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2,742,432

MINERAL OIL LUBRICATING COMPOSITIONS

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The present invention relates to improved mineral oil compositions and, particularly, to novel liquid compounded lubricating oil compositions possessing rust-preventive properties. More particularly, the invention relates to such compositions suitable for lubrication of steam turbines and containing components that markedly improve the compositions with respect to preventing rusting of metal components of the turbine.

The compositions embodied herein comprise a mineral oil in major amount, based on the weight of the composition, and a small but rust-inhibiting amount of an oil-soluble combination additive comprising a half amide-ester of an aliphatic dicarboxylic acid and an aliphatic amine. With reference to the mineral oil component of the compositions embodied herein, use is contemplated of mineral oil fractions of lubricating grade, derived from any of a variety of crudes, and which fractions should desirably be improved with respect to rust-preventive characteristics. In particular, however, the invention relates to use, as the mineral oil fraction, of highly refined lubricating oil fractions produced by solvent extracting, acid treating or other methods known to those skilled in the art for removal of more-aromatic components from mineral oil lubricating fractions to provide highly refined lubricating oils of more paraffinic nature than the fraction subjected to the refining treatment. Although it is not intended that the invention be limited thereto, a preferred embodiment relates to composition comprising, as the mineral oil component, a highly refined solvent extracted oil of lubricating grade produced by furfural extraction of a lubricating oil distillate fraction and an illustration of which is an oil such as the turbine oil used in preparation of the compositions set forth hereinafter in further description of the invention.

As set forth hereinbefore, one of the additive components of the compositions embodied herein is an oil-soluble half amide-ester of an aliphatic dicarboxylic acid, i. e. aliphatic dicarboxylic acids in which one carboxyl group has been esterified with an aliphatic alcohol and the other carboxyl group has been converted to an amide grouping in which at least one of the two hydrogen atoms linked to the amide nitrogen atom is substituted by an aliphatic radical. Illustrative of such a component are half amide-esters of acids such as succinic acid, glutaric acid, adipic acid, suberic acid, and the like, as well as alkylated dicarboxylic acids such as alkylated succinic acid and alkylated derivatives of other acids such as aforesaid. As to the esterified portion of the half amide-ester compounds, contemplated herein are such compounds that contain, as an aliphatic residue of an esterifying aliphatic alcohol, an aliphatic group of from about four to about 20 carbon atoms, and, more preferably, from about 8 to 20 carbon atoms. As to the amide por-

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tion of the half amide-ester compounds, contemplated herein are such compounds wherein at least one of the hydrogen atoms linked to the nitrogen atom has been substituted by an aliphatic group, preferably an alkyl group of about four to about twenty carbon atoms. With reference thereto, and as the half amide-esters contemplated are those soluble in mineral oil lubricating fractions in amounts which, though small, are sufficient to provide the desired rust preventive properties when used in combination with an aliphatic amine, the number of carbon atoms present in either or both the ester grouping and substituted amide grouping should be sufficient to impart such solubility characteristics to the half amide-esters. For purposes of illustration and not limitation, such half amide-esters include the lauryl ester of N-hexyl succinamic acid, the octadecyl ester of N-hexyl succinamic acid, the dodecyl ester of N-hexadecyl adipamic acid, the lauryl ester of N,N-dihexyl succinamic or adipamic acid, the octadecyl ester of N-hexyl alkenylated succinamic acid, and others. Half amide-esters as embodied for use herein are disclosed in my copending application, Serial No. 462,360, filed October 14, 1954, and may be prepared, as is also disclosed in said copending application, by subjecting a N-substituted monoamide of a dicarboxylic acid to reaction with an alcohol under esterification reaction conditions to esterify the free carboxyl group of the N-substituted monoamide the said half amide esters for use herein and the detailed method for preparing the same are disclosed in U. S. Patent No. 2,627,487 issued February 3, 1953 to Nathan L. Drake.

