Designation: D 1934 – 95 (Reapproved 2000)

Standard Test Method for Oxidative Aging of Electrical Insulating Petroleum Oils by Open-Beaker Method [1]

This standard is issued under the fixed designation D 1934; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes two procedures for subjecting electrical insulating oils to oxidative aging:

1.1.1 *Procedure A*, without a metal catalyst, and

1.1.2 *Procedure B*, with a metal catalyst.

1.2 This test method is applicable to oils used as impregnating or pressure media in electrical power transmission cables if less than 10 % of the oil evaporates during the aging procedures. It applies and is generally useful primarily in the evaluation and quality control of unused oils, either inhibited or uninhibited.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration [2]

D 923 Test Method for Sampling Electrical Insulating Liquids [3]

D 924 Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids [3]

D 1169 Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids [3]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens [4]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods [4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *metal catalyst*—any metal (for example, copper) that either increases the rate of oxidation of the oil or reacts with the oxidation products to increase oil dielectric loss.

3.1.2 *oxidative aging*—exposure of oil to oxygen under certain specified conditions.

4. Summary of Test Method

4.1 A 300 mL volume of oil, contained in 400 mL beaker is aged for 96 h in a circulating-air oven controlled at 115°C, either with or without the presence of catalyst.

5. Significance and Use

5.1 Open-beaker oxidative aging methods have been used for many years in laboratories of oil companies, electrical equipment manufacturers, and electric utility companies interested in the stability of electrical insulating oils under oxidative conditions. They are particularly useful as a check on the continuity of production and shipment of insulating oils. They are also useful as process and product checks for applicable type oils.

5.2 Specification limits for oils subjected to open-beaker oxidative aging by this method are established by agreement between individual producers and consumers of applicable type oils. These properties of the oil involved in specification limits for aging stability may be measured after the oxidative aging (and sometimes before aging) by appropriate test methods such as Test Method D 924, Test Method D 1169, and Test Method D 664.

6. Apparatus

6.1 *Oven*, electrically heated, thermostatically controlled, capable of maintaining a constant temperature of 115 ± 1°C (239 ± 2°F). Use an oven with a testing chamber large enough to test the anticipated number of test specimens at one time. A uniformity of temperature within ± 1 % of the differential between oven and ambient temperatures is required. (See Note 1.) Circulate air in the chamber with a low velocity fan during the aging period. The volume and condition of the circulated air is not considered to be critical. It is recommended that the oven provide several air changes per hour, and that vapors and fumes be removed if present.

NOTE 1—Refer to Specification E 145 for the measurement of the temperature uniformity of the oven.

6.1.1 *Procedure A*—For test specimens aged in the absence

[1] This test method is under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gasesand is the direct responsibility of Subcommittee D27.06on Chemical Test.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 10.03.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

of a metal catalyst the choice of a suitable oven design is not critical. Either fixed- or rotating-shelf stage ovens of satisfactory thermal quality may be used, although a rotating-shelf oven is preferred. If a fixed-shelf oven is used, it is recommended that test specimen positions within the oven be changed periodically (for example, at daily intervals) to minimize the effects of any temperature differentials that may exist.

6.1.2 *Procedure B*—When a metal catalyst, such as copper, is used, the rate of oxidation usually is increased, and the procedure becomes sensitive to movement of the oil past the metal surface. An aging oven equipped with a slowly rotating shelf has been adopted for uniformity when a metal catalyst is used. [5] Other oven designs having satisfactory thermal quality and a rotating shelf may be used.

6.2 *Beaker*, borosilicate glass, low-form, of 400-mL capacity. The approximate dimensions of a suitable beaker are 100 mm in depth and 70 mm inside diameter. Clean the beakers used in the aging test, and thoroughly dry before use. One recommended cleaning procedure is as follows: Remove residual oil from the beaker by rinsing in mineral spirits or equivalent. Rinse thoroughly with an acid cleaning solution consisting of 3 parts nitric acid (HNO_3) to 1 part sulfuric acid (H_2SO_4). Remove traces of cleaning solution by carefully rinsing with distilled water followed by rinsing in acetone and air drying. [6]

7. Reagents and Materials

7.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. [7]

7.2 *Hydrochloric Acid*, 10 % volume solution from concentrated hydrochloric acid.

7.3 *Nitric Acid*—Concentrated nitric acid (HNO_3).

