Designation: ~~D1934 – 95 (Reapproved 2012)~~ D1934 – 20

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

This standard is issued under the fixed designation D1934; 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 covers two procedures for subjecting electrical insulating ~~oils~~liquids 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~~ insulating liquids used as impregnating or pressure media in electrical power transmission cables if less than 10 % of the ~~oil~~ insulating liquid evaporates during the aging procedures. It applies and is generally useful primarily in the evaluation and quality control of unused ~~oils,~~ insulating liquids, either inhibited or uninhibited.

1.3 This test method is applicable to study the long-term behavior of an insulating liquid being considered for free breathing transformers. An unsealed vessel aging procedure, in presence of air or oxygen, allows greatly increased oxidation rate of the liquid. This procedure is rapid and provides a controlled thermal stress assessment.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.5 An open beaked test shall only be carried out on liquids with flash points at or above 130°C or 15°C above the oven temperature. *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* See 7.5 for a specific warning statement.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
D923 Practices for Sampling Electrical Insulating Liquids

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D924 Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids
D971 Test Method for Interfacial Tension of Insulating Liquids Against Water by the Ring Method
D974 Test Method for Acid and Base Number by Color-Indicator Titration
D1169 Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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~~liquid or reacts with the oxidation products to increase ~~oil~~ insulating liquid dielectric loss.

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

4. Summary of Test Method

4.1 A 300 mL volume of ~~oil,~~ insulating liquid, 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~~ insulating liquid companies, electrical equipment manufacturers, and electric utility companies interested in the stability of electrical insulating ~~oils~~liquids under oxidative conditions. They are particularly useful as a check on the continuity of production and shipment of insulating ~~oils.~~liquids. They are also useful as process and product checks for applicable type ~~oils.~~insulating liquids.

5.2 Specification limits for ~~oils~~ insulating liquids subjected to open-beaker oxidative aging by this method are established by agreement between individual producers and consumers of applicable type ~~oils.~~ insulating liquids. These properties of the ~~oil~~ insulating liquid 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~~Methods D924, D971~~Test Method~~ , D1169, and ~~Test~~D974 ~~Method~~or D664. Other test methods such as D445 can be used when deemed appropriate.

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.~~ critical enough to be recorded. 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 E145 for the measurement of the temperature uniformity of the oven.

6.1.1 *Procedure A*—For test specimens aged in the absence 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.~~inter-sample differentials.

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~~ insulating liquid past the metal surface. An aging oven equipped with a slowly rotating shelf has been adopted for uniformity when a metal catalyst is used. [3] Other oven designs having satisfactory thermal quality and a rotating shelf may be used.

[3] A suitable type oven is described in "Life Test for Transformer Oils," Appendix to Report of Committee D09 on Electrical and Electronic Insulating Materials, *Proceedings*, ASTM, Vol 27, Part I, 1927 pp. 541–549.

6.2 *Beaker,* borosilicate glass, low-form, of 400-mL capacity. A watch-glass cover over the beaker is required, which prevents particles from dropping into the liquid sample. A watch-glass top on a beaker still allows exchange of air as the means for oxidation of the liquid with or without a copper catalyst. 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~~ insulating liquid 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~~with acetone and air drying. (Ammonium persulfate is also a suitable cleaning reagent.)

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.[4]

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~~ insulating liquid 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,~~ insulating liquid, 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.~~ dried immediately before use. The cleaned hank should be handled with clean tongs. (**Warning**—Procedures listed herein involve the use of concentrated acid solutions. Acids and their vapors can cause severe burns. Handle concentrated acids with extreme care. This procedure must be used by persons knowledgeable in the safe handling and disposal of this material).

8. Procedure A—Aging Without a Metal Catalyst

8.1 Obtain the ~~oil~~ insulating liquid sample in accordance with Practices D923.

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~~ Insulating liquid depth in the beaker will be approximately 75 mm. Measure the mass of the ~~oil before test.~~insulating liquid and beaker before test. Alternately, a 400-mL beaker can be tared, so the amount of liquid can be calculated both before and after the 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.~~insulating liquid and beaker in the case the beaker was not tared.

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.