

1

3,340,190

RAILWAY DIESEL OIL

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This invention relates to an alkaline lubricating oil and more particularly pertains to lubricating oils of high dispersancy-detergency and high alkalinity for use as crank-case lubricant in heavy duty diesel engines such as railway freight diesel engines.

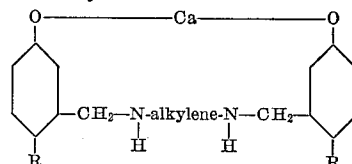
Heavy duty diesel engines requires crankcase lubricant oils which are stabilized against oxidation, are non-corrosive to silver bearings, keep in suspension combustion products which would deposit and/or form as sludge and/or varnish on pistons, cylinder liners and undercrown cavities. The diesel crankcase lubricant should prevent carbon deposition in the top ring piston grooves. The alkalinity life of the crankcase lubricating oil should be long for the purpose of reducing the need to add alkalinity imparting additives before complete change of crankcase lubricant oil or to reduce the frequency of changes of crankcase lubricating oil. In addition the crankcase lubricating oils for heavy duty diesel engines must be so formulated that silver connecting rod bearings and lead surfacing thereon are not attacked either by the additives in the oil formulation or by the dispersed, neutralized or decomposition products thereon during extended use.

The present invention is directed to a unique combination of lubricant oil addition agents dissolved in the required base oils for heavy duty diesel engine crankcase lubrication. The diesel engine crankcase lubricant compositions of this invention when used in field testing in new high speed railway diesel freight engines have demonstrated their ability to maintain a clean engine, provide reserve alkalinity in the used crankcase oil and protect the lead surfaced silver connecting rod bearing even after 77,000 miles of high speed freight service. Most of the parts of one engine replicate of several under test at the same time on the same oil formulation when disassembled showed negligible wear by precise micrometer measurements. That is, after 77,000 miles of high speed services most of the used engine's internal parts were within newly manufactured part tolerance ranges and none showed sufficiently high wear to warrant replacement.

The unique combination of lubricant addition agents according to this invention is a mixture of the exactly neutralized calcium salt of a N,N'-bis(alkyl substituted hydroxybenzyl) alkylene diamine and a bis (alkenylsuccinimide) of polyalkylene polyamine or of a urea condensation derivative of polyalkylene polyamine, N,N'-bis(polyazalkylamino) ureylene. By exactly neutralized calcium salt of N,N'-bis(alkyl substituted hydroxybenzyl) alkylene diamine is meant that calcium salt which contains one gram atom calcium for two molecular equivalents of hydroxyl group, that is one calcium replaces one hydrogen on each of two OH groups. The N,N'-bis (alkyl substituted hydroxybenzyl) alkylene diamines are well known compounds and are generally derived by reacting two moles alkyl substituted phenol, two moles formaldehyde and one mole alkylene diamine such as ethylene diamine, 1,2-diamino-propane, 1,3-diamino-propane, 1,2-diamino-butane, 1,3-diamino-butane, 1,4-diamino-butane, diamino-pentanes, diamino-hexanes, etc. Desirably, these alkylene diamines contain 2 to 10 carbon atoms. Also the alkyl substituent on the benzene ring of the starting phenol and therefore in the N,N'-bis (alkyl substituted hydroxybenzyl) alkylene diamine is de-

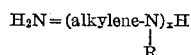
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sirably of a size (carbon number) to impart oil solubility to the calcium salt. Preferably, the alkyl substituent is alkyl hydrocarbon containing 5 to 70 carbon atoms. Thus the aforementioned exactly neutralized calcium salt can be illustrated by the formula:



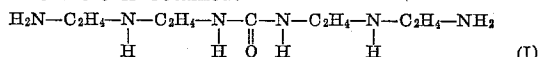
wherein "akylene" is a divalent C₂ to C₁₀ open chain hydrocarbon and R is a C₅ to C₇₀ alkyl hydrocarbon.

