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

This standard is issued under the fixed designation D 1275; 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.

1. Scope

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

1.2 Mineral insulating oils may contain substances that cause corrosion under certain conditions of use. This test method is designed to detect the presence of free sulfur and corrosive sulfur compounds by subjecting copper to contact with oil under prescribed conditions.

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

1.4 *This standard does not purport to address 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 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test[2]

E 11 Specification for Wire-Cloth and Sieves for Testing Purposes[3]

2.2 *Other Document:*

ANSI B74.10 Grading of Abrasive Microgrits[4]

3. Significance and Use

3.1 In most of their uses, insulating oils are continually in contact with metals that are subject to corrosion. The presence of corrosive sulfur compounds will result in deterioration of these metals. 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.

4. Apparatus

4.1 *Bath*—A hot-air oven or oil bath provided with suitable means of heating to, and controlling at, 140 ± 2°C. A circulating hot-air oven is preferred.

4.2 *Containers*—Narrow-mouth, 250-mL, ground-glass stoppered flasks, of chemically resistant glass, capable of holding 270 to 280 mL when filled completely to the stopper. Flasks of such capacity are required in order to allow sufficient space for expansion of the oil.

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

4.4 *Polishing Material*, consisting of 240-grit silicon carbide paper or cloth, and also 230-mesh silicon carbide grains and pharmaceutical absorbent cotton.

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*, cp.

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

6. Preparation of Apparatus

6.1 Chemically clean flasks with solvents to remove oil, then wash the flasks with phosphate-type cleaning powder or liquid. Rinse with tap water, then with distilled water, and dry in an oven.

6.2 Cut a strip of copper 6 by 25 mm (¼ by 1 in.) (Note 2) and remove blemishes from surfaces with the 240-grit silicon carbide paper. Strips may be stored in sulfur-free acetone at this

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

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

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

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, 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 the 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 only a few minutes and immediately immerse in the prepared test specimen of oil (Note 3).

NOTE 2—It has been found convenient to polish a larger piece of copper from which, after the final polishing, several strips of the proper size may be cut.

NOTE 3—This method of cleaning has been adapted from Test Method D 130.

7. Procedure

7.1 Use the oil to be tested as received. Do not filter the oil through paper. Promptly place the prepared copper strip in a clean 250-mL flask to which has been added 250 mL of the oil 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. Lubricate the ground-glass stopper with a small amount of the test specimen. Bubble nitrogen through the oil in the flask by means of a glass tube connected to the reduction or needle valve of the cylinder (rubber connections must be sulfur-free) for 1 min, and quickly put the stopper loosely in place.

7.2 Place the stoppered flask (immersed to the neck in the event an oil bath is employed) in the oven at 140°C. When the oil in the flask has reached approximately 140°C, tighten the stopper more firmly (Note 4). Remove the flask after heating for 19 h ± 10 min at 140 ± 2°C. Carefully take the copper strip from the flask and wash with acetone or other suitable solvent to remove all of the oil and let air dry. Do not use pressurized air to dry copper strip.

NOTE 4—The corrosive effects of unstable sulfur compounds present in the oil are reduced when aged under strong oxidizing conditions such as when oxygen is present. It is imperative to keep the flask well sealed after nitrogen bubbling.

7.3 To inspect, hold the test strip in such a manner that light reflected from it at an angle of approximately 45° will be observed.

8. Interpretation of Results

8.1 Classify the oil as corrosive or noncorrosive in accordance with Table 1. Classification of corrosive or noncorrosive may be aided through the use of ASTM Copper Strip Corrosion Standards as referenced in Test Method D 130.

9. Report

9.1 Report the following information:

9.1.1 Sample identification, and

9.1.2 Test specimen as being corrosive or noncorrosive.

10. Precision and Bias

10.1 No statement is made about either the precision or bias of this test method since the result merely states whether there is conformance to the criteria for success specified in the procedure and is not quantitative.

11. Keywords

11.1 corrosion; corrosive sulfur; insulating; mineral; oils; petroleum; sulfur

TABLE 1 Copper Strip Classifications

Classification	Description
Noncorrosive	Orange, red, lavender, multicolored with lavender blue or silver, or both, overlaid on claret red, silvery, brassy or gold, magenta overcast on brassy strip, multicolored with red and green showing (peacock) but no gray
Corrosive	Transparent black, dark gray or dark brown, graphite or lusterless black, glossy or jet black, any degree of flaking