With reference to the aliphatic amine component, contemplated for use are oil-soluble primary-, secondary-, and tertiary aliphatic amines including normal aliphatic amines and branched chain aliphatic amines. The oil-soluble aliphatic amines employed may vary over a rather wide range with respect to the number of carbon atoms in their aliphatic radical or radicals as is apparent from the examples set forth hereinafter illustrating practice of this invention with primary alkyl amines of relatively small carbon atom content (e. g. n-hexyl amine), a primary 18 carbon atom branched chain alkyl amine, a secondary alkyl amine (e. g. di-n-butylamine), a primary branched chain amine of relatively small carbon atom content (e. g. t-butylamine), and a tertiary alkyl amine (tributylamine). Thus, and although oil-soluble aliphatic amines preferred for use herein are those containing an alkyl group of from about four to about eighteen carbon atoms, amines having an aliphatic group of more than eighteen carbon atoms (e. g. up to about 24 carbon atoms) are contemplated for use herein.

In order to further describe the invention and the improved rust-preventive characteristics imparted to a mineral oil composition by practice of the invention, the following tabulation sets forth compositions prepared by dissolving, in a turbine lubricating oil (150 SUS at 100° F.) prepared by furfural extraction of a petroleum lubricating oil distillate, the amounts shown (weight per cent) of the half amide-esters and aliphatic amines set forth. The tabulation also sets forth the results obtained by subjecting the compositions to the test, known as ASTM D-665-52T, for determining rust-preventive characteristics. Briefly, the test provides that polished cylindrical steel specimens be partially immersed in a bath maintained at 140° F., and composed of 300 ml. of lubricant and 30 ml. of distilled water, and observing the time required for rusting, if any, to occur on the immersed portion of the steel specimens. Compositions that in-

hibited rusting for twenty-four hours under the test conditions were considered as satisfactorily passing the rusting test.

No.	Composition							ASTM D-665-52T Rusting Test (Distilled Water)
	Half Amide-Ester		Aliphatic Amine					
	Octadecyl ester of N-hexyl succinamic acid	Lauryl ester of N-hexyl succinamic acid	18 carbon atom branched chain primary amine	Tributyl Amine	Di-n-butyl Amine	n-hexyl Amine		
1	0.05							Failed (1½ hrs.).
2	0.03							Failed (1 hr.).
3			0.009					Do.
4	0.05		0.009					Passed (24 hrs.).
5				0.008				Failed (1 hr.).
6	0.05			0.008				Passed (24 hrs.).
7					0.008			Failed in under 24 hrs.
8	0.05				0.008			Passed (24 hrs.).
9						0.008		Failed in under 24 hrs.
10	0.05					0.008		Passed (24 hrs.).
11							0.007	Failed in under 7 hrs.
12	0.05						0.007	Passed (24 hrs.).
13		0.1						Failed (1 hr.).
14		0.05						Do.
15		0.1	0.009					Passed (24 hrs.).

As is apparent from the foregoing tabulation, the oil compositions containing the half amide-esters, or the aliphatic amines, but not a combination thereof, imparted little, if any, rust-preventive characteristics whereas use of the additives in combination provided highly effective protection against rusting. In illustration, composition No. 1 which contained 0.05% by weight of the octadecyl ester of N-hexyl succinamic acid failed to protect against rusting for more than one and a half hours and composition No. 3 containing 0.009% by weight of the primary 18 carbon atom alkyl amine, and devoid of the half amide-ester, failed to protect against rusting for more than one hour. However, composition No. 4, containing the half amide-ester of composition No. 1 and the amide of composition No. 3 in the amounts used in compositions 1 and 3 protected against rusting for at least twenty-four hours, thereby evidencing the substantial more-than-additive protection provided by use of the aforesaid additives in combination.