The bis(alkenylsuccinimide) of polyalkylene polyamines or of urea polyalkylene polyamine condensation derivative are not as easily illustrated by structural formula. In general, they are obtained by reacting about two moles of alkenyl (or alkyl) substituted succinic anhydride with one mole of polyalkylene polyamine. The alkenyl or alkyl substituent on the bis-succinimide also must be of a size (carbon number) to impart oil solubility. For this purpose alkyl or alkenyl groups having at least 30 carbon atoms (about 420 molecular weight) up to 215 carbon atoms (about 3000 molecular weight) are desired. Such C_{30} to C_{215} alkyl or alkenyl succinic anhydrides are obtained by reacting in a known manner maleic anhydride with wax hydrocarbons, low molecular weight (M of 420 to 3000) polymers of propylene, isobutylene or copolymers of propylene or isobutylene with a different C_2 to C_5 mono-olefin. The polyalkylene polyamine can be considered a product of the reaction of ammonia and a dihaloalkane, i.e., dichlorethane. Thus the polyalkylene polyamines can be illustrated by the formula:

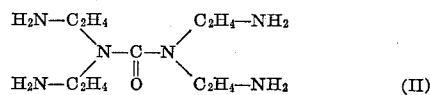


wherein "alkylene" is a divalent, open chain C_2 to C_{10} hydrocarbon, R is lower alkyl hydrocarbon (i.e. C_1 to C_4) and x is a number of from 2 to 10. Illustrative of these polyalkylene polyamines include diethylene triamine, triethylene tetramine, tetraethylene pentamine, pentaethylene hexamine, heptaethylene octamines (3, 6, 9, 12, 15, 18-hexazeicosane-1,20-diamine), dipropylene triamine, tripropylene tetramine, tetrapropylene pentamine, pentapropylene hexamine and heptapropylene octamine (4, 8, 12, 16, 20, 24-hexazaheptacosane-1,27-diamine) among others.

The urea-polyalkylene polyamine condensation product (polyazalkyl amino) ureylene is obtained according to the method of a contemporary by reacting two moles of polyalkylene polyamine with one mole of urea at temperatures up to about 500° F. accompanied by the evolution of two moles ammonia. From urea and diethylene triamine there is obtained:

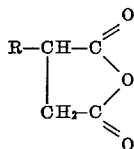


which can be named N,N'-bis(5-amino-3-azaheptyl) ureylene. For the preparation of these urea-polyalkylene polyamine condensation products the polyalkylene polyamines hereinbefore defined by structural formula are desirable. It will be appreciated that the liberation of ammonia need not come from the terminal NH_2 group of the polyalkylene polyamine but may well come from urea and thus the carboxyl group of urea may be linked to one of the secondary nitrogens in the chain as in:



which can be named 1,3-tetra(2-aminoethyl) urea. From

the condensation of two moles polyalkylene polyamine (e.g. diethylene triamine) with one mole urea there can be mixtures of (I) and (II) as well as mixtures of (I), (II) and N-(5-amino-3-azaheptyl) N'-(2-amino-ethyl) ureylene. Such mixtures would provide reactive primary amino (NH₂) groups for reaction with the alkyl or alkenyl substituted succinic anhydride:



wherein R is a C₃₀ to C₂₁₅ alkyl or alkenyl hydrocarbon substituent as hereinbefore disclosed to form a disuccinimide.

The preferred combinations of addition agents are the exactly neutralized calcium salt of N,N'-bis(C₅ to C₇₀ alkyl substituted hydroxybenzyl) ethylene diamine with bis(C₃₀ to C₂₁₅ alkenylsuccinimide) of tetraethylene pentamine or with bis(C₃₀ to C₂₁₅ alkenyl succinimide) of the N,N'-bis(polyazalkylamino) ureylene derived from two moles tetraethylene pentamine condensed with one mole urea. The most preferred exactly neutralized calcium salts are of N,N'-bis(tert.-octylhydroxybenzyl) ethylene diamine, N,N'-bis(nonylhydroxybenzyl) ethylene diamine and N,N'-bis(C₂₈ alkyl hydroxybenzyl) ethylene diamine.

TABLE I

	Present Invention Formulation LF	Commercial Formulation A
Viscosity Index.....	73	67
Total Base Number ¹	7.5	6.2
pH ¹	11.6	10.4
Calcium-Weight percent.....	0.16	0.21

¹ Method of ASTM-D-664.

Although Present Invention Formulation LF has a higher total base number than Commercial Formulation A, the latter has the higher, more than 31% higher, calcium content.

