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STEAM-TURBINE LUBRICATING OILS

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5 Claims. (Cl. 252—50)

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My invention relates to improvements in lubricating oils and more particularly to an improved steam turbine lubricating oil composition comprising a mineral lubricating oil containing a small percentage of a pyrazoline compound as an oxidation inhibitor.

Steam turbine oils are used under conditions which are very severe as far as oxidation is concerned because the oil is constantly in contact with water and with steam, as well as being subjected to relatively high temperatures.

The primary object of my invention is therefore to provide an improved lubricating oil composition which will be relatively stable under the operating conditions encountered in steam turbines.

A further object of my invention is to provide an improved method of lubricating steam turbines.

In my work with lubricating oils, I have discovered that a mineral lubricating oil of a grade suitable for lubricating steam turbines containing a small percentage of an oil soluble organic pyrazoline will be inhibited against oxidation for long periods of time. These compounds all contain the pyrazoline ring which is a five membered ring containing two nitrogen atoms linked together and to a chain of three carbon atoms.

In accordance with a preferred form of my invention, I employ a pyrazoline compound containing substituents on two or more positions of the pyrazoline ring, and particularly select such substituents so that the resulting compound antioxidant will be soluble in the mineral lubricating oil stock and will not be removed to any appreciable extent by being brought in contact with water and steam.

The features of my invention may be illustrated in connection with certain specific examples showing the effectiveness of certain pyrazolines in turbine oils.

Example I

In this example two petroleum oil stocks were tested in accordance with the standard oxidation testing procedure for steam turbine oils (Proc. Am. Soc. Test. Mat. 1943, p. 275 et seq.). Oil A was a solvent refined neutral oil having a Saybolt universal viscosity of 150 at 100° F. Oil B was an acid refined neutral oil having a Saybolt universal viscosity of 200 at 100° F.

In the table below the induction periods for these oils are given when uninhibited against oxidation and when inhibited with various percentages of the pyrazoline compound, 1-phenyl-3,5,5-trimethyl pyrazoline.

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	House-Induction Period	
	Oil A	Oil B
Uninhibited oil	60	180
Oil+0.25% Pyrazoline compound	600	1,440
Oil+0.50% Pyrazoline compound	1,000	2,400
Oil+1.0% Pyrazoline compound	2,000	3,500

It will be noted that the particular pyrazoline compound used in these tests was much more effective with the acid refined neutral oil than it was with the solvent refined oil. However, the effectiveness is very considerable when it is noted that by using only 1% of the pyrazoline the induction period was extended from 60 hours for the uninhibited oil to 2,000 hours.

Example II

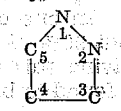
In this instance the same oils as tested in Example I had incorporated in them an appropriate percentage of the pyrazoline compound, 1-phenyl-3-isobutenyl-5,5-dimethyl pyrazoline. The results were as follows:

	Hours-Induction Period	
	Oil A	Oil B
Uninhibited oil	60	180
Oil+0.50% Pyrazoline compound	----	2,200
Oil+1.0% Pyrazoline compound	470	2,180

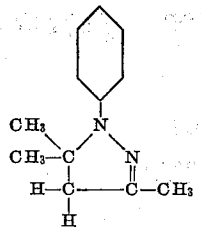
In these tests the lower percentage of inhibitor was not used with oil A.

The use of the pyrazoline compound inhibitors are found to have very little effect in changing the other properties of the oils tested, for example, the acid number, color, gravity and pour point remained substantially unchanged by the addition of 1% of the pyrazoline. The flash, fire and viscosities were lowered slightly while the steam emulsion number went up somewhat.

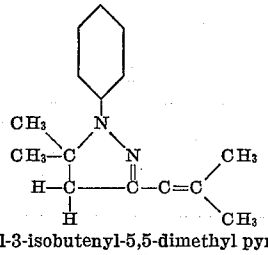
My discovery of the usefulness of the pyrazoline compounds as oxidation inhibitors for lubricating oils and particularly for steam turbine oils is based upon my conception that pyrazoline compounds containing the pyrazole ring would be effective as antioxidizing agents. The structure of this ring is well known and may be indicated as follows:



The positions of the various elements in the ring are indicated by numbers, and accordingly, the pyrazolines referred to in the foregoing examples have the following structures in which it will be seen that the carbon atoms in the pyrazole ring are saturated:



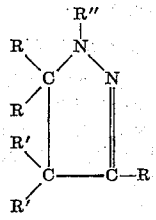
1-phenyl-3,5,5-trimethyl pyrazoline



1-phenyl-3-isobutenyl-5,5-dimethyl pyrazoline

My conception that the pyrazole ring was the effective part of the pyrazolines was supported by tests which showed that the various hydrocarbon substituents upon the ring were ineffective in themselves as oxidation inhibitors.