7.4 *Sulfuric Acid*—Concentrated sulfuric acid (H_2SO_4).

7.5 *Metal Catalyst* with 15 cm^2 of clean surface available for exposure to the oil for use in Procedure B. If the metal catalyst is copper wire, it is convenient to wind an appropriate length into a loose hank which is then cleaned to remove oil, oxide, and the other extraneous matter. The metal catalyst may also be used in the form of strips, but the strips require special attention to maintain the desired amount of exposed surface. One good method of cleaning copper hanks is to immerse the hank for 30 s in a 10 % solution of hydrochloric acid (HCl), after which the hank is rinsed three times in distilled water then in acetone and air dried. The cleaned hank should be handled with clean tongs.

8. Procedure A—Aging Without a Metal Catalyst

8.1 Obtain the oil sample in accordance with Methods D 923.

8.2 Adjust the oven temperature to 115 ± 1°C.

8.3 Pour without preheating 300 mL of the test specimen to be tested into a clean dry 400-mL beaker. Oil depth in the beaker will be approximately 75 mm. Measure the mass of the oil before test.

8.4 Place the beaker containing the test specimen in the preheated oven. To minimize temperature fluctuation it is desirable to place all test specimens in the oven at the same time.

8.5 Start the oven shelf into rotation if the oven is so equipped.

8.6 Remove the test specimen from the oven at the end of the 96-h aging period and measure the mass of the oil.

8.7 Determine the condition of the aged test specimen by means of appropriate test methods. The condition of an unaged test specimen usually should also be determined at the same time for comparison.

9. Procedure B—Aging with a Metal Catalyst

9.1 Proceed exactly as in Procedure A (Section 8) except before placing the beaker in the oven add the metal catalyst to the oil specimen in the beaker, with the specified amount of surface exposed to the oil.

9.2 Measure the mass of the oil plus catalyst before and after the test.

10. Report

10.1 Report the following information:

10.1.1 Designation of this test method (D 1934),

10.1.2 Whether or not a metal catalyst was used (Procedure A or B) and the nature and form of the metal catalyst, if used,

10.1.3 Test specimen identification,

10.1.4 Values of the property or properties measured on the aged (and unaged) test specimens, and

10.1.5 Designations of the test methods used to determine the condition of the oil test specimens.

10.1.6 The measured change in mass percent of the oil to validate the test (less than 10 % evaporation).

11. Precision and Bias [8]

11.1 *Precision*:

11.1.1 The precision of the open-beaker oxidative aging defined by this test method has not yet been adequately defined in accordance with Practice E 177. However, precision estimates for both single-laboratory repeatability and multilaboratory reproducibility of changes in oil dissipation factor (power factor) due to open-beaker oxidative aging are available from a 1958–59 round robin with ten participating laboratories.

11.2 In the case of four naphthenic insulating oils with different viscosities (55, 100, 760, and 2200 SUS at 100°F, and 38, 39, 60 and 105 SUS, respectively, at 210°F) with and without 2.8 cm^2 of copper wire catalyst, random errors were proportional to the ratio, *Q*:

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[5] A suitable type oven is described in "Life Test for Transformer Oils," Appendix to Report of Committee D-9 on Electrical Insulating Materials, *Proceedings*, ASTM, Vol 27, Part I, 1927 pp. 541–549.

[6] Ammonium persulfate is also a suitable cleaning reagent. Available as Nochromix from Interex Corp., 3 Strathmore Rd., Natick, MA 01760.

[7] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

[8] Supporting data are available from ASTM Headquarters. Request RR:D27-1007.

$$Q = \frac{\text{dissipation factor after aging}}{\text{dissipation factor before aging}}$$

The data were normalized by a logarithmic transformation.

11.3 *Repeatability*—The standard deviation of the quantity log$Q$ has been estimated to be 0.07 (coefficient of variation of $Q = 18$ %) for duplicate aging tests run at the same time in a given oven in the same laboratory. The standard deviation of log$Q$ has been estimated to be 0 (coefficient of variation of $Q = 60$ %) for duplicate aging tests run at different times in the same laboratory.

11.4 *Reproducibility*—The standard deviation of the quantity log$Q$ has been found to be 0.37 (coefficient of variation of $Q = 135$ %) for duplicate aging tests run in different laboratories.

11.5 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method for measuring the true value of the property involved, no statement on bias is being made.

12. Keywords

12.1 aging; electrical insulating oil; open beaker test; oxidative; petroleum