The EMD Corrosion and Oxidation Test is conducted with fresh Present Invention Formulation LF at 285° F. and at 325° F. both for 72 hours. At each of these temperatures 300 grams of fresh formulated lubricant composition is stirred with a "z" stirrer at 300 r.p.m. with weighed silver and copper corrosion specimens. This test determines viscosity changes (measured as increase at 100° F. in S.S.U.), loss of silver and copper, pentane insolubles as oxidation products, pH change as indication of extent of oxidation and rejection of alkalinity and total base number. The results of this test conducted with the foregoing oil formulation are shown in Table II.

TABLE II.—EMD CORROSION AND OXIDATION TEST

Values Measured	Present Invention Formulation LF				
	Fresh	72 hrs.-285° F.	Demerit Rating	72 hrs.-325° F.	Demerit Rating
Ag. loss, mg.....		0.7		0.5	
Cu loss, mg.....		3.7		11.8	
100° F. viscosity, S.S.U.....	1,020	1,094	2.5	1,214	6.5
Viscosity increase percent.....		7.3	2.5	19	6.3
Pentane Insolubles, percent.....		0.0	0.0	0.0	0.0
pH.....	11.6	10.1	5.0	9.2	7.9
Total Base Number.....	7.7	4.5	12.7	2.1	22.4
Total Acid Number.....	0.0	0.2	0.6	0.3	1.1
Total Demerits.....			23.3		44.2

Alkalinity of the lubricant oil formulation is provided the lubricant oil formulation by both the bis-succinimide and the calcium salt of the bis(alkyl hydroxybenzyl) diamine. However, each of these additive essentials in the unique combination of this invention provide different alkalinity types which appear to enhance each other in a manner not fully understood. For example a diesel crankcase lubricant oil formulation is prepared using the unique combination of additives of this invention and tested in a corrosion and oxidation test established by the Electro-Motive Division of General Motors Corporation (EMD Corrosion and Oxidation Test) as a performance test for candidate crankcase lubricants for further testing engines. For comparison, there is presented alkalinity characteristics of a commercially accepted and used railway diesel crankcase lubricant composition, a proprietary composition of a formulator different than the assignee of this invention, containing a high based oil-soluble calcium sulfonate (a calcium sulfonate complex containing more calcium than in an exactly neutralized calcium sulfonate) and a calcium salt of a bis(alkyl hydroxybenzyl) alkylene polyamine or of a poly(alkyl hydroxybenzyl) amine of a polyethylene imine having a molecular weight of 30,000 to 40,000. These two lubricant oil formulations are hereinafter designated "Present Invention Formulation LF," and "Commercial Formulation A," respectively. These two lubricant oil formulations have the following alkalinity characteristics:

The above data indicate that against a permissible demerit rating of 25 at 285° F. and 50 at 325° F., the test oil showed a demerit rating of 23.3 at 285° F. and 44.2 at 325° F., which indicates a highly satisfactory lubricant as evidenced by this test procedure.

For the purpose of this invention to achieve long alkalinity life there should be a certain ratio range of the bis-succinimide, to calcium salt of bis(alkyl hydroxybenzyl) ethylene diamine to provide the unique combination of these additives and obtain the mutual compatibility of each other without detracting adversely from the usefulness of the other. The foregoing ratio to provide the unique combination of additives according to this invention can be expressed as range of mole ratio of bis-succinimide to range of gram atom calcium to take into consideration the fact that the bis-succinimide not only can vary in molecular weight as its substituted succinic anhydride moiety and its polyamine moiety vary in molecular weight as well as the fact that various concentrates of solutions of the bis-succinimides can be used as a blending agent. Likewise, the calcium salt, as hereinbefore indicated, can vary in molecular weight as the alkyl substituent on the phenol moiety varies. Also the calcium salt content of the concentrates used for blending can vary. To meet these situations on a more consistent basis it is therefore more fitting to define the combination of additives of this invention on the foregoing basis of

mole range of bis-succinimide to calcium gram atom range.

