



Designation: D 2440 – 99

Standard Test Method for Oxidation Stability of Mineral Insulating Oil ¹

This standard is issued under the fixed designation D 2440; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method determines the resistance of mineral transformer oils to oxidation under prescribed accelerated aging conditions. Oxidation stability is measured by the propensity of oils to form sludge and acid products during oxidation. This test method is applicable to new oils, both uninhibited and inhibited, but is not well defined for used or reclaimed oils.

NOTE 1—A shorter duration oxidation test for evaluation of inhibited oils is available in Test Method D 2112.

NOTE 2—For those interested in the measurement of volatile acidity, reference is made to the work of IEC Subcommittee 10A. ²

1.2 The values stated in metric units are to be regarded as the standard.

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:

- B 1 Specification for Hard-Drawn Copper Wire³
- D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration⁴
- D 974 Test Method for Acid and Base Number by ColorIndicator Titration⁴
- D 2112 Test Method for Oxidation Stability of Inhibited Mineral Insulating Oil by Rotating Bomb⁵
- D 2272 Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel⁴
- D 3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus⁵

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

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² ASTM Research Report No. RR: D-27-1001, available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

³ Annual Book of ASTM Standards, Vol 02.03.

⁴ Annual Book of ASTM Standards, Vol 05.01.

⁵ Annual Book of ASTM Standards, Vol 10.03.

2.2 IEC Publication:

CEI/IEC 61125: 1992 Unused Hydrocarbon-Based Insulating Liquids—Test Methods for Evaluating the Oxidation Stability⁶

3. Summary of Test Method

3.1 A test specimen of mineral transformer oil is oxidized at a bath temperature of 110°C, in the presence of a copper catalyst coil, by bubbling oxygen through duplicate test specimens for 72 and 164 h, respectively. The oil is evaluated at the end of each aging period by measuring the amount of sludge and acid formed. The test specimen is diluted with *n*-heptane and the solution filtered to remove the sludge. The sludge is dried and weighed. The sludge-free solution is titrated at room temperature with standard alcoholic base to the end point indicated by the color change (green-brown) of the added *p*-naphthol-benzene solution.

4. Significance and Use

4.1 The oxidation stability test of mineral transformer oils is a method for assessing the amount of sludge and acid products formed in a transformer oil when the oil is tested under prescribed conditions. Good oxidation stability is necessary in order to maximize the service life of the oil by minimizing the formation of sludge and acid. Oils that meet the requirements specified for this test in Specification D 3487 tend to minimize electrical conduction, ensure acceptable heat transfer, and preserve system life. There is no proven correlation between performance in this test and performance in service, since the test does not model the whole insulation system (oil, paper, enamel, wire). However, the test can be used as a control test for evaluating oxidation inhibitors and to check the consistency of oxidation stability of production oils.

5. Apparatus

5.1 *Aging (Oxidation) Bath*—An oil bath, wax bath, or aluminum block heater (see IEC Standard 61125) of a suitable type capable of controlling the temperature at 110 ± 0.5°C with a temperature gradient of less than 1°C in the body of the liquid. Use any nontoxic liquid having low volatility at 110°C

⁶ International Electrotechnical Commission. Available from American National Standards Institute, 1430 Broadway, New York, NY 10018.

and containing no volatile additives and having a flash point above the test temperature. Mineral oils, waxes and silicone oils have been used satisfactorily. Circulation of the oil or wax heating medium by means of a pump or stirrer is recommended. See also 5.4, relating to the depth of fluid in aging bath and position of oil receptacles.

5.2 Drying Tower—A drying tower 25 to 30 cm in height, fitted with a ground-glass stopper and side tubes for conditioning of the oxygen supply.

5.3 Oil Receptacle and Head—An oil receptacle consisting of a heat-resistant glass test tube 25 mm in outside diameter, 1.25 mm in wall thickness, 210 mm in overall length including a standard taper 24/40 outer joint, with a Dreschschel-type head consisting of a standard taper 24/40 inner joint with side outlet tube 5.0 mm in outside diameter and an oxygen delivery tube 5.0 mm in outside diameter and 3 mm, minimum, in inside diameter which extends to within 2.5 ± 0.5 mm of the bottom of the oil receptacle and has its end ground at an angle of 30° to the axis of the tube. The design is shown in Fig. 1.

