the dynamic water test was not carried out since this would have been useless.

The results show that with Carbitol at least 10% Amine "O" is necessary for satisfactory performance in the static boiling water and that at least 14% is necessary in the dynamic water test. This shows that Carbitol, diethylene glycol monoethyl ether having two ethyl-

ene glycol units, is not as effective as the higher polyalkylene glycol ethers such as the Ucons, which are polymers of ethylene and 1,2-propylene glycol units, in cooperating with the Amine "O" to give good dynamic water resistance.

Examples 13 to 17

Five grease compositions were prepared of the following formula:

	Weight percent	
	Examples 13, 14, 15, 16	Example 17
Santocel ARD	9.0	9.0
Amine "O"	[1] 0.72	[1] 0.72
Ucon LB-625	2.0	2.0
Paratac	1.0	1.0
Paraflow PDX	0.5	0.5
Ortholeum 300 (Du Pont)	0.5	0.5
Solvent extracted bright stock [2] (2,000 SSU at 100° F.)	86.28	
Solvent extracted bright stock (78 SSU at 210° F.)		86.28

[1] 8% by weight of Santocel.
[2] 46.5 volume percent 78 SSU at 210° F., 53.5 volume percent 250 SSU at 210° F.

The oil-soluble additives were dissolved in the oil, then the Amine "O" and Ucon were mixed in and finally the Santocel was added to the blend incrementally as rapidly as possible. Mixing was continued at the temperature and for the times indicated in the table.

TABLE IX

Example No.	Bath temp., ° F.		Grease temp., ° F.		Shell pens., time in minutes				
	Initial	Final	Initial	Final	30	60	90	120	
13	100	95	90	108	151	159	165	165	Immediate.
					175	182	189	194	24-hour.
14	150	140	150	138	119	153	153	158	Immediate.
					168	179	194	196	24-hour.
15	250	250	248	256					
					255	>300			24-hour.
16	160	150	162	160	174	189	194	204	Initial.
					189	190	[1] 215	242	24-hour.

[1] Mixer oil bath temperature and grease temperature accidentally allowed to rise to 170° F.

The results for Examples 13, 14 and 16 show that no effect in grease yield is obtained by increasing the processing temperature from 95 to 150–160° F. When the processing temperature is increased to 250° F. a noticeable drop in grease yield is apparent. The immediate penetration values for Examples 13 and 14 are lower than the values obtained after twenty-four hours. This is attributed to the fact that these greases are stiffer while hot.

Example 15 was too soft after one hundred and twenty minutes of paddle mixing to fall within the range of penetrometer measurement. Therefore it was further processed by one pass through a homogenizer. This

achieved a distinct increase in grease consistency, showing that a homogenization is quite effective as a means for increasing the grease yield following processing at high temperatures.

Another portion of the grease of Example 15 was recycled through a gear pump. The grease possessed an original Shell penetration of 242. After recycling in a gear pump for six to seven minutes, the grease was pumped out. The Shell penetration was 183. After repeating the cycle again the Shell penetration reached 171, but the grease temperature was 122° F. After permitting the grease to stand overnight, a Shell penetration obtained at room temperature was 180. This shows that gear pump cycling also is effective to increase the grease yield after processing at a high temperature.

The grease prepared using the 78 SSU bright stock was processed at 95° F. and the mixing time was limited to sixty minutes. The ASTM penetrations appear below:

Example 17

Time	ASTM pen.		Temperature, °F. (initial Temp. 90° F.)
	0	60	
15 min	307	311	104
30 min	321	324	102
60 min	327	331	100

The working or mechanical stability of these greases was evaluated in the ASTM worker and by four hours' working in the gear working assembly. The results are shown in the following tables:

TABLE X

10,000-STROKE WORKING STABILITY

Ex. No.	Processing Temperature	ASTM Pens., No. Strokes						Δ Pen.	
		0	60	2,500	5,000	7,500	10,000	0–60	60–10[3]
13	95° F.	319	334	327	324	325	323	15	−11
14	150° F.	335	342	327	319	324	316	7	−26
15	250° F. (homogenized).	267	319	333	335	338	[1] 342	52	23
16	160–170° F. (gear pumped).	330	337	340	339	334	343	7	6
17	95° F.	325	339	332	332	332	338	14	−1

[1] After standing undisturbed overnight, pen.—298.