To provide the unique combination of bis-succinimide and calcium salt of bis(alkyl hydroxybenzyl) ethylene diamine there are employed in the range of from 0.001 to 0.01 mole for each 0.025 to 0.05 gram atom of calcium in the calcium salt. More concisely stated, the foregoing ratios are a range of from 0.02 to 0.4 mole bis-succinimide per gram atom of calcium. These ratios of mole ratio range to gram atom range are applicable for additive concentrates, containing 10 to 85% of each additive, to be diluted with lubricant base oil of the 60 to 75 viscosity index range. Usually blends of SAE 30, 40 and/or 50 weight oils are used to prepare railway diesel engine crankcase lubricant oil compositions. The formulation hereinbefore described as "Present Invention Formulation LF" contains the bis-succinimide and calcium salt within said ratios of mole bis-succinimide range to gram atom calcium range. Formulations having said ratios of mole ratio range to gram atom range are prepared from the following specific bis-succinimides and calcium salt of bis(alkyl hydroxybenzyl) ethylene diamines:

Bis-Succinimide A.—Solution of 40 weight percent of borated (0.67 weight percent boron) bis-succinimide of tetraethylene pentamine wherein the succinimide groups are derived from 960 molecular weight alkenyl substituted succinic anhydride.

Bis-Succinimide B.—Solution of 50 weight percent of bis-succinimide of tetraethylene pentamine wherein the succinimide groups are derived from 960 molecular weight alkenyl substituted succinic anhydride.

Bis-Succinimide C.—Solution of 45 weight percent bis-succinimide of amine product of two moles tetraethylene pentamine with one mole urea and the succinimide groups are derived from 960 molecular weight alkenyl substituted succinic anhydride.

Bis-Succinimide D.—Solution of 50 weight percent of borated bis-succinimide described for Bis-Succinimide C having a boron to nitrogen weight ratio of about 0.35.

Calcium amine-coupled phenate.—A 40 weight percent solution of exactly neutralized calcium salt of N,N'bis (nonylhydroxybenzyl) ethylene diamine.

Typical concentrations of this invention from the foregoing additives as those resulting from the amounts of each indicated to prepare 100 pounds of concentrate:

Concentrate A:	Pounds
Bis-Succinimide A	17
Calcium amine-coupled phenate	83
Concentrate B:	
Bis-Succinimide A	41.2
Calcium amine-coupled phenate	58.8
Concentrate C:	
Bis-Succinimide B	17
Calcium amine-coupled phenate	83
Concentrate D:	
Bis-Succinimide B	41.2
Calcium amine-coupled phenate	58.8
Concentrate E:	
Bis-Succinimide C	17
Calcium amine-coupled phenate	83
Concentrate F:	
Bis-Succinimide C	41.2
Calcium amine-coupled phenate	58.8
Concentrate G:	
Bis-Succinimide D	17
Calcium amine-coupled phenate	83
Concentrate H:	
Bis-Succinimide D	41.2
Calcium amine-coupled phenate	58.8

The foregoing concentrates contain the mole of bis-succinimide and gram atom (g.a.) of calcium as shown in Table III.

TABLE III

MOLE BIS-SUCCINIMIDE AND GRAM ATOM CALCIUM PER 100 GRAMS CONCENTRATE

Concentrate	Bis-Succinimide-Mole	Calcium-Gram Atom	Mole per g.a. Ca
A	0.0028	0.059	0.047
B	0.0068	0.042	0.16
C	0.0034	0.059	0.058
D	0.0081	0.042	0.198
E	0.0026	0.059	0.044
F	0.0063	0.042	0.15
G	0.003	0.059	0.051
H	0.0074	0.042	0.176

The foregoing concentrates are diluted with a base oil blend having a viscosity index of 73. In the following formulations said blend is referred to as "73 VI Base Oil."

Formulation 1:	Pounds
Concentrate A	100
73 VI Base Oil	1450
Formulation 2:	
Concentrate B	100
73 VI Base Oil	900
Formulation 3:	
Concentrate C	100
67 VI Base Oil	1450
Formulation 4:	
Concentrate D	100
68 VI Base Oil	900
Formulation 5:	
Concentrate E	100
71 VI Base Oil	1450
Formulation 6:	
Concentrate F	100
73 VI Base Oil	900
Formulation 7:	
Concentrate G	100
73 VI Base Oil	1450
Formulation 8:	
Concentrate H	100
73 VI Base Oil	900

In the foregoing Formulations 1, 3, 5 and 7 have about 1.0 weight percent of the respective bis-succinimides and Formulations 2, 4, 6 and 8 have about 4.0 weight percent bis-succinimides.

