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Standard Test Method for Corrosive Sulfur in Electrical Insulating Liquids[1]

This standard is issued under the fixed designation D1275; 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 adopted for use by government agencies to replace Method 5328-2 of Federal Test Method Standard No. 791b. This standard was adopted as an ASTM-IP Standard.

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

INTRODUCTION

Prior to 2006, this test method existed as a singular method in which the main aging parameters of the test specimen were 19 h at 140 °C. In 2006, it was determined that those parameters were inadequate (not sensitive enough to detect all levels of corrosive sulfur) and Method B was instituted in which the main aging parameters were 48 h at 150 °C. The old parameters were kept as Method A to avoid any confusion. The 2015 edition of this test method for the copper corrosion procedure reverts back to a singular method in which the main aging parameters are the same as the previous Method B (2006-2014). Results from the current test method cannot be compared or correlated to the method prior to 2006 and only to Method B from 2006 to 2014.

1. Scope

1.1 This test method describes the detection of corrosive sulfur compounds (both inorganic and organic) in electrical insulating liquids.

1.2 New and in-service insulating liquids may contain elemental sulfur or sulfur compounds, or both, that cause corrosion under certain conditions of use. This test method is designed to detect the presence of, or the propensity to form, free (elemental) sulfur and corrosive sulfur compounds by subjecting copper or silver to contact with an insulating liquid under prescribed conditions.

1.3 The values stated in SI units are to be regarded as the standard. Inch-pound units are included for informational purposes.

1.4 *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.*

1.5 *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]

D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D923 Practices for Sampling Electrical Insulating Liquids

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

2.2 *Other Document:*

ANSI B74.10 Grading of Abrasive Microgrits[3]

DIN 51 353 Testing of Insulating Oils; detection of corrosive sulfur, silver strip test[4]

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[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.

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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.

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

[4] Available from Deutsches Institut für Normung e.V.(DIN), Am DIN-Platz, Burggrafenstrasse 6, 10787 Berlin, Germany, http://www.din.de or http://www.bleuth.de

3. Significance and Use

3.1 In most of their uses, insulating liquids are continually in contact with metals that are subject to corrosion. The presence of elemental sulfur or corrosive sulfur compounds will result in deterioration of these metals and cause conductive or high resistive films to form. The extent of deterioration is dependent upon the quantity and type of corrosive agent and time and temperature factors. Detection of these undesirable impurities, even though not in terms of quantitative values, is a means for recognizing the hazard involved.

3.2 Two methods are provided, one for copper corrosion and one for silver corrosion. Copper is slightly less sensitive to sulfur corrosion than silver but the results are easier to interpret and less prone to error. The silver corrosion procedure is provided especially for those users who have applications where the insulating liquid is in contact with a silver surface.

4. Apparatus

4.1 *Bath*—A hot-air oven or liquid bath provided with suitable means of heating to, and controlling at 150 °C ± 2 °C. A circulating hot-air oven is preferred.

4.2 *Bottles*[5]—Heavy-walled, 250 mL, bottles of chemically resistant glass constructed with necks to receive a polytetrafluoroethylene (PTFE) threaded plug equipped with a fluoroelastomer O-ring. Bottles of such capacity and design are required in order to allow sufficient space for expansion of the insulating liquid and to eliminate intrusion from atmospheric gases. Flat bottomed bottles are preferred.

4.3 *Copper Foil, 99.9+ % pure,* 0.127 mm to 0.254 mm (0.005 in. to 0.010 in.) in thickness.

4.4 *Silver Foil,* 99.99+ % pure, 0.5 mm thick.

4.5 *Polishing Material,* consisting of 240-grit silicon carbide paper or cloth (refer to Specification E11), and also 230-mesh silicon carbide grains and pharmaceutical absorbent cotton. Aluminum oxide or silicon carbide sanding pads can be substituted but the surface finish of the foil piece must closely mirror that of using silicon paper or cloth.

Note 1—It should be noted that 240-grit silicon carbide paper and 230-mesh silicon carbide grains have particle sizes of about the same size (63 µm). In the United States, abrasive papers are classified in accordance with ANSI B74.10. Abrasive powders are classified by ASTM mesh size.

5. Reagents

5.1 *Acetone,* ACS reagent grade.

5.2 *Hydrochloric Acid*—ACS reagent grade, 36.5 % to 38.0 % assay.

5.3 *Nitrogen Gas*—Commercial cylinders of nitrogen gas are satisfactory for this purpose.

5.4 *Suitable Solvent*—Technical grade acetone, heptane, hexane and pentane.

5.5 *Water,* distilled.

6. Summary of Test Methods

6.1 *Copper Corrosion*—220 mL of insulating liquid is aged in a sealed heavy-walled bottle for 48 h at 150 °C in the presence of a copper strip.

6.2 *Silver Corrosion*—220 mL of insulating liquid is aged in a sealed heavy-walled bottle for 48 h at 150 °C in the presence of a silver strip.

7. Preparation of Apparatus

7.1 Chemically clean bottles and PTFE screw plugs with solvents to remove insulating liquid residue, then wash the bottles with a suitable solvent such as heptane or hexane, or use a phosphate-type cleaning powder or liquid. Rinse with tap water, then with distilled water, and dry in an oven. Replace the fluoro-elastomer O-ring before each test to avoid cross contamination or a compromised seal.

7.2 Polish a larger piece of copper or silver foil from which, after the final polishing, several strips of the proper size may be cut. Remove blemishes from both surfaces of the copper or silver foils with the 240-grit silicon carbide paper or sanding pads. Cut a strip(s) of copper or silver foil 6 mm by 25 mm (1⁄4 in. by 1 in.). Strips may be stored in sulfur-free acetone at this point for future use. Do the final polishing of the strip by removing it from the acetone, holding it in the fingers protected with ashless filter paper or nitrile gloves, and rubbing with 230-mesh silicon carbide grains picked up from a glass plate with a pad of absorbent cotton moistened with a drop of acetone. Wipe the strip with fresh pads of cotton and subsequently handle only with stainless steel forceps (do not touch with fingers). Rub in the direction of the long axis of the strip. Clean all metal dust and abrasive from the strip, using successive clean cotton pads until a fresh pad remains unsoiled. Bend the clean strip in a V-shape at approximately a 60° angle and wash successively in acetone, distilled water, and acetone. Dry in an oven for 3 min to 5 min at 80 °C to 100 °C and immediately immerse the copper or silver strip in the prepared test specimen of insulating liquid (Note 2). Do not use compressed air or an inert gas to dry the metal strip.

Note 2—This method of cleaning has been adapted from Test Method D130.

8. Copper Corrosion Procedure

8.1 Retrieve a sample for testing in accordance with Practices D923. Use the insulating liquid to be tested as received. Do not filter the insulating liquid.

8.2 Promptly place the prepared copper strip in a clean 250 mL bottle to which has been added 220 mL of the insulating liquid to be tested. Place the bent copper strip standing on its long edge so that no flat surface lies along the glass bottom of the vessel. Bubble nitrogen through the insulating liquid in the bottle by means of a 1.5 mm inner diameter (approximately 1⁄16 in. inner diameter) glass or stainless steel tube connected to the reduction or needle valve

[5] The sole source of supply of the Bottles and PTF screw plugs known to the committee at this time is Prism Research Glass, P.O. Box 14187, Research Triangle Park, NC 27709, part number DOB-B-250. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend. Laboratories already using bottles from Ace Glass can continue.