



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

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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 %.

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:

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals²

E 276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials²

E 663 Practice for Flame Atomic Absorption Analysis³

E 877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials³

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory³

3. Summary of Test Method

3.1 The sample is dissolved in hydrochloric acid with the addition of a small amount of nitric and hydrofluoric acids. After evaporation to fumes with perchloric acid, the solution is diluted with water and filtered. A portion of the solution is examined by atomic absorption spectroscopy using standards containing approximately the same amount of iron as the test sample.

¹ This test method is under the jurisdiction of ASTM Committee E-1 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 *Annual Book of ASTM Standards*, Vol 03.05.

³ *Annual Book of ASTM Standards*, Vol 03.06.

4. Significance and Use

4.1 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 concentration for metallurgical consideration.

4.2 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 E 882.

5. Interferences

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

6. Apparatus

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

6.1.1 *Minimum Sensitivity*—The absorbance of the highest calibration solution (see 7.5) must be at least 0.3.

6.1.2 *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 7.5).

6.1.3 *Minimum Stability*—The coefficient of variation 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 1—A strip chart recorder or digital readout device, or both is advisable to measure the criteria in 6.1 and for all subsequent measurements.

NOTE 2—A background corrector equipped with a hydrogen or a deuterium hollow cathode lamp is advisable for the concentration range from 0.003 to 0.010 % Cu.

NOTE 3—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

7. Reagents and Materials

7.1 *Purity and Concentration of Reagents*—The purity and concentration of the common chemical reagents used shall conform to Practice E 50. Special apparatus and reagents required are located in separate section preceding the procedure.

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

7.3 *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.

7.4 *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.

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

7.5.1 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.

7.6 *Hydrochloric Acid* (HCl) (1 + 1)—Mix 1 volume of hydrochloric acid (HCl) with 1 volume of water.

7.7 *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 perchloric acid (HClO₄) and evaporate to fumes. Allow to fume for 10 min, cool, and dilute to 1 L.

7.8 *Nitric Acid* (HNO₃) (1 + 1)—Mix 1 volume of nitric acid (HNO₃) with 1 volume of water.

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

8. Sampling, Test Specimens, and Test Units

8.1 Collect and prepare the test unit in accordance with Practice E 877.

8.2 The analytical sample shall be pulverized so that at least 95% passes a No. 100 (150- μ m) sieve in accordance with Test Methods E 276.

NOTE 4—To facilitate decomposition, some ores, such as specular hematite, require grinding to pass a No. 200 (75- μ m) sieve.

9. Procedure

9.1 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 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 weight loss. Transfer the sample to a 250-mL beaker and reweigh the capped bottle to the nearest 0.1 mg. The difference between the two weights is the weight of the sample taken for analysis.

9.2 Carry a reagent blank through all steps of the procedure starting with 9.3.

9.3 *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 HNO₃ and heat for 10 min. Remove cover, add 3 mL of hydrofluoric acid, and heat for 10 min. Rinse wall of the beaker with water, add 5 mL of HClO₄, and evaporate slowly to dense white fumes of HClO₄. Fume for 2 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 hydrofluoric acid and 2 drops of sulfuric acid. Add a few drops of nitric acid. Mix and evaporate to fumes of sulfuric acid. Cool, cautiously add 2 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 concentration exceeds 0.2 %, transfer 20 mL of the test solution to a 100-mL volumetric flask, add 16 mL of iron background solution, dilute the volume, and mix.

9.4 *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.

9.5 *Adjustment of Atomic Absorption Spectrometer*—In accordance with Practice E 663, set the initial instrument parameters to the values in Note 3, 6.1, 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 5—The manufacturers 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 6—Optimize the adjustment of the background corrector, if necessary (copper range from 0.003 to 0.010 %).

9.6 *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.

10. Calculation

10.1 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.

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

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

10.4 Calculate the percentage of copper as follows:

$$\text{Copper, \%} = (A \times 100)/(B \times 10\,000) \quad (1)$$

where:

A = micrograms of copper per millilitre, and

B = grams of sample represented in the 100-mL volume of the final test solution.

11. Precision and Bias ⁴

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

11.2 *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.

12. Keywords

12.1 **agglomerates; concentrates; copper content; iron ores**

⁴Supporting data giving the results of cooperative testing are available from ASTM Headquarters. Request RR: E16-1004.

TABLE 1 Precision Data

Iron ore	<i>X</i>	Repeatability (<i>R</i> ₁ , E173)	Reproducibility (<i>R</i> ₂ , E173)	<i>n</i>
Malmbergets concentrate	0.001	0.0008	0.0014	35
Kiruna	0.011	0.0020	0.0022	39
Purpurez	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 concentration, %, and

n = number of laboratories.

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