The combination of addition agents of this invention give outstanding performance in diesel engine crankcase lubricating oil compositions in actual service conducted under severe conditions. To illustrate this, the results of the use of a typical formulation, e.g., Formulation 1 is herein-after set forth. The test employed is known as the 300 hour EMD 2-567 Engine Performance Test (EMD is Electro-Motive Division of General Motors). In this test the diesel engine crankcase of a clean engine having new moving parts and bearings is filled with the lubricating oil composition. The engine is started and operated for 9 hours and 20 minutes under break in conditions at the end of which an engine speed of 835 r.p.m. and load of 210 horsepower is achieved. Then the engine is operated at 835 r.p.m. with a steady 210 horsepower load, engine cooling water at 195° F. and oil sump temperature of 240° F. for 300 hours. During the test samples of the used oil are taken at 100, 200 and 300 hours of operation. These samples are subject to evaluation for SSU (Saybolt Seconds Universal) at 100° F. and 210° F. to determine percent viscosity change, the weight percent pentane and benzene insolubles content, the total acid number, the total base number and pH of the used oil composition. At the end of the 300 hour test under load, the engine is disassembled and its parts are inspected for piston groove filling, deposits on land above top ring, deposit under piston crown, piston skirt discoloration (evaluated on a 0 to 800 scale where 0 is clean and 800 is black), oil filter screen deposits and filter condition, bearings performance, and general corrosion as well as corrosion of bronze thrust washer

and copper-lead camshaft bearings. The results of this test are as follows:

EMD 2-567 ENGINE PERFORMANCE

Engine deposits:

Piston ring groove filling, percent:	
Top ring -----	73
No. 2 ring -----	90
No. 3 ring -----	36
Land above top ring, percent filled -----	13
Piston discoloration No.:	
(0=clean, 800=black) -----	740
Undercrown deposit thickness, in. -----	<0.002
Oil screen deposits, percent -----	None
Oil filter condition -----	Good

Bearing performance:

Silver bearing type -----	D
Right bearing weight loss 0-300 hours, gram -	0.24
Weight loss leaded insert (lead overlay intact) -	0.57
Metallurgical inspection of unleaded insert at 300 hours—no silver corrosion.	
Bronze thrust washer average weight loss, gram -----	0.32
Metallurgical inspection—no sulfidic attack.	
Copper-lead camshaft bearings average weight loss 0-300 hours, gram -----	0.02
Metallurgical inspection—no corrosion.	

EMD 2-567 ENGINE PERFORMANCE (CONTINUED)
[Used Oil Inspections]

	Viscosity, SSU	
	100° F.	210° F.
Hours:		
0 -----	1,038	79.7
100 -----	1,116	82.7
200 -----	1,141	84.0
300 -----	1,166	84.9
Percent Viscosity Increase at 300 Hours -----	12	7

	Insolubles, Percent	
	Pentane	Benzene
Hours:		
100 -----	0.10	0.10
200 -----	0.09	0.05
300 -----	0.15	0.07

	Acidity		
	Total Acid Number	Total Base Number	pH
Hours:			
0 -----	0	7.80	11.1
100 -----	0.79	3.01	8.6
200 -----	0.95	2.16	8.4
300 -----	0.89	1.68	8.8

A lubricant oil composition of this invention was field tested as crankcase lubricating oil in eight new diesel electric freight locomotives each equipped with one Electro-Motive Division of General Motors 16 cylinder type 567-D3A turbo-charged diesel engine. After delivery to Railroad Company the initial fill crankcase oil was drained and filled with lubricant oil Formulation 1. These freight locomotives were placed in transcontinental high speed service. At the end of about five months service the mileage of use of the eight locomotives varied from 68,000 to 80,000 miles. At this time one locomotive, hereinafter referred to as Locomotive 85, was available in the locomotive shops of Railroad Company for a field test interim inspection. Locomotive 85 had accumulated about 77,000 miles of service with about 80% of working

time in eighth throttle notch, full power setting of throttle and had experienced no oil drain and refills.

