Designation: E2575 – 08

Standard Test Method for Determination of Oxygen in Copper and Copper Alloys[1]

This standard is issued under the fixed designation E2575; 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 oxygen in copper and copper alloys in concentrations from 0.0005 to 0.04 %.

1.2 *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:*[2]

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques
E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System

2.2 *Material Certificates:*

BCR No. 18 Oxygen in Phosphorus-Deoxidized Copper[3]
BCR No. 22 Oxygen in ETP Copper[3]
BCR No. 58 Oxygen in Continuous Cast Copper Rod[3]
NCS NS 41004 Oxygen in Pure Copper[4]
NIST SRM 885 Refined Copper[5]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

4. Summary of Test Method

4.1 This test method is intended for use with commercially available inert gas fusion determinators.

4.2 The test specimen is fused in a graphite crucible under a flowing inert gas stream (argon (Ar), helium (He), or nitrogen (N_2)) at a temperature sufficient to release oxygen. Oxygen from the specimen combines with carbon from the crucible to form carbon monoxide (CO). The detector output is converted to the mass fraction of oxygen in the specimen using a previously establish calibration. Depending on the instrument design, CO is oxidized to carbon dioxide (CO_2) or left as CO and swept by the inert gas stream into an infrared detector.

4.3 In a typical instrument based on infrared detection the evolved gases are swept into an infrared cell through which infrared energy is transmitted. The CO in the gas stream absorbs some of the transmitted infrared energy and the decrease in the energy reaching the detector is processed and displayed directly as percent oxygen. Some instruments oxidize the CO to CO_2, which is subsequently measured by an infrared cell designed to measure CO_2.

5. Significance and Use

5.1 This test method is primarily intended as a referee test for compliance with compositional specifications. It is assumed that all who use this test method will be trained analysts, capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

6. Interferences

6.1 The elements usually present in copper and its alloys do not interfere.

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[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.05 on Cu, Pb, Zn, Cd, Sn, Be, their Alloys, and Related Metals.

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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 European Commission, Joint Research Centre, Institute for Reference Materials and Measurements, Retieseweg 111, B-2440 Geel, Belgium, http://irmm.jrc.ec.europa.eu/.

[4] Available from Central Iron and Steel Research Institute (CISRI), No. 76 Xueyan Nanlu, Haidian District, Beijing, China 100081, http://www.cisri.com/.

[5] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

7. Apparatus

7.1 *Instrument*—Fusion and measurement apparatus consisting of an electrode furnace, provision for scrubbing impurities from analytical gas stream, infrared measurement system, and auxiliary gas purification systems. (See Note 1.)

NOTE 1—The apparatus and analysis systems have been previously described in Test Method E1019. Several models of commercial oxygen determinators are available and presently in use by industry. Each has its own unique design characteristics and operational requirements. Consult the instrument manufacturer's manual for operational details.

7.2 *Graphite Crucibles*—The crucibles must be made of high-purity graphite as recommended by the instrument manufacturer and be of the dimensions recommended by the instrument manufacturer.

7.3 *Tweezers*—Approximately 6 in. (152 mm), used during the sample preparation process.

7.4 *Balance*—Measurement apparatus with capacity up to 10 g capable of weighing ±0.1 mg accurately or as specified by the instrument manufacturer.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals 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.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

8.2 *Graphite Powder/Carbon Black*—High-purity graphite powder or carbon black specified by the instrument manufacturer.

8.3 *Inert Gas*—Use the purity and type (Ar, He, or N_2) specified by the instrument manufacturer.

8.4 *Magnesium Perchlorate* ($Mg(ClO_4)_2$)*—Anhydrous*—Used as a moisture trap. Use purity specified by the instrument manufacturer.

8.5 *Rare Earth/Copper Oxide*—Reagent used in some instruments to oxidize CO to CO_2. Use purity specified by the instrument manufacturer.

8.6 *Sodium Hydroxide Impregnated Clay*—Used to absorb CO_2 in the inert gas stream. Use purity specified by the instrument manufacturer.

8.7 *Methanol*—Used in sample preparation, see Section 10.

8.8 *Copper Pickle Solution*—Prepare a fresh solution of equal parts of concentrated nitric acid (HNO_3), concentrated acetic acid (CH_3OOH), and concentrated phosphoric acid (H_3PO_4). Used in sample preparation, see Section 10.

9. Hazards

9.1 For hazards to be observed in the use of certain reagents in this test method refer to Practice E50.

9.2 Use care when handling hot crucibles and operating furnaces to avoid injury by either burn or electrical shock.

10. Sampling and Sample Preparation

10.1 Use only solid samples to minimize the potential for errors due to surface oxidation. Samples must be of the proper size to permit free introduction into the sample-loading device of the instrument. Sample weight range should be 0.5 to 2.0 g. Refer to instrument manufacturer's recommendations.

10.2 Cut the sample to an appropriate size using a silicon carbide, water-cooled cut-off wheel or by other means that will avoid overheating. Avoid oxide cutting or abrading materials.

10.3 Etch sample with concentrated hydrochloric acid (HCl) at 20°C for 3 minutes.

10.4 Etch sample in a mixture of equal parts of concentrated HNO_3, concentrated CH_3COOH, and concentrated H_3PO_4 at 70°C for one minute.

10.5 Rinse sample in three (3) successive distilled water rinses.

10.6 Rinse sample in three (3) successive methanol rinses.

10.7 Dry in a stream of hot air (hair drier).

10.8 Do not touch sample with fingers during and following the final stages of cleaning. Store the prepared sample in a desiccator. If samples are not analyzed within four hours of preparation, repeat 10.3-10.7 prior to analysis. (See Note 2.)

NOTE 2—Careful adherence to the sample preparation procedure described above is critical to obtaining accurate and precise results. The use of small and irregular shaped samples requires a diligent effort to ensure that all surface contamination has been removed.

11. Preparation of Apparatus

11.1 Assemble the apparatus as recommended by the manufacturer. Make the required power, gas, and water connections. Turn on the instrument and allow sufficient warm up time to stabilize the system.

11.2 Change the chemical reagents and filters as required. Test the furnace and the analyzer to insure the absence of leaks. Make a minimum of two determinations using a sample as directed in Section 13 to condition the instrument before attempting to calibrate the system or to determine the value of the blank.

11.3 Calibrate balance as recommended by the manufacturer using internal or external weights traceable to the SI (see IEEE /ASTM SI 10).

12. Calibration

12.1 *Calibration Reference Materials* (see Note 3)—Use only copper or copper alloy reference materials. Select four reference materials containing approximately 0.0005, 0.01, 0.03, and 0.04 % oxygen and designate them as Calibrants A, B, C, and D respectively.

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[6] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC, www.chemistry.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, http://www.usp.org.