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LUBRICANT

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This invention relates to an improved turbine oil. It relates more particularly to a lubricating oil composition consisting principally of a petroleum lubricating oil, the characteristics of the oil being modified by the addition thereto of a relatively small proportion of certain phenol-morpholine-aniline condensation products.

A lubricating oil composition to be used as a turbine oil, and especially in modern marine steam turbines, is subject to very exacting requirements. Not only must it perform the ordinary function of lubricating the turbine over prolonged periods without interruption but usually it must serve as a coolant, to lubricate the gearing mechanism and to operate oil-actuated governors or control mechanisms having very nice tolerances and lubricate other auxiliary equipment.

Many lubricating oil compositions highly satisfactory for the lubrication of other mechanisms have been found wholly unsuitable for use as a turbine oil. This is probably due primarily to the fact that in normal use turbine oils rapidly become contaminated with water. Whatever the cause, it is generally recognized that the performance of a turbine oil is not predictable from conventional tests applicable to other oil lubricants.

Essential characteristics of a satisfactory turbine oil include, in addition to ordinary lubricating requirements, extraordinary resistance to oxidation and emulsion in the presence of water. The avoidance of rusting of the metal parts within the oil system of the turbine and auxiliary apparatus under operating conditions is a further desirable characteristic.

The use of many lubricating oil compositions, otherwise satisfactory as turbine oils, has resulted in the oxidation of the oil and the rusting of metal parts within the oil system with consequent serious interference with the operation of the turbine including the oil-actuated governors and other parts, depending upon close tolerances. The results of such oxidation and rusting not only interfere with the operation of and tend to clog the delicate clearances of the oil system but the products of the rusting appear to catalyze oxidation of the oil with resultant sludge formation which may further aggravate such conditions. The products of such rusting also appear to act as emulsifying agents.

We have found that the previously experienced oxidation of the turbine oil may be substantially inhibited by incorporating in the oil a minor amount of certain morpholinomethyl-anilino-methyl-phenol compounds, more specifically, bis-(morpholinomethyl)-anilinomethyl-phenol and morpholinomethyl-bis-(anilinomethyl)-phenol.

The bis-(morpholinomethyl)-anilinomethyl-phenol used in the compounding of our improved turbine oil can be produced as follows: There are placed in a 1-liter, 3-necked flask, equipped with a thermometer, dropping funnel, and a motor-driven stirrer, 47 grams (0.5 mol.) of phenol, 51.1 grams (0.55 mol.) of aniline and 95.7 grams (1.1 mols.) of morpholine. To this mixture, cooled to 68° F. there is added dropwise over a period of 40 minutes with stirring, 134 grams of formalin (1.65 mols. formaldehyde), the mixture being cooled during this addition so as to maintain the temperature of the mixture between 68° and 78° F. Thereafter, the reaction mixture is stirred for 2 hours at a temperature of 80° F. and then heated on a steam bath for 4¾ hours, using a reflux condenser for returning volatile materials to the flask. Moisture and any unreacted material present in the resulting mixture are then distilled off by heating at an absolute pressure of 0.8 mm. of mercury using an oil bath of 220° F. By this procedure, we have obtained the bis-(morpholinomethyl)-anilinomethyl-phenol as a very viscous, cloudy, white liquid residue analyzing 9.67% nitrogen as compared with the theoretical nitrogen content of 10.6% for pure bis-(morpholinomethyl)-anilinomethyl-phenol. The product thus obtained was equivalent to 108% of the theoretical yield.

The morpholinomethyl-bis-(anilinomethyl)-phenol used in the compounding of our improved turbine oil can be produced as follows: In the apparatus previously described there are placed 47 grams (0.5 mol.) of phenol, 46.1 grams (0.53 mol.) of morpholine and 97.6 grams (1.05 mols.) of aniline. While maintaining the temperature of the mixture between 68° F. and 74° F. and while stirring, there are added over a period of 50 minutes 48 grams (1.6 mols.) of formaldehyde, as 148 grams of a 32% aqueous solution, and 200 c. c. of ethyl alcohol. The reaction mixture will become turbid and is then stirred for 1 hour, the temperature being maintained at 68° F. to 74° F. Thereafter, the mixture is heated by a steam bath for 2¾ hours, using a reflux condenser to return volatile material to the flask. Moisture and any unreacted materials present are then distilled off by heating on the steam bath under an absolute pressure of 2 mm. of mercury. By this procedure we have obtained a 205 gram yield of the morpholinomethyl-bis-

