



Designation: E841 – 12

Standard Test Method for Determination of Copper in Iron Ores and Related Materials by Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation E841; 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 iron ores, concentrates, agglomerates, and related materials in the concentration range from 0.003 % to 1 %.

NOTE 1—As used in this test method (except as related to the term “relative standard deviation”, “%” refers to a mass fraction (wt / wt %) (g/100g).

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

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

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals³

E276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials

E663 Practice for Flame Atomic Absorption Analysis³

E877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods

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 The sample is dissolved in HCl with the addition of a small amount of HNO₃ and HF acids. After evaporation to fumes with HClO₄, the solution is diluted with water and filtered. A portion of the solution is examined by atomic absorption spectrometry using standards containing approximately the same amount of iron as the test sample.

5. Significance and Use

5.1 In the making of iron and steel during the reduction of iron ores, copper forms alloy with iron and steel hence the necessity of determining the copper content for metallurgical consideration.

³ Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

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 E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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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.

5.2 This test method is intended to be used for compliance with compositional specifications for copper content. It is assumed that all who use these procedures 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 and that proper waste disposal procedures will be followed. Appropriate quality control practices must be followed, such as those described in Guide E882.

6. Interferences

6.1 None of the elements normally found in iron ores interfere with this test method.

7. Apparatus

7.1 *Atomic Absorption Spectrometer*, meeting the following criteria:

7.1.1 *Minimum Sensitivity*—The absorbance of the highest calibration solution (see 8.6) must be at least 0.3.

7.1.2 *Curve—Linearity*—The difference between the readings of the two highest calibration solutions must be more than 1.4 times the difference between the readings for the zero solution and the lowest calibration solution (see 8.6).

7.1.3 *Minimum Stability*—The relative standard deviation of a number of measurements of the highest calibration solution and of the zero calibration solution must be less than 1.5 % and 0.5 % respectively, relative to the measurement of the highest calibration solution.

NOTE 2—A strip chart recorder or digital readout device, or both is advisable to measure the criteria in 7.1 and for all subsequent measurements.

NOTE 3—A background corrector equipped with a hydrogen or a deuterium hollow cathode lamp is advisable for the compositional range from 0.003 % to 0.010 % Cu.

NOTE 4—Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. Solutions were aspirated into an air-acetylene flame of a premix burner.

Hollow cathode lamp, mA	3
Wavelength, nm	324.7
Air flow-rate, L/min	10
Acetylene flow-rate, L/min	2.5

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 conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available⁴. 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. 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.

to Type I or II of Specification D1193. Type III or IV may be used if they effect no measurable change in the blank or sample.

8.3 *Copper Standard Solution A (1 mL = 1.0 mg Cu)*—Dissolve 1.000 g of copper metal (minimum 99.5 %) in 30 mL of HNO₃ (1 + 1) and dilute to 1 L.

8.4 *Copper Standard Solution B (1 mL = 0.10 mg Cu)*—Transfer 100 mL of Standard Solution A to a 1-L volumetric flask, dilute to volume, and mix.

8.5 *Copper Standard Solution C (1 mL = 0.01 mg Cu)*—Transfer 100 mL of Standard Solution B to a 1-L volumetric flask, dilute to volume, and mix.

8.6 *Copper Standard Calibration Solution*—Depending on the expected copper content of the sample, prepare a calibration solution using copper Standard Solution B or C. For the range of copper content from 0.01 % to 0.2 %, use copper Standard Solution B and for the range of copper content from 0.003 % to 0.02 %, use copper Standard Solution C.

8.6.1 Transfer (1.0, 3.0, 5.0, 7.0, and 10.0)-mL portions of copper Standard Solution B or C to 100-mL volumetric flasks. Add 20.0 mL of background solution, dilute to volume, and mix.

8.7 *Iron Background Solution*—Dissolve 15 g of high-purity iron metal in 150 mL HCl (1 + 2) and oxidize by the dropwise addition of HNO₃. Add 250 mL of HClO₄ and evaporate to fumes. Allow to fume for 10 min, cool, and dilute to 1 L.

8.8 *Reference Solution*—Transfer 20 mL of the background solution to a 100-mL volumetric flask, dilute to volume, and mix.

9. Hazards

9.1 For precautions to be observed in this method, refer to Practice E50.

10. Sampling and Sample Preparation

10.1 Collect and prepare the test unit in accordance with Practice E877.

10.2 The analytical sample shall be pulverized so that at least 95 % passes a No. 100 (150- μ m) sieve in accordance with Test Methods E276.

NOTE 5—To facilitate decomposition, some ores, such as specular hematite, require grinding to pass a No. 200 (75- μ m) sieve.

11. Procedure

11.1 Transfer approximately 0.5 g of the sample to a small weighing bottle previously dried at about 150 °C. Dry bottle and contents for 1 h at 105 °C to 110 °C. Cap the bottle and cool to room temperature in a desiccator. Momentarily release the cap to equalize the pressure and weigh the capped bottle and sample to the nearest 0.1 mg. Repeat drying and weighing until there is no further loss of mass. Transfer the sample to a 250-mL beaker and reweigh the capped bottle to the nearest 0.1 mg. The difference between the two masses is the mass of the sample taken for analysis.

11.2 Carry a reagent blank through all steps of the procedure starting with 11.3.

11.3 *Decomposition of Sample*—Moisten the sample with a few millilitres of water, add 25 mL of HCl, cover, and digest