



# Standard Test Method for Determination of Silver in Copper Concentrates by Flame Absorption Spectrometry<sup>1</sup>

This standard is issued under the fixed designation E 1898; 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 silver in the range of 50 µg/g to 1000 µg/g by acid dissolution of the silver and measurement by atomic absorption spectrophotometry. Copper concentrates are internationally traded within the following concentration ranges:

Element	Unit	Concentration Range		
Aluminum	%	0.05	to	2.50
Antimony	%	0.0001	to	4.50
Arsenic	%	0.01	to	0.50
Barium	%	0.003	to	0.10
Bismuth	%	0.001	to	0.16
Cadmium	%	0.0005	to	0.04
Calcium	%	0.05	to	4.00
Carbon	%	0.10	to	0.90
Chlorine	%	0.001	to	0.006
Chromium	%	0.0001	to	0.10
Cobalt	%	0.0005	to	0.20
Copper	%	10.0	to	44.0
Fluorine	%	0.001	to	0.10
Gold	µg/g	1.40	to	100.0
Iron	%	12.0	to	30.0
Lead	%	0.01	to	1.40
Magnesium	%	0.02	to	2.00
Manganese	%	0.009	to	0.10
Mercury	µg/g	0.05	to	50.0
Molybdenum	%	0.002	to	0.25
Nickel	%	0.0001	to	0.08
Silicon	%	0.40	to	20.0
Silver	µg/g	18.0	to	8000
Sulfur	%	10.0	to	36.0
Tin	%	0.004	to	0.012
Zinc	%	0.005	to	4.30

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 its use. Specific precautions are given in WARNING paragraphs.

## 2. Referenced Documents

### 2.1 ASTM Standards:

E 29 Practice For Using Significant Digits in Test Data to

<sup>1</sup> 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 Metal Bearing Ores, Concentrates and Related Metallurgical Materials.

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### Determine Conformance With Specifications<sup>2</sup>

E 50 Practices for Apparatus, Reagents and Safety Precautions for Chemical Analysis of Metals<sup>2</sup>

E 663 Practice for Flame Atomic Absorption Analysis<sup>3</sup>

E 882 Guide for Accountability and Quality Control in the Chemical Analysis of Metals<sup>3</sup>

E 1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry<sup>3</sup>

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method<sup>2</sup>

E 1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods<sup>2</sup>

## 3. Summary of Test Method

3.1 The analyst has the option of either digesting the sample in nitric and hydrochloric acids or nitric and perchloric acids, depending on their preference and equipment availability. The filtered solutions are aspirated into an air-acetylene flame of an atomic absorption spectrophotometer. Spectral energy at approximately 328.1 nm from a silver hollow cathode lamp is passed through the flame and the absorbance is measured. This absorbance is compared with the absorbance of a series of standard calibration solutions.

## 4. Significance and Use

4.1 In the primary metallurgical processes used by the mineral processing industry for copper bearing ores, copper and silver associated with sulfide mineralization are concentrated by the process of flotation for recovery of the metals.

4.2 This test method is intended to be a referee method for the determination of silver in copper concentrates. It is assumed that all who use this procedure 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 Elements normally found in copper concentrates do not

<sup>2</sup> Annual Book of ASTM Standards, Vol 03.05.

<sup>3</sup> Annual Book of ASTM Standards, Vol 03.06.

interfere. Use of instrumental background correction is required to compensate for non specific absorption interferences in the flame.

## 6. Apparatus

6.1 *Atomic Absorption Spectrophotometer*, capable of resolving the 328.1 nm silver line, equipped with an air-acetylene premix burner and a silver hollow cathode lamp. The performance of the instrument must be such that the response of silver absorbance is linear over the range of 0.5 to 4.0 µg/mL.

## 7. Reagents and Materials

7.1 *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.<sup>4</sup> 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.

7.2 *Silver Standard Solution A* (1 mL = 1 mg Ag)—Dissolve 1000 g of silver metal (purity: 99.99 %, minimum) in 50 mL of nitric acid (1+1). Dilute to about 100 mL and boil gently to expel the oxides of nitrogen. Cool and transfer to a 1-L volumetric flask. Dilute to the mark and mix. Solution must be clear, otherwise, discard it and repeat the preparation. Store in a dark bottle.

