



Designation: E1898 – 21

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

This standard is issued under the fixed designation E1898; 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 ( $\epsilon$ ) 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 13  $\mu\text{g/g}$  to 500  $\mu\text{g/g}$  by acid dissolution of the silver and measurement by atomic absorption spectrometry. Copper concentrates are internationally traded within the following content ranges:

Element	Unit	Content 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	$\mu\text{g/g}$	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	$\mu\text{g/g}$	0.05	to 50.0
Molybdenum	%	0.002	to 0.25
Nickel	%	0.0001	to 0.08
Silicon	%	0.40	to 20.0
Silver	$\mu\text{g/g}$	18.0	to 8000
Sulfur	%	10.0	to 36.0
Tin	%	0.004	to 0.012
Zinc	%	0.005	to 4.30

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

<sup>1</sup> 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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1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D1193 Specification for Reagent Water

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

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

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 (Withdrawn 2015)<sup>3</sup>

## 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 analyst has the option of either digesting the sample in  $\text{HNO}_3$  and  $\text{HCl}$  or  $\text{HNO}_3$  and  $\text{HClO}_4$ , depending on their preference and equipment availability. The filtered solutions are aspirated into an air-acetylene flame of an atomic absorption spectrometer. Spectral energy at approximately 328.1 nm from a silver hollow cathode lamp is passed through the flame

<sup>2</sup> 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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

and the absorbance is measured. This absorbance is compared with the absorbance of a series of standard calibration solutions.

## 5. Significance and Use

5.1 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.

5.2 This test method is a comparative method and is intended to be a referee method for compliance with compositional specifications for metal content or to monitor processes.

5.3 It is assumed that all who use this method will be trained analysts capable of performing 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 Elements normally found in copper concentrates do not interfere. Use of instrumental background correction is required to compensate for non specific absorption interferences in the flame.

## 7. Apparatus

7.1 *Atomic Absorption Spectrometer*, 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 µg/mL to 4.0 µg/mL.

## 8. Reagents and Materials

8.1 *Silver Calibration Solutions*—By means of pipets, transfer (0, 5, 10, 20, 30, and 40) mL of Silver Standard Solution B (8.3) 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/mL, respectively.

8.2 *Silver Standard Solution A* (1 mL = 1 mg Ag)—Dissolve 1000 g of silver metal (purity: 99.99 %, minimum) in 50 mL of HNO<sub>3</sub> (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.

8.2.1 A certified reference solution meeting these specifications may also be used.

8.3 *Silver Standard Solution B* (1 mL = 10 µg Ag)—Pipet 10 mL of Silver Standard Solution A (8.2) into a 1 L volumetric flask containing about 500 mL of water and 250 mL of HCl. 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.

8.4 *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.

8.5 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I or II of Specification D1193. Other reagent water types, Type III or Type IV, may be used if they do not cause silver precipitation or effect non-measurable change in the blank or sample.

## 9. Hazards

9.1 For precautions to be observed in the use of certain reagents and equipment in this test method, refer to Practices E50.

## 10. Sampling and Sample Preparation

10.1 The gross sample must be collected and prepared so that it is representative of the lot of copper concentrate to be analyzed.

NOTE 2—Gross samples may need to be dried at low temperature in order to determine the mercury content without drying losses.

10.2 The laboratory sample must be ground, if necessary, so that 100 % passes a 150 µm sieve.

NOTE 3—Verify the adequacy of grind on a separate sub-sample. Do not pass the laboratory sample through the 150 µm sieve.

## 11. Preparation of Apparatus

11.1 Follow the instrument manufacturer's instructions to adjust the instrument for silver at 328.1 nm. Warm up the instrument with background correction applied in accordance with the manufacturer's instructions.

11.2 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.

11.3 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 8.1 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.

11.4 Aspirate the silver solution used in 11.3 to ensure that the absorbance reading is repeatable. Record six absorbance readings, and calculate the standard deviation, *s*, of the readings, as follows:

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

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC, [www.chemistry.org](http://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>.

where:

- $A$  = highest of the six values found, and  
 $B$  = lowest of the six values found.

NOTE 4—Can also be calculated as follows:

$$s = \sqrt{\frac{\sum(c - d)^2}{(e - 1)}}$$

where:

- $c$  = individual absorbance readings,  
 $d$  = mean of absorbance readings, and  
 $e$  = number of readings (6).

11.5 Using water as a zero reference, and beginning with the solution to which no addition of silver was made in 8.1, 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 8.1 differs from the average of the six values in 11.4 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 11.2 – 11.5.