TABLE XI

LABORATORY GEAR WORKING STABILITY

Example No.	Sohio micropenetrations				Δ penetration	
	Original	4-hr.	24-hr.	24-hr.-stir	0–4 hrs.	0–24 hr.-stir
13	138	139	115	187	1	49
14	130	112	103	158	−18	28
15	93	123	113	157	30	64
17	117	146	139	188	29	71

All of the greases had very satisfactory performance in each of the tests.

The dynamic water stability resistance of the greases was evaluated with the following results:

TABLE XII

DYNAMIC WATER STABILITY

Example No.	Shell penetrations		Δ pen.	Approximate percent H_2O emulsion
	Original	600 strokes w. 100% H_2O		
13	181	140	−41	60
14	185	121	−64	55
15	145	134	−11	55
16	180	135	−45	65
17	187	154	−33	70

In all cases the emulsions remained water-in-oil and the products retained their greaselike characteristics even though the greases emulsified with approximately 55 to 70% of water.

Another sample of Example 14 was subjected to incremental addition of water with working in the same test. The grease became stiffer upon emulsifying the first 5% of water into the grease structure. Beyond this point the consistency of the grease was fairly stable. The test was halted after 80% water had been added. The grease continued to emulsify water until 60% water had been added after which free water remained present in the worker.

The greases of Examples 13, 14, 15 and 17 were evaluated for high temperature characteristics and were run in duplicate for five complete cycles. The greases of Examples 14, 15 and 17 showed excellent high temperature stability. The grease of Example 13 had borderline performance which is attributed to the low process temperature used. The high temperature stability would be improved by a further heat treatment of the grease. Best overall high temperature stability was shown by the grease of Example 14, but the grease of Example 15 also was excellent in this respect.

The grease of Example 13 was evaluated by a dynamic rust test. No rusting was evident on the balls or on the entire top race and inside face of the bottom race. The grease of Example 13 was given an A rating in the test. In comparison, an aerogel grease of the same formulation but without the Ucon oil failed the test with a D rating.

The grease of Example 14 was evaluated by the ANG–15 bearing washout test to evaluate the resistance to water washout of the grease from a rotating bearing. The duration of the test is one hour, after which the loss in weight of the grease from the bearing is determined. The test ran the full hour and most of the grease in the bearing had become emulsified with water. Hardly any grease was evident in the wash water. In contrast, a test run with an aerogel grease of the same formulation without the Ucon oil ran for approximately ten minutes, after which failure was evidenced by loud squealing noises from the bearing. Almost all of the grease had emulsified and inverted and the washout from the bearing itself appeared in the wash water as a milky emulsion.

The thickened lubricants of the invention are useful in many field grease applications. In the critical field of wheel bearing lubricants, these greases have performed very satisfactorily. Other successful field applications include chassis lubrication, general ball and roller bearing applications, foundry ladle trunnion bearings, phonograph bearings, textile spinning wheels, cam followers, kiln car bearings, farm equipment, worm gear-radar antennae, outdoor playground equipment, shaker screens and ring gears and mixing vessels.

The greases of the invention are characterized by their ease and reproduceability of preparation and their high static and dynamic water resistance and high temperature consistency stability. In addition they have excellent oxidation resistance and mechanical stability. All of these are requisites for a multipurpose lubricating grease.

Testing the high temperature stability of the thickened lubricants of the invention by heating the greases to 400° F. is an extreme test inasmuch as the highest temperature to which a grease is subjected under even extraordinary conditions of use is about 300° F., but the temperature was adopted as a suitable test standard because a grease stable at 400° F. definitely will have the stability necessary to withstand heating to 300° F. It will be understood that for normal purposes the thickened lubricants of the invention need not be stable at temperatures above about 300° F. and that the greases of the invention at least meet this requirement. Where the term "high temperature stability" is used, it will be understood to mean that the thickened lubricant is stable against loss of consistency at temperatures of at least 300° F.