NOTE 1—All reagents must be free of chloride contamination.

7.3 *Silver Standard Solution B* (1 mL = 10 µg Ag)—Pipet 10 mL of Silver Standard Solution A into a 1-L volumetric flask containing about 500 mL of water and 250 mL of hydrochloric acid. Mix, cool, dilute to the mark, and remix. If turbid, discard the solution and repeat the preparation. Transfer to a dark bottle. Prepare immediately before use.

## 8. Hazards

8.1 For precautions to be observed in the use of certain reagents and equipment in this test method, refer to Practices E 50.

## 9. Sampling and Sample Preparation

9.1 The gross sample must be collected and prepared so that it is representative of the lot of copper concentrate to be analyzed. The laboratory sample must be pulverized, if necessary, so that 100 % passes a No. 100 (150 µm) sieve.

## 10. Calibration and Standardization

10.1 *Calibration Solutions*—By means of pipets, transfer 0, 5, 10, 20, 30, and 40 mL of Silver Standard Solution B into 100-mL volumetric flasks. Dilute to the mark with HCl (1+3) and mix. These solutions are 0, 0.5, 1.0, 2.0, 3.0, and 4.0 µg Ag/mL, respectively.

10.2 *Photometry*:

10.2.1 With the silver hollow cathode lamp in position, energized and stabilized, adjust the wavelength to maximize the energy response of the 328.1 nm line.

10.2.2 Light the burner, allow it to reach thermal equilibrium, and adjust the instrument to zero while aspirating water. Aspirate the silver solution with the highest concentration from the series prepared in 10.1 and adjust the burner, acetylene, and air flow rates to obtain maximum response. Whenever one or more of these parameters is changed, recalibration is necessary.

10.2.3 Aspirate the silver solution used in 10.2.2 to ensure that the absorbance reading is repeatable. Record six absorbance readings, and calculate the standard deviation, *s*, of the readings, as follows:

$$s = (A-B) \times 0.40 \quad (1)$$

where:

*A* = highest of the six values found, and

*B* = lowest of the six values found.<sup>5</sup>

10.2.4 Using water as a zero reference, and beginning with the solution to which no addition of silver was made in 10.1, aspirate each calibration solution in ascending order of concentration, and record its absorbance. If the value of the solution with the highest concentration used in 10.1 differs from the average of the six values in 10.2.3 by more than twice the standard deviation, or by more than 0.01 multiplied by the average of the six values, whichever is greater, repeat the measurement. If a problem is indicated, determine the cause, take appropriate corrective measures, and repeat 10.2.1-10.2.4.

10.2.5 *Calibration Curve*—Plot the average net absorbance values versus micrograms of silver per millilitre on rectangular coordinate paper. Test for linearity as described in Guide E 1024.

## 11. Procedure

### 11.1 Test Solution:

11.1.1 Select three test samples for each concentrate and transfer an appropriate amount to tared weighing vessels. Dry at 105°C in a drying oven for at least two hours to constant weight. Select final weights in accordance with the following:

Est. Ag, µg/g	Sample Wt., g	Wt. Tolerance, mg	Dil., mL
20-150	2.0	0.1	100
150-300	2.0	0.1	200
300-500	1.0	0.1	200
500-1000	1.0	0.1	500
1000-3000	1.0	0.1	1000

Transfer to a 250-mL beaker.

### 11.1.2 Nitric and Hydrochloric Acid Decomposition:

11.1.2.1 Place each test sample in a beaker as in 11.1.1. Add 35 mL HNO<sub>3</sub> (1+1), cover with a watchglass, and heat gently until dissolution is complete. Add 10 mL HCl, slip cover aside, and evaporate to dryness. Dissolve the dry salts in 25 mL HCl (1+3). Heat to boiling, then cool. Rinse watchglass and beaker with HCl (1+3) and filter through a dry high-wet strength, medium-porosity filter paper<sup>5</sup> into a clean volumetric flask, as shown in 11.1.1. Wash the paper eight times with HCl (1+3) and discard the paper. Dilute to volume with HCl (1+3) and mix.

<sup>4</sup> "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. Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

<sup>5</sup> Whatman No. 54 or No. 541 has been found suitable.