## 12. Calibration

12.1 *Calibration Curve*—Plot the average net absorbance values versus micrograms of silver per millilitre on rectangular coordinate paper or calibrate in accordance with the manufacturer’s instructions.

12.2 Using water as a zero reference, aspirate calibration standards alternately until stable readings occur (within 2 %). Alternate readings: three samples, a standard, three samples and another standard, etc., until the absorbance for all samples and standards have been determined at least three times. (The absorbances should repeat within 2 %). Average all values for standards and samples. Plot curve of absorbance versus  $\mu\text{g/mL}$  silver in the standards. Read sample absorbance from the curve to obtain  $\mu\text{g/mL}$  values of the samples. Standards and blank must plot a straight line near zero absorbance on the graph.

## 13. Procedure

### 13.1 Test Solution:

13.1.1 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, $\mu\text{g/g}$	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.

### 13.1.2 $\text{HNO}_3$ and $\text{HCl}$ Decomposition:

13.1.2.1 Place each test sample in a beaker as in 13.1.1. Add 35 mL  $\text{HNO}_3$  (1+1), cover with a watch glass, and heat gently until dissolution is complete. Add 10 mL  $\text{HCl}$ , slipcover aside, and evaporate to dryness. Dissolve the dry salts in 25 mL  $\text{HCl}$  (1+3). Heat to boiling, then cool. Rinse watch glass and beaker with  $\text{HCl}$  (1+3) and filter through a dry high-wet strength,

medium-porosity filter paper<sup>5</sup> into a clean volumetric flask. Wash the paper eight times with  $\text{HCl}$  (1+3) and discard the paper. Dilute to volume with  $\text{HCl}$  (1+3) and mix.

NOTE 5—Some copper concentrates may not be totally decomposed by these digestion methods. The user is advised to check any residue from the digestion procedure for residual silver.

### 13.1.3 $\text{HNO}_3$ and $\text{HClO}_4$ Decomposition:

13.1.3.1 Place each test sample in a beaker as in 13.1.1. Add 35 mL  $\text{HNO}_3$  (1+1), cover with a watch glass, and heat gently until dissolution is complete and oxides of nitrogen are dispelled. Cautiously add 10 mL of  $\text{HClO}_4$  and continue heating (see Note 6). Evaporate to dense white fumes. Continue fuming to near dryness.

NOTE 6—**Warning**—Handle  $\text{HClO}_4$  in accordance with Practices E50 as follows:

$\text{HClO}_4$  can be used safely, but only under carefully prescribed conditions. Dilute  $\text{HClO}_4$  has the same hazardous properties as other strong acids, but the concentrated acid, especially when it is hot, reacts rapidly and often with violently explosive force with oxidizable materials. Only well-established procedures should be employed for  $\text{HClO}_4$  oxidations and the procedures should be followed *exactly* as written. Specially designed hoods are specified for handling  $\text{HClO}_4$  fumes and any hood in which  $\text{HClO}_4$  may be fumed should not be used for other operations that permit easily oxidizable material to collect in the ducts or blower.

13.1.3.2 Cool slightly and dissolve salts in 35 mL  $\text{HCl}$  (1+3). Heat to boiling. Allow to cool again. Rinse watch glass and beaker with  $\text{HCl}$  (1+3) and filter through a dry, high wet-strength, medium-porosity filter paper<sup>5</sup> into a volumetric flask. Wash the paper eight times with  $\text{HCl}$  (1+3) and discard the paper. Dilute to volume with  $\text{HCl}$  (1+3) and mix.

13.2 Prepare a reagent blank by treating the same amount of all reagents as directed in 13.1.2 or 13.1.3. Use reagents from the same lots for blank and test solutions.

13.3 *Measurement*—Using water as a zero reference, aspirate calibration standards alternately until stable readings occur (within 2 %). Alternate readings: three samples, a standard, three samples and another standard, etc., until the absorbance for all samples and standards have been determined at least three times. (The absorbances should repeat within 2 %). Average all values for standards and samples. Plot curve of absorbance versus  $\mu\text{g/mL}$  silver in the standards. Read sample absorbance from the curve to obtain  $\mu\text{g/mL}$  values of the samples. Standards and blank must plot a straight line near zero absorbance on the graph.

## 14. Calculation

14.1 Convert the absorbance of the test sample solutions and the reagent blank solution to micrograms of silver per millilitre of the final dilution volume by means of the calibration curve. Calculate the content of silver as follows:

$$E = C(A - B)/D \quad (2)$$

where:

- $A$  =  $\mu\text{g}$  of silver per mL in the final test solution,  
 $B$  =  $\mu\text{g}$  of silver per mL in the final reagent blank,  
 $C$  = final volume of test solution, mL,

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