Inspections were made of top deck, cover and air box of Locomotive 85. Negligible deposits were found on the top deck and cover and these deposits were easily removed by wiping which left clean bare metal. Deposits in air boxes were only thin where deposits were found tapering to clean metal areas.

The condition of pistons and rings were evaluated by inspection through engine ports. All of the piston rings showed initial machinery marks which is indicative of low wear. All the rings were bright, metallic in appearance indicative of satisfactory ring function.

Power assemblies in positions Numbers 6 and 14 were removed for deposit and wear measurements. The excellent results are seen from the following summarized evaluations:

ENGINE DEPOSIT RATINGS ^a

Piston:	
Ring + groove deposits, percent filled ^b :	
Number 1 -----	85
Number 2 -----	60
Number 3 -----	40
Number 4 -----	10
Number 5 -----	0
Number 6 -----	0
Skirt deposits ^c -----	190
Undercrown deposits, average thickness, inches -	0.004

Liner port restriction—None.
Valve deposits—Negligible.
Top deck—Wipeable oil, clean.
Top deck cover—Wipeable oil, clean.

^a Average of two power assemblies.
^b 100% filled=0.054".
^c 0=no deposit, 400=100% covered brown deposit, 800=100% covered black deposit.

ENGINE WEAR MEASUREMENTS ¹
[Inch]

		Typical New Part
Ring Gaps: ²		
No. 1 -----	0.045	0.04
Avg. 1, 2, 3, 4 -----	0.042	0.04
Ring-Groove Clearance:		
No. 1 -----	0.009	0.007
Avg. 2, 3, 4 -----	0.010	0.010
Piston Groove Width:		
No. 1 -----	0.198	0.196
Avg. 1, 2, 3, 4 -----	0.196	0.196
Piston Skirt Diameter -----	8.486	8.489
Cylinder Liner Diameter -----	8.502	8.501
Thrust Washer -----	0.186	0.186
Silver Bearing -----	(³)	(³)

¹ Average of two power assemblies.
² Machine marks 100% visible.
³ Lead overlay 90% intact. No corrosion or wiping of exposed silver.

The data show that the engine was exceptionally clean. There were moderate deposits in compression ring grooves; however, the oil control rings and grooves were completely clean. Piston skirts were clean with skirt deposits covering less than 20% of the total area. Inlet port and valve deposits were negligible.

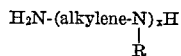
The silver bearings were in excellent condition. The lead flashing still covered about 90% of the total area, and where free silver was exposed, there was no evidence of corrosion or wiping. The rod bearings and slipper bearing surface showed no evidence of wear. The thrust washer also looked satisfactory and measured within new tolerance limits. The pistons, rings and liners showed little or no wear. The addition of about 0.2 to 1.0 desirably 0.3 to 0.8 and preferably 0.4 to 0.8, weight percent oil-soluble calcium salt of alkyl (C₁₂ to C₃₀) benzene sulfonic acid to Formulations 1 through 4 may favorably effect reduction of ring carbon deposit.

As indicated in the description of bis-succinimides A and D, the bis-succinimide product of reacting the alkyl

or alkenyl substituted succinic anhydride with polyalkylene polyamine or preferably the polyalkylene polyamine-urea condensation product can be modified by boration. This boration is accomplished by the use of boron acid in an amount to provide 0.1 to 1.0 weight percent boron.

What is claimed is:

1. A lubricant composition comprising a lubricating mineral oil having dissolved therein as its essential additive ingredients a combination consisting of the exactly neutralized calcium salt of a N,N'-bis(alkyl substituted hydroxybenzyl) alkylene diamine and a bis(alkenyl substituted succinimide) of a (polyazalkyl amino)-ureylene that is a condensation product of two moles of polyalkylene polyamine with one mole urea, wherein said alkyl substituted hydroxybenzyl the alkyl substituent is alkyl hydrocarbon of from 5 to 70 carbon atoms, wherein said alkylene diamine the alkylene group is a divalent open chain hydrocarbon of from 2 to 10 carbon atoms, wherein said alkenyl substituent of said succinimide has from 30 to 215 carbon atoms and wherein said polyalkylene polyamine has the formula



wherein "alkylene" is a divalent open chain hydrocarbon of from 2 to 10 carbon atoms, R is selected from the class consisting of hydrogen and lower alkyl hydrocarbon of 1 to 4 carbon atoms and x is a number of from 2 to 10; wherein said combination there is present in the range of from 0.02 to 0.4 mole of said bis(alkenyl substituted succinimide) per gram atom of calcium of said exactly neutralized calcium salt; and wherein said lubricant composition there is present from 0.5 to 10 weight percent of said bis(alkenyl substituted succinimide).

