Standard Test Method for Determination of Gold in Copper Concentrates by Fire Assay Gravimetry[1]

This standard is issued under the fixed designation E1805; 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 is for the determination of gold in copper concentrates in the content range from 0.2 µg/g to 17 µg/g.

NOTE 1—The lower scope limit is set in accordance with Practice E1601.

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.* For specific warning statements, see 11.3.1, 11.5.4, and 11.6.5.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E135.

4. Summary of Test Method

4.1 A test sample of copper concentrate is fluxed and fused in a clay crucible. The precious metals are reduced, collected in a lead button, and then cupelled to remove the lead. The remaining doré bead is parted with nitric acid to remove the silver and other impurities from the gold. The gold is then annealed, cleaned, and weighed on a microbalance.

5. Significance and Use

5.1 In the metallurgical process used in the mining industries, gold is often carried along with copper during the flotation concentration process. Metallurgical accounting, process control, and concentrate evaluation procedures for this type of material depend on an accurate, precise measurement of the gold in the copper concentrate. This test method is intended to be a reference method for metallurgical laboratories and a referee method to settle disputes in commercial transactions. It is also a definitive method intended to test materials for compliance with compliance with compositional specifications and to provide data for certification of reference materials. It is essential that each performance of the method be validated by applying it to appropriate reference materials at the same time and in the same manner as it is applied to the unknowns.

5.2 It is assumed that all who use this test method will be trained analysts capable of performing skillfully and safely. It is expected that the work will be performed in a properly equipped laboratory under appropriate quality control practices such as those described in Guide E882.

6. Interferences

6.1 Elements normally found in copper concentrates within the limits of 1.1 do not interfere. High concentrations of arsenic, antimony, tellurium, bismuth, nickel, and platinum group metals (and, in some instances, copper), however, may interfere with the fusion and cupellation steps.

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[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.

7. Apparatus

7.1 *Analytical Balance,* capable of weighing to 0.1 g.

7.2 *Assay Mold,* 100-mL capacity.

7.3 *Cube or Cone Mixer,* 1000-g capacity.

7.4 *Cupel,* magnesite or bone ash, 40-g lead capacity.

7.5 *Drying Oven,* forced air circulation with temperature control, 104 °C.

7.6 *Fire Assay Bead Brush.*

7.7 *Fire Assay Bead Pliers.*

7.8 *Fire Assay Clay Crucible,* 20-g to 30-g sample capacity.

7.9 *Fire Assay Muffle Furnace,* gas-fired or electric, equipped with air circulation systems and with draft controls, capable of temperatures to 1100 °C ± 10 °C, and with ventilation controls for acid and lead fumes.

7.10 *Fire Assay Tongs,* crucible and cupel.

7.11 *Fire Assay Tumble Mixer*—an industrial mixer-crucible tumbler.

7.12 *Hot Plate,* with variable temperature control and ventilation controls for acid fumes.

7.13 *Jaw Crusher,* capable of reducing cupels and slag to pass a 4-mm sieve.

7.14 *Ring Pulverizer,* capable of 250-g minimal capacity.

7.15 *Semi-Microbalance,* capable of weighing to 0.001 mg.

7.16 *Steel Hammer.*

8. Reagents and Materials

8.1 *Borax Glass ($Na_2B_4O_7$).*

8.2 *Cupel Correction Flux*—Blend the following ingredients in the listed proportions:

Borax Glass	15 g
Flour	2 g
Lead Oxide	30 g
Potassium Carbonate	45 g
Silica	12 g

8.3 *Fire Assay Flux Mixture*—Blend the following ingredients in the listed proportions:

Borax Glass	15 g
Lead Oxide	55 g
Potassium Carbonate	6 g
Potassium Nitrate	13 g
Silica	6 g
Sodium Carbonate	20 g

NOTE 2—Perform a preliminary fusion to determine lead button weight. If a 30-g to 40-g lead button is not obtained, adjust the amount of KNO_3 and try again. Increasing the KNO_3 produces a smaller lead button, and decreasing the KNO_3 produces a larger one.

8.4 *Flour,* ground wheat.

8.5 *Lead Oxide, Litharge, (PbO)*—Containing less than 0.02 µg/g gold and less than 0.40 µg/g silver.

8.6 *Potassium Carbonate,* Potash (K_2CO_3).

8.7 *Potassium Nitrate,* Niter (KNO_3).

8.8 *Silica (SiO_2),* 95 % minimum purity, particle size less than 180 µm.

8.9 *Silver Foil,* 99.9 % purity with less than 0.10 µg/g gold content.

8.10 *Silver Solution (1 g/L)*—Add 1.557 g silver nitrate to 1000 mL of water containing 5 mL of HNO_3, mix. Store in a dark bottle.

8.11 *Sodium Carbonate,* Soda Ash (Na_2CO_3), anhydrous technical grade.

8.12 *Sodium Chloride,* salt (NaCl).

8.13 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used provided it is first ascertained that the reagents are of sufficiently high purity to permit their use without lessening the accuracy of the determination.[3]

8.14 *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. Type III or IV may be used if they effect no measurable change in the blank or sample.

9. Hazards

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

9.2 See specific warnings in 11.3.1, 11.5.4, 11.6.5.

10. Sampling and Sample Preparation

10.1 Collect, store, and handle gross samples in accordance with the safety and materials guidelines in Practice E50. Gross samples must be free of all extraneous materials.

10.2 Dry the laboratory sample to constant weight at 104 °C.

NOTE 3—If the gross sample was dried at a low temperature (e.g. 60 °C for mercury) take the low temperature portion(s) and a separate moisture sample, prior to drying at 104 °C.

10.3 Grind the laboratory sample in a ring mill so that 100 % passes through a 150-µm sieve and blend the prepared sample in a cube or cone blender, if necessary to further reduce the heterogeneity of the laboratory sample. Obtain the test samples by incremental division by mixing the prepared sample and spreading it on a flat non moisture-absorbing surface so that the prepared sample forms a rectangle of uniform thickness. Divide into at least 20 segments of equal area. With a flat bottom, square-nose tool, take scoopfuls of approximately equal size from each segment from the full depth of the bed. Combine the scoopfuls to form the test sample.

NOTE 4—Verify the adequacy of the grind on a separate sub-sample, do

[3] *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 *Reagent Chemicals and Standards*, by Joseph Rosin, D. Nostrand Co., Inc., New York, NY, and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.