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INSULATING OIL COMPOSITIONS

Charles E. Trantman, East McKeesport, Pa., assignor to Gulf Research & Development Company, Pittsburgh, Pa., a corporation of Delaware

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This invention or discovery relates to insulating oil compositions; and it comprises such a composition, particularly a transformer oil, highly resistant to deterioration by oxidation, including a petroleum oil containing a small amount of an alkyl ester of an amino-benzoic acid, advantageously methyl anthranilate and other short chain alkyl esters of ortho-amino-benzoic acid; all as more fully hereinafter set forth and as claimed.

In many types of electrical transformers the core and windings are immersed in a suitable petroleum oil, for serving as a dielectric and for carrying heat away from the core and windings. The requirements of a transformer oil include good dielectric properties, low viscosity to facilitate heat transfer for convection, low pour point to avoid solidification of the oil by cold, high flash and fire points to minimize danger of fire and explosion and low volatility to reduce evaporation losses. In addition the oil must be resistant to oxidation, and must be of such character as to resist emulsification with water.

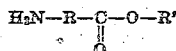
Transformer oils ordinarily are distillate oils refined in such manner as to secure the specified characteristics to as great an extent as possible. Achievement of satisfactory oxidation resistance is difficult, for while oxidational changes are slow, transformer oils are ordinarily expected to remain in service over long periods. Oxidational changes result in the formation of insoluble matter or sludge. The sludge tends to render the oil more miscible with water, with production of emulsions in the presence of condensed water and a consequent degradation of the dielectric properties of the oil body. The sludge also deposits on the windings and core with reduction of the efficiency of heat transfer. Oxidation of transformer oils tends to be promoted by contact with metals, especially copper, and with the usual varnishes and insulating materials.

Various materials have been proposed for incorporation into transformer oils with the object of inhibiting oxidation, and some of these have come into use. The usual anti-oxidants may be divided in two principal classes: (1) those which are associated with a lengthening of the induction period, that is the time which elapses before appearance of sludge in the oil subject to oxidation, and (2) those which reduce the amount of sludge formed per day after it once begins to form. In evaluating oils containing anti-oxidants, the most useful criterion is a value which takes into account both these characteristics, viz., the time required, under the test conditions, to

form a definite amount of sludge, say 0.1 per cent by weight of the oil.

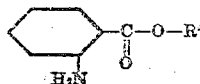
I have discovered a new class of antioxidant compounds which bring about both a lengthening of the induction period and a marked lessening of the rate of sludge formation thereafter, when incorporated in transformer oils in even minimal amounts. These antioxidants have certain other advantages. They are quite soluble in oil, more so than many other antioxidants, but are highly insoluble in water, thereby minimizing the tendency to form aqueous emulsions. Also they are non-acid and do not deleteriously affect the acid number of the transformer oil when incorporated therein.

These antioxidant compounds are alkyl esters of an amino-benzoic acid which can be represented by the structural formula:



wherein R represents a phenylene group and R' represents an alkyl group. The esters containing a short chain alkyl group, particularly those of ortho-amino-benzoic acid, are advantageous. They are very effective in preventing or retarding sludging and other oxidational deterioration of the oil.

In the preferred embodiments of this invention, the antioxidants employed are alkyl esters of ortho-amino-benzoic acid which can be represented by the structural formula:

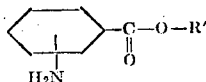


wherein R' is a short chain alkyl group; these esters being also known as alkyl anthranilates. For instance, methyl anthranilate is an advantageous antioxidant in transformer oils and other insulating oil compositions. It is readily soluble in transformer oils and when incorporated in even minimal amounts effectively inhibits sludge formation.

Likewise, other alkyl anthranilates are useful and advantageous. That is, the methyl group (CH₃) can be replaced by other short chain alkyl groups such as ethyl, propyl, amyl or butyl groups; ethyl anthranilate, propyl anthranilate and the like being also advantageous and giving similar results. As a class, these alkyl esters of ortho-amino-benzoic acid are readily soluble in the oil and are good anti-oxidants for the present purposes.

Further, the alkyl esters of other amino-benzoic acids, wherein the amino group is in the para or meta position, are also useful. Instance, the methyl ester of para-amino-benzoic acid is an effective antioxidant. However, such esters are somewhat less soluble in the oil than the alkyl anthranilates. Nevertheless, they are sufficiently soluble for the present purposes and improve the resistance of the oil to oxidational deterioration.

In the broad practice of this invention, insulating oils such as transformer oils and the like can be advantageously improved by incorporating alkyl esters which can be generically represented by the structural formula:



wherein R' represents a short chain alkyl group.

These compounds or antioxidants when added to transformer oils or other electrical insulating oils in small amounts, advantageously amounts corresponding to about 0.05 to 0.5 per cent by weight of the oil, effectively stabilize such oils against oxidational deterioration. For instance, the beneficial effect may be obtained with amounts as small as 0.01 per cent. In such amounts they are readily soluble in such oils.

In the following table comparative oxidation test results are set forth for oils containing these new antioxidants, for an untreated oil and for certain other antioxidants not a part of the invention. The test data reported in the table were obtained in accordance with the sludge formation test for mineral transformer oils described by F. M. Clark and E. A. Snyder in their paper entitled "Testing for Sludge Formation in Mineral Transformer Oil" presented at the Thirtieth Annual Meeting for the American Society for Testing Materials. The test procedure described in the aforesaid paper was developed in collaboration with the laboratories of a considerable number of firms engaged in oil refining and in the manufacture of electrical equipment.