As is further apparent from the foregoing tabulation, a marked improvement in rust-preventive characteristics is imparted to the mineral oil composition by dissolving therein a small amount of the combination additive. In setting forth compositions containing the half amide-ester and aliphatic amines in the specific amounts shown, such amounts have been used as illustrations of concentrations suitable for practice of the invention. It should be understood, however, that amounts other than those specifically set forth are contemplated as, for example, a combination in amounts of from about 0.02 to about 1.0% by weight of the composition. The relative proportion of the half amide-esters to aliphatic amine in the combination additive may also be varied, depending upon particular requirements with the actual proportion employed being such that, for a particular mineral oil composition, the combination additive comprises a proportional amount of the half amide-esters to aliphatic amine such that a more-than-additive degree of rust preventive characteristics is obtained by use of the combination additive. Although, as aforesaid the relative proportion of the half amide-esters to aliphatic amine may be varied to meet particular requirements, the ingredients are usually employed in amounts of one part of the aliphatic amine to from about one to about twenty parts of the half amide-ester and, preferably, one part of the amine to from about five to fifteen parts of the half amide-ester.

Although the present invention has been described in conjunction with certain preferred embodiments thereof, those skilled in the art will recognize that variations and modifications can be made. Such modifications and var-

iations are to be considered to be within the purview of the specification and scope of the appended claims. Moreover, it should be understood that compositions as

embodied herein may also contain additional ingredients for imparting other desired properties to the compositions. In illustration, the compositions may contain anti-foam agents, e. g. of the silicone type, viscosity index improvers such as the polyisobutylenes, pour point depressants such as the wax alkylated naphthylenes, and others.

I claim:

1. A lubricating oil composition comprising a mineral oil of lubricating grade in major amount based on the weight of the composition and a small amount, sufficient to impart rust-preventive properties to said composition, of an additive comprising (1) an oil-soluble half amide-aliphatic hydrocarbon ester of an aliphatic hydrocarbon dicarboxylic acid and (2) an oil-soluble aliphatic hydrocarbon amine, said composition being characterized by containing said amine and half amide-ester in a weight ratio of one part of amine to from about one to about twenty parts of half amide-ester and said half amide-ester being characterized in that (3) its amide portion contains an aliphatic hydrocarbon substituent of about four to about twenty carbon atoms for at least one of the hydrogen atoms linked to the amide nitrogen atom and (4) its esterified portion contains an aliphatic hydrocarbon group of from about four to about twenty carbon atoms.
2. A lubricating composition, as defined in claim 1, wherein the aliphatic hydrocarbon dicarboxylic acid is a saturated aliphatic hydrocarbon dicarboxylic acid.
3. A lubricating composition, as defined in claim 1, which contains the additive in an amount of from about 0.02 to about 1.0 per cent based upon the weight of the composition.
4. A lubricating composition, as defined in claim 1, wherein the amine is an alkyl amine containing an alkyl group of from about four to about twenty-four carbon atoms.
5. A lubricating composition, as defined in claim 1, wherein the aliphatic hydrocarbon dicarboxylic acid is a saturated aliphatic hydrocarbon dicarboxylic acid, the amine is an alkyl amine containing an alkyl group of from about four to about twenty-four carbon atoms, and the additive is present in the composition in an amount of from about 0.02 to about 1.0 per cent based upon the weight of the composition.
6. A lubricating oil composition comprising a refined mineral oil of turbine lubricating grade in major amount based on the weight of the composition, and from about 0.02 to about 1.0% by weight of an oil-soluble additive consisting essentially of a half amide-ester of an aliphatic dicarboxylic acid and an alkyl amine in a weight ratio

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of one part of amine to from one to about twenty parts of said half amide-ester, said half amide-ester being selected from the group consisting of the octadecyl ester of N-hexyl succinamic acid and the lauryl ester of N-hexyl succinamic acid and said alkyl amine characterized by containing an alkyl group of from about four to about twenty-four carbon atoms.

7. A composition, as defined in claim 6, wherein the amine is a member from the group consisting of t-butyl amine, di-n-butylamine, tributylamine, n-hexyl amine, and

an eighteen carbon atom branched chain alkyl primary amine.

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