(anilinomethyl)-phenol as a milky, semi-solid residue analyzing 9.5% nitrogen as compared with the theoretical nitrogen content of 10.4% for pure morpholinomethyl-bis-(anilinomethyl)-phenol. This yield of 205 grams is equivalent to 102% of the theoretical yield.

In referring to bis-(morpholinomethyl)-anilinomethyl-phenol and morpholinomethyl-bis-(anilinomethyl)-phenol herein and in the appended claims, we refer, respectively, to the above-described products although, of course, we intend to refer by these terms to the same materials by whatever process they may be made. It will be understood that our invention is not predicated upon the identification of the addends as a matter of terminology.

The lubricating oil constituent of our improved turbine oil may consist of a petroleum lubricating fraction, such as ordinarily specified for turbine oils. It may with advantage be a highly refined lubricating oil, for instance an acid-treated petroleum lubricating oil fraction or one which has been subjected to solvent refining, such as a phenol-treated fraction from East Texas crude. The solvent-refined oils have generally been found more resistant to sludging. For example, phenol-treated East Texas neutrals having the following characteristics have been used with advantage:

	Sample I	Sample II
Gravity—A. I. I.	28.9	26.5
Flash °F.	405	400
Fire °F.	455	455
Viscosity at 100° F.—S. S. U.	192.3	228.1
Viscosity at 210° F.—S. S. U.	45.3	46.9
Viscosity index (Dean & Davis)	87.1	79.2
Pour °F.	+10	+5
Neutralization number	0.025	0.05
Saponification do	0.27	0.27
Carbon residue (Conradson) percent	0.031	0.044
Ash do	0.000	0.002
Sulfur do	0.21	0.34
Steam emulsion number	1	42
Demulsibility	1,620+	600

The "Steam emulsion numbers" appearing in the foregoing tabulation were determined in accordance with the method designated "Standard method of testing for steam emulsion of lubricating oils, A. S. T. M., D157–36."

The "demulsibility" values appearing in the foregoing tabulation were determined in accordance with the method designated "Demulsibility test for lubricating oils" prescribed by "Federal Standard Stock Catalog, section IV, (part 5), Federal Specifications for Lubricants and Liquid Fuels, general specifications (Methods for sampling and testing), VV–L–791a, October 2, 1934, method 320.32."

The unique requirements of a turbine oil have resulted in the formulation of special test methods for determination of the characteristics of the oil with respect to rusting and oxidation. The results of rusting tests and oxidation tests, hereinafter noted, were obtained in accordance with methods prescribed by the American Society of Testing Materials and designated, respectively, "A. S. T. M. specification D–665–42T for turbine oils" and "Proposed method for determining oxidation characteristics of turbine oils, section III, Technical committee C, A. S. T. M. committee D–2, July 2, 1941."

By incorporating a minor proportion of either of the above-identified addends in a suitable lubricating oil constituent, oxidation of the oil under conditions to which turbine oils are subjected is materially inhibited. Depending upon the severity of the oxidation conditions involved, including temperature, access of air to the oil, amount of water present and the amount of salt or the like in the oil, the proportion of the addend used may with advantage be varied from about 0.05% to about 1% by weight of the oil.

For example, the oxidation induction period of the East Texas phenol-treated neutral previously identified herein as sample I, was found by the previously-identified test for determining oxidation characteristics of turbine oils to be 80 hours. By compounding with this oil 0.5% of bis-(morpholinomethyl)-anilinomethyl-phenol, the oxidation induction period of the resultant turbine oil was increased to 1300 hours. Similarly determined, the oxidation induction period of the oil previously identified herein as sample II was 150 hours and, by compounding with this oil 0.2% of bis-(morpholinomethyl)-anilinomethyl-phenol the oxidation induction period was increased to 525 hours. Also, by the addition of 0.2% of morpholinomethyl-bis-(anilinomethyl)-phenol to the sample II base oil, the oxidation induction period was increased to 375 hours and, by the addition of 0.5% of this addend, the oxidation induction period was increased to 725 hours.

