Designation: D2440 – 13 (Reapproved 2021)

Standard Test Method for Oxidation Stability of Mineral Insulating Oil[1]

This standard is issued under the fixed designation D2440; 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 U.S. 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 D2112.

NOTE 2—For those interested in the measurement of volatile acidity, reference is made to IEC Method 61125. [2]

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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:*[3]

B1 Specification for Hard-Drawn Copper Wire

D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D974 Test Method for Acid and Base Number by Color-Indicator Titration

D2112 Test Method for Oxidation Stability of Inhibited Mineral Insulating Oil by Pressure Vessel

D2272 Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel

D3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus

2.2 *IEC Publication:*

IEC 61125: 1992 Unused Hydrocarbon–Based Insulating Liquids—Test Methods for Evaluating the Oxidation Stability[4]

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-benzein 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 D3487 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

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

Current edition approved Nov. 1, 2021. Published November 2021. Originally approved in 1965 as D2440 – 65 T. Last previous edition approved in 2013 as D2440 – 13. DOI: 10.1520/D2440-13R21.

[2] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D27-1001. Contact ASTM Customer Service at service@astm.org.

[3] 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.

[4] International Electrotechnical Commission. Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

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 Method 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. When initially setting up the bath, a measurement of the temperature gradient with the tubes immersed is to be conducted to ensure that no temperature gradient exists as the tubes may obstruct a uniform circulation. Use any nontoxic liquid having low volatility at 110 °C 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 required. 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 at least 25 cm in length 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 Dreschsel-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 Method 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. Both types of tubes can be used.

5.5 *Glass or Porcelain Crucibles*—50 mL size.

5.6 *Filtering Membranes*—Cellulose ester type membrane filters of 5-µm porosity, 47 mm.

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. [5]

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

[5] "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."

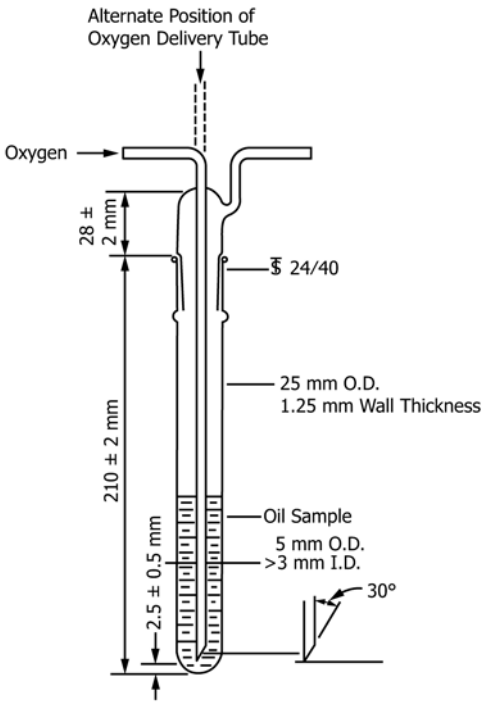

FIG. 1 Oil Receptacle and Head

6.3 *n-Heptane,* 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 D974. Prepare a solution containing 10 g *p*-naphtholbenzein per litre of titration solvent (see 6.7).

6.5 *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 D974. 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 B1. 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.