5.4 Position the oil receptacle in the aging bath in accordance with the drawing shown in Fig. 2.

NOTE 3—The oil receptacle and position in the aging bath are essentially the same as specified in IEC Standard 61125, "Test Method for Oxidation Stability of Inhibited Mineral Insulating Oils." Glassware dimensions have been altered slightly to conform to sizes readily available in the United States.

5.5 Filtering Crucibles—A glass filter crucible having a maximum diameter of the pores between 5 and 15 μm as determined in accordance with the method described in the Appendix. Cellulose ester type membrane filters of 5- μm porosity have been used successfully also.

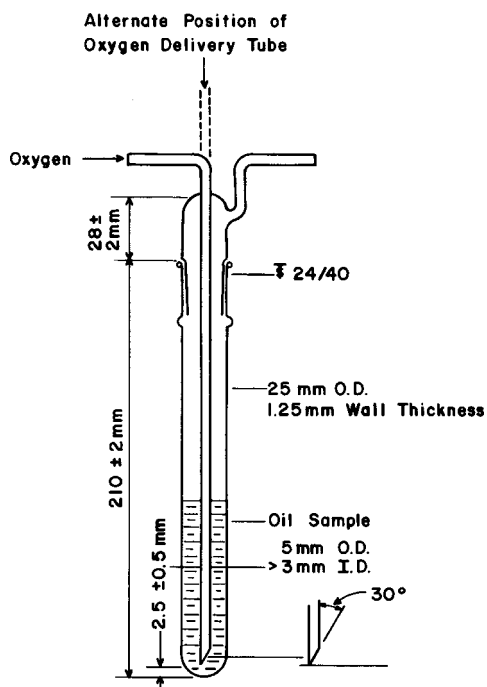


FIG. 1 Oil Receptacle and Head

6. Reagents and Materials

6.1 Purity of Reagents—Use reagent grade chemicals in all tests. Unless otherwise indicated, all reagents are to conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁷

6.2 Chloroform, cp, or **acetone**, cp.

6.3 n-Heptane,⁸ for dilution of the oxidized oil, precipitation, and washing of the sludge shall conform to the following requirements:

Relative Density at 20°C	0.6836 to 0.6839
Refractive index at 20°C	1.3876 to 1.3879
Solidification temperature, min, °C	-90.72
Distillation	50 % shall distill between 98.38 and 98.48°C. Temperature rise between 20 and 80 % recovery shall be 0.20°C maximum

6.4 p-Naphtholbenzein Indicator Solution—The specifications for *p*-naphtholbenzein are prescribed in Test Method D 974.⁹ Prepare a solution containing 10 g *p*-naphtholbenzein per litre of titration solvent (see 6.7).

6.5 Oxygen—Obtained from liquid oxygen, minimum purity 99.4 %.

6.6 Potassium Hydroxide Solution, Standard Alcoholic (0.1 N)—Prepare alcoholic potassium hydroxide solution and standardize as described in Test Method D 974. Commercially prepared Alcoholic Potassium Hydroxide Solution is available.

6.7 Titration Solvent—Mix 3 parts by volume of toluene with 2 parts by volume of isopropyl alcohol.

6.8 Silicon Carbide Abrasive Cloth, 100-grit with cloth backing.

6.9 Wire Catalyst—AWG No. 18 (0.0403-in. (1.01-mm) diameter) 99.9 % purity conforming to Specification B 1. Soft-drawn copper wire of equivalent grade may also be used.

6.10 Hydrochloric Acid, 10 Volume %.

7. Hazards

7.1 Consult Material Safety Data Sheets for all materials used in this test method.

8. Preparation of Oxygen Supply

8.1 Oxidize the oil by contact with oxygen of 99.4 % minimum purity conditioned as follows:

8.1.1 Use metal or glass tubing to deliver the oxygen to the oxygen delivery tube. Eliminate rubber connections if possible. If used, the rubber should be at room temperature. Limit the length of rubber tubing exposed to the oxygen flow to 13 mm. Do not use rubber connections in contact with the liquid of the heating bath or under conditions where the temperature of the rubber will be above room temperature. Where it is necessary

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 A suitable material is ASTM *n*-Heptane Knock Test Reference Fuel, marketed by the Phillips Petroleum Co., Bartlesville, OK.

⁹ Solid *p*-naphtholbenzein conforming to the specifications indicated is available from Distillation Products Industries, Eastman Organic Chemicals Dept., Rochester 3, NY.