In accordance with my invention it is to be understood that the pyrazolines may have various hydrocarbon substituents or radicals on the pyrazole ring, and therefore the compound may have the general formula



in which R may be an aliphatic or an aromatic group, R′ a hydrogen or an aliphatic or an aromatic group, and R″ is preferably an aromatic group but may be an aromatic or an aliphatic group. R′ is preferably hydrogen.

Various methods are known for the preparation of pyrazoline compounds, but I have found the following general method effective and to give high yields. For example, 1-phenyl-3,5,5-trimethyl pyrazoline is prepared by mixing 108 parts by weight of phenylhydrazine with 120 parts by weight of mesityl oxide in a suitable still equipped with a refluxing arrangement. These proportions provide an excess of the oxide so as to use up substantially all of the phenylhydrazine, the surplus being recovered for use in succeeding operations. A 50–50 mixture of ethyl alcohol and acetic acid is added to the still, as for example about 25 parts by weight, and the mixture is refluxed for about 2 hours which is usually sufficient to complete the action. Thereafter, the mixture is vacuum distilled to remove the alcohol, water, acetic acid and the excess of mesityl oxide. At 12 millimeters of mercury pressure the portion distilling between 150° C. and 170° C. is recovered as the pyrazoline product. In one operation this corresponded with about 90% of the theoretic yield and the product having this wide boiling range of about 20° C. was found to be as effective as an inhibitor as were the narrower fractions of the product.

It is to be understood that other methods may be employed in making the pyrazoline compounds, and it will furthermore be understood that the compounds do not have the same effectiveness in a given oil. As indicated in the foregoing examples, a particular compound may have a substantially different induction period with different oils. The pyrazoline antioxidants have a special utility and are specially used with steam turbine lubricating oils, but it is to be understood that they may be used in other mineral or petroleum lubricating oil stocks, and in conjunction with an antirust agent.

The oil stocks A and B described above and containing suitable proportions of a pyrazoline inhibitor are steam turbine lubricating oil stocks and effectively serve that purpose in lubricating steam turbines for long periods of time without being oxidized to any appreciable extent. In fact, in most cases the actual service given by these oils materially exceeds that indicated by the tests, since, as is well understood, the conditions of such tests are more severe than the conditions to which the oil is subjected in the steam turbine.

While the pyrazoline compounds referred to are readily soluble in the steam turbine stocks and other mineral oils, and may be mixed directly therewith, they may be first dissolved in a base oil stock to provided a concentrated solution containing, for example, from 25 to 50% of the oxidation inhibitor, and this concentrated solution then used as the addition material which is mixed with the oil stock to be protected against oxidation.

What I claim as new is:

1. A steam turbine oil composition comprising a refined mineral steam turbine lubricating oil having incorporated therein an antioxidant selected from the group consisting of 1-phenyl-3,5,5-trimethyl pyrazoline and 1-phenyl-3-isobutenyl-5,5-dimethyl pyrazoline.

2. A steam turbine oil composition comprising a refined mineral steam turbine oil having incorporated therein an antioxidant in sufficient proportion to effectively inhibit the oxidation of the oil for a substantial period of time, said antioxidant comprising 1-phenyl-3,5,5-trimethyl pyrazoline.

3. A steam turbine oil composition as defined by claim 2 in which the composition contains from 0.50% to 1.0% of the oxidation inhibitor.

4. A steam turbine oil composition comprising a refined mineral steam turbine oil having incorporated therein an antioxidant in sufficient proportion to effectively inhibit the oxidation of the oil for a substantial period of time, said antioxidant comprising 1-phenyl-3-isobutenyl-5,5-dimethyl pyrazoline.

5. A steam turbine oil composition as defined by claim 4 in which the composition contains from 0.50 per cent to 1.0 per cent of the oxidation inhibitor.

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