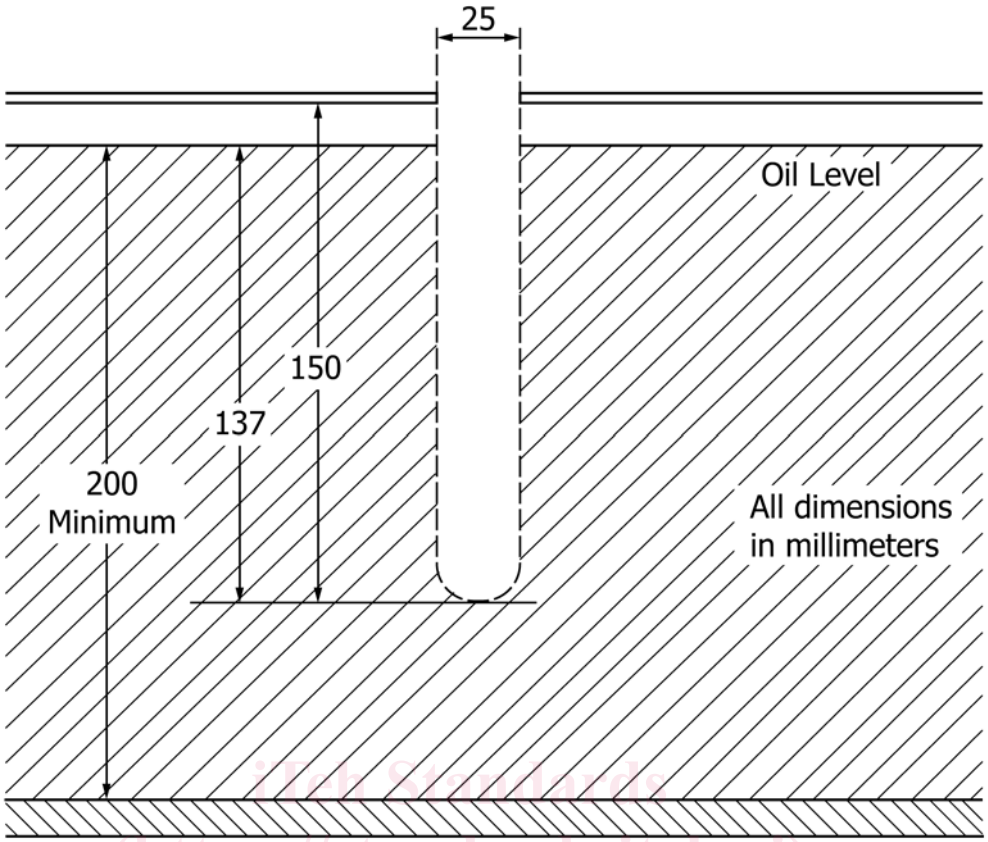

FIG. 2 Position of Oil Receptacle in Aging Bath

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 tubing connections when possible. If used, the tubing should be at room temperature. Limit the length of tubing exposed to the oxygen flow to 40 mm. Do not use tubing connections in contact with the liquid of the heating bath or under conditions where the temperature of the tubing will be above room temperature. Where it is necessary to use tubing connections, only sulfur-free tubing is permissible. FOA/USP Nalgene tubing and certain types of PVC tubing have been found satisfactory in this application.

8.1.2 Dry the oxygen by forcing it through a solid desiccant of high moisture-absorbing capacity.[6] Arrange the desiccant in the drying tower to a depth of 205 to 254 mm. Change the desiccant when the indicator begins to change colors from moisture absorption. If an indicator is not used, change the desiccant at least weekly.

8.1.3 After passing oxygen through the drying tower, admit it directly to the receptacle containing the oil to be tested. Do not preheat the oxygen.

8.1.4 Determine the rate of oxygen supply with an electronic flow meter, calibrated rotameter, or soap bubble buret, and adjust the flow rate to deliver the conditioned oxygen at a rate of 1.0 ± 0.1 L/h to each tube of oil being tested.

9. Preparation of Oil Receptacle

9.1 Wash each oil receptacle thoroughly, first with acetone and then with soap and water, and rinse in acid solution. The following reagents have been found suitable: chromic acid, aqua regia, and ammonium persulfate. Wash each receptacle free of acid, using tap water, and finally rinse with distilled water. Dry in an oven at 105 to 110 °C for at least 3 h; cool to room temperature before use.

10. Preparation of Copper Catalyst

10.1 Immediately before use, polish the copper wire with silicon carbide abrasive cloth and wipe free of abrasive with a clean dry cloth. Clean the copper wire with acetone or chloroform before it is wound to remove the debris from polishing.

10.2 Wind a 300-mm length of the polished wire into a helical coil approximately 16 mm in outside diameter and 50 mm in height. Clean the coil thoroughly with chloroform or acetone, air dry, and insert immediately into the oil receptacle. Handle the clean copper coil only with clean tongs and clean gloved hands to avoid contamination.

10.3 Commercially available, prepackaged, preformed coils that meet the requirements described in this test method may be used as an alternative method of catalyst preparation. Clean the

[6] Anhydrous magnesium perchlorate (Anhydrone or Dehydrite) is a suitable desiccant for this purpose.