2. The composition of claim 1 wherein there is present said bis(alkenyl substituted succinimide) in the range of from 1.0 to 5.0 weight percent.

3. The composition of claim 1 wherein the lubricating mineral oil has a viscosity index of 60 to 75 and there is present from 0.5 up to 10 weight percent of said bis(alkenyl substituted succinimide).

4. The composition of claim 1 wherein said bis(alkenyl substituted succinimide) is as bis(polybutenyl substituted succinimide) whose polybutenyl succinic anhydride moiety has a molecular weight of about 960 and is the succinimide of the amine derived by reacting tetraethylene pentamine with urea in the ratio of two moles of said pentamine per mole urea accompanied by the evolution of two moles ammonia for each mole urea.

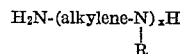
5. The composition of claim 4 wherein said bis-succinimide is modified with 0.5 to 0.7 weight percent of boron.

6. The composition of claim 1 wherein said exactly neutralized calcium salt is the exactly neutralized calcium salt of N,N'-bis(nonyl hydroxybenzyl) ethylene diamine.

7. The composition of claim 1 wherein said bis(alkenyl substituted succinimide) is a bis(polybutenyl substituted succinimide) whose polybutenyl succinic anhydride moiety has a molecular weight of about 960 and is the succinimide of the amine derived by reacting tetraethylene pentamine with urea in the ratio of two moles of said

pentamine per mole urea accompanied by the evolution of two moles ammonia for each mole urea, and said exactly neutralized calcium salt is the exactly neutralized calcium salt of N,N'-bis(nonyl hydroxybenzyl) ethylene diamine.

8. A concentrate comprising 10 to 85% by weight of the exactly neutralized calcium salt of a N,N'-bis(alkyl substituted hydroxybenzyl) alkylene diamine and of a bis(alkenyl substituted succinimide) of a (polyazalkyl amino) ureylene that is a condensation product of two moles of polyalkylene polyamine with one mole urea, wherein said alkyl substituted hydroxybenzyl the alkyl substituent is alkyl hydrocarbon of from 5 to 70 carbon atoms, wherein said alkylene diamine the alkylene group is a divalent open chain hydrocarbon of from 2 to 10 carbon atoms, wherein said alkenyl substituent of said succinimide has from 30 to 215 carbon atoms and wherein said polyalkylene polyamine has the formula



wherein "alkylene" is a divalent open chain hydrocarbon of from 2 to 10 carbon atoms, R is selected from the class consisting of hydrogen and lower alkyl hydrocarbon of 1 to 4 carbon atoms and x is a number of from 2 to 10; and said bis-succinimide is present in an amount to provide 0.02 to 0.4 mole per gram atom of said exactly neutralized calcium salt.

9. The concentrate of claim 8 also containing a calcium salt of a C₁₂ to C₃₀ alkyl substituted benzene sulfonic acid in an amount when the concentrate is diluted with a SAE 30 to SAE 50 base oil said calcium sulfonate is present in a concentration of from 0.2 to 1.0% by weight.

10. A lubricant composition comprising a major amount of a mineral lubricating oil of SAE 30 to SAE 50, 0.5 to 10% by weight of a bis(polybutenyl substituted succinimide) of a (polyazalkyl amino) ureylene prepared from one mole urea and two moles of tetraethylene pentamine whose polybutenyl substituted succinic acid moiety has a molecular weight of about 960 and an amount of exactly neutralized calcium salt of N,N'-bis(nonyl hydroxybenzyl) ethylene diamine to provide for each gram atom of calcium from 0.02 to 0.4 mole of said bis-succinimide.

11. The composition of claim 10 wherein there is also present 0.02 to 1.0 weight percent of the calcium salt of a C₁₂ to C₃₀ alkyl substituted benzene sulfonic acid.

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