In this test, samples of oil are maintained in tubes containing copper wire at a constant temperature of approximately 120° C. The tubes are open at the top to an enclosed air space and dry air is passed at a controlled rate of 1½ to 2 cubic feet per hour through the enclosed space above the sample tubes. Into each tube is introduced 22 grams of oil or oil composition to be tested, a number of tubes being employed. At prescribed intervals the tubes are successively removed and the amount of sludge determined. The results of these tests are reported in terms of the induction period, the percent of sludge formed per day and the total number of days taken to form 0.1 per cent of sludge.

The oil employed in all of the tests set forth hereinbelow was a typical petroleum transformer oil, commonly used for the purpose indicated. The addition agents employed were as follows:

Methyl anthranilate, designated as "MA"
Ethyl anthranilate, designated as "EA"
Methyl-p-amino benzoate, designated as "MPAB"
Anthranilic acid, designated as "A"
2:6-di-tertiary-butyl-4-methyl-phenol, designated as "OP"

The results of tests on the base oil alone and the same oil containing the above-mentioned

addition agents are given in the following table:

Test No.	Transformer oil composition	Induction period, days	Rate of sludge formation, after induction period percent per day	Total time to form 0.1% sludge, days
1	Oil (no inhibitor)	0.5	0.0100	10
2	Oil+0.05% MA	2.0	0.0047	23
3	Oil+0.1% MA	2.0	0.0034	31
4	Oil+0.3% MA	2.0	0.0015	70
5	Oil+0.5% MA	2.0	0.0015	70
6	Oil+0.3% EA	2.0	0.0027	40
7	Oil+0.1% MPAB	0.0	0.0075	12
8	Oil+0.1% A	0.0	0.0041	22
9	Oil+0.3% OP	12.5	0.0205	18
10	Oil+0.05% MA and 0.3% OP	10.0	0.0047	32
11	Oil+0.3% MA and 0.3% OP	3.0	0.0035	30

In the table, tests 2 to 7, inclusive, 10 and 11 relate to improved insulating oil compositions contemplated in this invention; the oil compositions tested being improved transformer oils containing a short chain alkyl ester of amino-benzoic acid. For purposes of comparison tests on the transformer oil alone (1) and on the oil containing anthranilic acid (8) and 2,6-di-tertiary-butyl-4-methyl phenol (9) have been included in the table.

As indicated in tests 2 to 5, inclusive, the benefits obtained with methyl anthranilate increase with proportions thereof up to around 0.5 per cent. In most cases the further increase does not further reduce the oxidation sensitivity of the oil. As methyl anthranilate gives good results, it is ordinarily employed in the practice of the present invention.

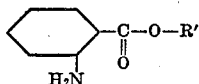
However, as shown ante, other short chain alkyl anthranilates are likewise effective and may be also used to improve transformer oils. For instance, as shown in test 6, ethyl anthranilate produces similar improvements. The use of alkyl esters of other amino benzoic acids is illustrated in test 7 wherein the antioxidant is the methyl ester of para-amino-benzoic acid and like improved results are obtained. Further, these alkyl esters of amino-benzoic acids may be used in conjunction with other improvement agents; they being compatible and miscible with such agents and oil compositions containing the same, as well as soluble in oil alone. This is illustrated in tests 10 and 11 wherein methyl anthranilate may be used in conjunction with an alkylated phenol having antioxidant properties, specifically 2:6-di-tertiary-butyl-4-methyl phenol. As there shown, improvements are obtained which are not obtainable with either of these agents. This is evident from the test results given ante.

While this invention has been described herein with particular reference to transformer oil compositions, these short chain alkyl esters of amino-benzoic acid are equally effective antioxidants and improvement agents in other forms of electrical oils, and insulating oils generally containing stabilizing amounts of these compounds are included within the scope of this invention.

What I claim is:

1. In the operation of electrical transformers containing a petroleum oil as the dielectric and cooling medium, the improvement which comprises insulating and cooling the windings and core of a transformer with a petroleum oil containing dissolved therein a small amount of an

alkyl anthranilate having the following formula



wherein R' represents a short-chained alkyl group selected from the class consisting of methyl and ethyl groups, the amount of said short-chained alkyl anthranilate being sufficient to render the petroleum oil resistant to sludging and other oxidational deterioration.

2. In the operation of electrical transformers containing a petroleum oil as the dielectric and cooling medium, the improvement which comprises insulating and cooling the windings and core of a transformer with a petroleum oil con-

taining 0.01 to 0.5 per cent by weight of methyl anthranilate dissolved therein, the amount of methyl anthranilate being sufficient to render the petroleum oil resistant to sludging and other oxidational deterioration.

3. In the operation of electrical transformers containing a petroleum oil as the dielectric and cooling medium, the improvement which comprises insulating and cooling the windings and core of a transformer with a petroleum oil containing 0.01 to 0.5 per cent by weight of ethyl anthranilate dissolved therein, the amount of ethyl anthranilate being sufficient to render the petroleum oil resistant to sludging and other oxidational deterioration.

CHARLES E. TRAUTMAN.