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Standard Test Method for COPPER IN ELECTRICAL INSULATING OILS BY PHOTOMETRIC ANALYSIS¹

This standard is issued under the fixed designation D 2608; 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 the determination of copper in insulating oil in concentrations of 0.1 ppm or greater.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Method

2.1 A 1+1 mixture of oil and hexane is extracted with a 2+8 combination of hydrochloric and glacial acetic acids. The copper is concentrated in the acid phase, which is then washed free of organic colored material with chloroform. Copper is determined colorimetrically as the neocuproine complex at approximately 450 nm. Since interfering organic substances in the visible range have been removed a simple colorimeter with a blue filter is adequate.

3. Significance and Use

3.1 Electrical insulating oil may contain small amounts of dissolved metals derived either directly from the base oil or from contact with metals during refining or service.

4. Apparatus

4.1 *Spectrophotometer* or filter photometer with a blue filter capable of producing light of 450-nm wavelength.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chem-

icals shall be used 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.² Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—All water shall be sufficiently deionized to give no color reaction with neocuproine reagent.

5.3 *Acetic Acid, Glacial.*

5.4 *Acetic-Hydrochloric Acid Mixture*—Add slowly while stirring 200 mL of concentrated HCl (sp gr 1.19) to 800 mL of glacial acetic acid.

5.5 *Chloroform.*

5.6 *Copper, Standard Solution (1 mL = 0.010 mg Cu)*—Dissolve 0.2000 g of pure copper metal (99.9 % copper) in 10 mL of concentrated nitric acid (HNO₃, sp gr 1.42). Add 5 mL of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) and heat to white fumes. Allow to cool and transfer quantitatively to a 1-L volumetric flask containing 200 to 300 mL of distilled water and dilute to 1000 mL. Dilute a 50 mL aliquot of this solution to 1 L; 1 mL of the resulting solution will contain 0.010 mg of copper.

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 "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."