The Shell penetrations are in accordance with the Shell Microcone Penetration Test, Institute Spokesman (NLGI), volume VI, Number 12, page 1 (1943).

The Sohio micropenetration technique employed required a microcone and cup. The microcone was specially built, and its dimensions are compared in Table II with those of the standard ASTM cone, ASTM Designation 217–48, described on page 143 of the November 1948 edition of D-2 Specifications for Petroleum Products. The cone and grease cup employed in obtaining the test results required a minimum sample size of 35 ml.

TABLE XIII

MICROCONE DIMENSIONS

	ASTM		SOHIO	
	Cone	Cup	Cone	Cup
Diameter, mm	65	78	20	43
Height, mm	45		17	
Depth, mm		65		24.5
Surface, sq. mm	7,070	4,778	620	1,470
Volume, cc		290		35.6
Cone diam./cup surface		0.136		0.136
Cone height/cup depth		0.693		0.693
Weight of assembly, gms	150		[1] 13	
Weight of assembly/sq. mm. cone surface		0.021		0.021

[1] Calculated.

All parts and percentages are by weight of the thickened lubricant unless otherwise indicated.

We claim:

1. A thickened lubricant of high temperature stability and good dynamic water resistance, consisting essentially of a mineral lubricating oil of lubricating viscosity as the major component, a finely-divided inorganic water-susceptible oil thickener in an amount sufficient to impart a grease consistency to the oil, a water-insoluble oil-miscible polyalkylene glycol ether having one terminal hydroxy group and a sufficient proportion of oxyalkylene units of at least three carbon atoms to impart water-insolubility, and a viscosity at 100° F. within the range of 285 to 1145 SSU, and a water-insoluble oil-dispersible cationic surface-active imidazoline having an aliphatic group substituted at the 2-position and a hydrophilic group substituted at the 1-position of the imidazoline, said glycol ether being in an amount within the range from 0.25 to about 2.5% by weight of the thickened lubricant, and said imidazoline being in an amount within the range from 4 to 14% by weight of the inorganic water-susceptible oil thickener, each being in amounts sufficient in combination to impart high temperature stability and dynamic water resistance to the thickened lubricant.

2. A thickened lubricant in accordance with claim 1 in which the polyalkylene glycol ether is a polypropylene glycol ether.

3. A thickened lubricant in accordance with claim 1 in which the imidazoline is 1-β-hydroxyethyl-2-heptadecenyl imidazoline.

4. A thickened lubricant in accordance with claim 1 in which the inorganic oil thickener is a silica aerogel.

5. A thickened lubricant of high temperature stability and good dynamic water resistance, consisting essentially of a mineral lubricating oil of lubricating viscosity as the major component, a finely-divided silica aerogel in an amount sufficient to impart a grease consistency to the oil, a water-insoluble oil-miscible mixed polypropylene glycol ether having one terminal hydroxy group and a sufficient proportion of oxyalkylene units of at least three carbon atoms to impart water-insolubility, and a viscosity at 100° F. within the range of 285 to 1145 SSU, and a water-insoluble oil-dispersible cationic surface-active imidazoline having an aliphatic group substituted at the 2-position and a hydrophilic group substituted at the 1-position of the imidazoline, said glycol ether being in an amount within the range from 0.25 to about 2.5% by weight of the thickened lubricant, and said imidazoline being in an amount within the range from 4 to 14% by weight of the inorganic water-susceptible oil thickener, each being in amounts sufficient in combination to impart high temperature stability and dynamic water resistance to the thickened lubricant.

6. A thickened lubricant in accordance with claim 5, in which the imidazoline is 1-β-hydroxyethyl-2-heptadecenyl imidazoline.

7. A thickened lubricant in accordance with claim 5, in which the polypropylene glycol ether is a mixed polyethylene 1,2-propylene glycol ether.

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