Designation: E841 – 12a

Standard Test Method for
Determination of Copper in Iron Ores and Related Materials by Flame Atomic Absorption Spectrometry[1]

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E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

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E663 Practice for Flame Atomic Absorption Analysis (Withdrawn 1997)[3]

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 HNO_3 and HF acids. After evaporation to fumes with $HClO_4$, 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.

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.

---

[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.

Current edition approved June 1, 2012. Published July 2012. Originally approved in 1981. Last previous edition approved in 2012 as E841 – 12. DOI: 10.1520/E0841-12A.

[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] The last approved version of this historical standard is referenced on www.astm.org.

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 available[4]. 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 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 HNO_3 (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 HNO_3. Add 250 mL of $HClO_4$ 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-µm) 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-µm) 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 below the boiling point until no further attack is apparent. Add 5 mL of HNO_3 and heat for 10 min. Remove the cover, add 3 mL of HF, and heat for 10 min. Rinse the wall of the beaker with water, add 5 mL of $HClO_4$, and evaporate slowly to dense white fumes of $HClO_4$. Fume for 2 min to 3 min and allow the solution to cool. Add 50 mL of water and warm until the soluble salts are in solution. Filter the solution through a fine texture paper, wash the residue with warm water, receiving the filtrate and washings in a 100-mL volumetric flask. Ignite the filter paper containing the insoluble residue in a platinum crucible, moisten the residue with a few drops of water, add about 3 mL of HF and 2 drops of H_2SO_4. Add a few drops of

---

[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.

H_2NO_3. Mix and evaporate to fumes of H_2SO_4. Cool, cautiously add 2 mL to 3 mL of water to dissolve the salts, and combine with the reserved filtrate in the volumetric flask. Cool, dilute to volume, and mix. Use the test solution directly if the expected content of copper in the sample is between 0.003 % and 0.2 %. If the copper content exceeds 0.2 %, transfer 20 mL of the test solution to a 100-mL volumetric flask, add 16 mL of iron background solution, dilute to volume, and mix.

11.4 *Reagent Blank Solution*—Transfer the reagent blank solution to a 100-mL volumetric flask, add 20 mL of iron background solution, dilute to volume, and mix.

11.5 *Adjustment of Atomic Absorption Spectrometer*—In accordance with Practice E663, set the initial instrument parameters to the values in Note 4 (7.1), light the burner, and aspirate water until the instrument comes to thermal equilibrium. Optimize instrument response by adjusting the wavelength, fuel, air, burner, and nebulizer while aspirating the highest calibration solution to obtain maximum absorption or absorbance. Aspirate water until a steady signal is obtained and adjust the instrument readout system to obtain zero absorption or absorbance.

NOTE 6—The manufacturer's instructions for igniting and extinguishing the air – acetylene burner should be strictly followed to avoid possible explosion hazards. Tinted safety glasses should be worn by the operator whenever the flame is burning.

NOTE 7—Optimize the adjustment of the background corrector, if necessary (copper range from 0.003 % to 0.010 %).

11.6 *Measurements*—Aspirate water until the initial reading is again obtained. Aspirate the calibration and test solutions in the order of increasing absorption, starting with the reagent blank and reference solution. When a stable response is obtained for each solution, record the readings. Aspirate and record the readings of the test solutions at the proper points in the calibration series. Aspirate water between each calibration and test solution. Repeat all measurements at least two more times.

TABLE 1 Precision Data

Iron ore	*X*	Repeatability (R_1, E173)	Reproducibility (R_2, E173)	*n*
Malmbergets concentrate	0.001	0.0008	0.0014	35
Kiruna	0.011	0.0020	0.0022	39
Purpurerz	0.072	0.0036	0.0109	39
Russ Abbrande	0.380	0.0165	0.0358	40
Forsbo	0.787	0.0322	0.0521	40

where:

X = average copper content, %, and
n = number of laboratories.

12. Calculation

12.1 If necessary, convert the average of the readings for each calibration solution to absorbance. Obtain the net absorbance of each calibration solution by subtracting the average absorbance of the reference solution. In a similar manner, obtain the net absorbance of the test solution by subtracting the absorbance of the reagent blank solution.

12.2 Prepare a calibration curve by plotting the net absorbance values of the calibration solutions against micrograms of copper per milliliter.

12.3 Convert the net absorbance value of the sample solution to micrograms of copper per milliliter by means of the calibration curve.

12.4 Calculate the percentage of copper content as follows:

$$\text{Copper, \%} = \frac{A}{B \times 100} \tag{1}$$

where:

A = copper content of the test solution, µg/mL and
B = weight of sample used, g.

13. Precision and Bias[5]

13.1 *Precision*—Table 1 indicates the precision of the test method within and between laboratories.

13.2 *Bias*—No information on the bias of this test method is known. Accepted reference materials may have not been included in the materials used in the interlaboratory study. Users of the method are encouraged to employ accepted reference materials, if available, and to judge the bias of the method from the difference between the accepted value for the copper content and the mean value from interlaboratory testing of the reference material.

NOTE 8—This test method has been evaluated in accordance with Practice E173 (withdrawn 1997). The Reproducibility R_2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. The Repeatability R_1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601.

14. Keywords

14.1 agglomerates; concentrates; copper content; flame atomic absorption spectrometry; iron ores

---

[5] Supporting data giving the results of cooperative testing are available from ASTM International Headquarters. Request RR:E16-1004.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).