In addition to substantially inhibiting oxidation, we have found the above-identified addends to materially reduce rusting of the metal parts within the oil system of steam turbines. For example, the previously-identified oil sample I, when subjected to the previously-identified rusting test for turbine oils, resulted in the rusting of 75% to 100% of the surface of the test specimen. By adding to this oil 0.5% of bis-(morpholinomethyl)-anilinomethyl-phenol and subjecting the oil composition to the identical test, substantially less than 25% of the surface of the test specimen showed indications of rusting. While the morpholinomethyl-bis-(anilinomethyl)-phenol has not generally been found as effective in preventing rusting, as has the bis-(morpholinomethyl)-anilinomethyl-phenol, each has been found to materially reduce the amount of oxidation and rusting.

The ability of the addends of the present invention effectively to inhibit oxidation of turbine oils is unexpected and not readily accounted for. This is particularly evident from the fact that other similar chemical compounds have been found to be either inert or pro-oxidants under conditions encountered by turbine oils. Examples of such compounds found ineffective as anti-oxidants in turbine oils are morpholinomethyl-phenol and morpholinomethyl-p-tert.-amylphenol.

In the compounding of our improved turbine oil, a small amount of one of the above-identified addends is admixed with a suitable petroleum lubricating oil in the conventional manner of compounding such oil compositions. In addition to the lubricating oil constituent and the addend previously described, various other addition agents having the ability favorably to influence the characteristics of the turbine oil may be incorporated in the improved turbine oil of our present invention further to improve the properties thereof in various respects.

A further essential characteristic of turbine oils is that they do not form objectionable emulsions under conditions of use. Consequently, in the compounding of such oils, it is necessary to

avoid the use of addends which might deleteriously affect the emulsibility of the oil. A further advantage of our improved turbine oil is its satisfactory emulsifying characteristics.

For example, an accepted method for determining the emulsifying characteristic of turbine oils is that designated "Emulsion test for lubricating oils" prescribed by "Federal Standard Stock Catalog, section IV (part 5), Federal Specifications for Lubricants and Liquid Fuels, general specifications (methods for sampling and testing), VV-L-791*a*, October 2, 1934, method 320.12," conventionally known as "Navy emulsion test." The base oil, sample II, used in the preceding specific illustration of the invention, satisfactorily complied with the requirements of this test and the emulsion characteristics of the oil were not destroyed by the addition of either of these addends.

A further notable characteristic of our improved turbine oil is its ability to withstand contamination by water without material separation of the addend from the oil or substantial deterioration of the addend itself.

As previously indicated, depending upon conditions of use, the addend may with advantage be used in proportions ranging from about 0.05% to 1% on the weight of the oil. Proportions even in excess of 1% may be used but such larger proportions have not been found necessary. Though proportions less than 0.05% may be used, such smaller proportions are usually not sufficiently effective. Accordingly, proportions ranging from about 0.05% to about 1% are generally recommended.

We claim:

1. An improved turbine oil which comprises a petroleum lubricating oil containing a proportion of a compound of the class consisting of bis-(morpholinomethyl)-anilinomethyl-phenol and morpholinomethyl-bis-(anilinomethyl)-phenol effective to retard oxidation of the oil.

2. An improved turbine oil which comprises a petroleum lubricating oil containing about 0.05% to about 1% of a compound of the class consisting of bis-(morpholinomethyl)-anilinomethyl-phenol and morpholinomethyl-bis-(anilinomethyl)-phenol.

3. An improved turbine oil which comprises a petroleum lubricating oil containing about 0.2% to about 1% of bis-(morpholinomethyl)-anilinomethyl phenol.

4. An improved turbine oil which comprises a petroleum lubricating oil containing about 0.2% to about 1% of morpholinomethyl-bis-(anilinomethyl)-phenol.

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