



Designation: E 1805 – 02

Standard Test Method for Fire Assay Determination of Gold in Copper Concentrates by Gravimetry¹

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1. Scope

1.1 This test method is for the determination of gold in copper concentrates in the concentration range from 0.2 to 17 $\mu\text{g/g}$ (0.007 to 0.500 Troy oz/short ton).

NOTE 1—The lower scope limit is set in accordance with Practice E 1601.

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

2. Referenced Documents

- 2.1 *ASTM Standards:*²
 - D 1193 Specification for Reagent Water
 - E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
 - E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals
 - E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
 - E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods
 - E 882 Guide for Accountability and Quality Control in the Quality Control Laboratory
 - E 1601 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 E 135.

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.

4. Summary of Test Method

4.1 A test sample of copper concentrate is fused in a clay crucible. The precious metals are reduced, collected in a lead button, and then cupelled in a bone ash crucible, (cupel), 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 then is 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.

5.2 It is assumed that all who use this test method will be trained analysts capable of performing common laboratory operations 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 E 882.

6. Interferences

6.1 Elements normally found in copper concentrates 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.

7. Apparatus

- 7.1 *Analytical Balance*, capable of weighing to 0.1 g.
- 7.2 *Semi-Microbalance*, capable of weighing to 0.001 mg.
- 7.3 *Assay Mold*, 100-mL capacity.
- 7.4 *Cube or Cone Mixer*, 1000-g capacity.
- 7.5 *Cupel*, magnesite or bone ash—40-g lead capacity.
- 7.6 *Dry Oven*, forced air circulation with temperature control, 104°C.
- 7.7 *Fire Assay Bead Brush*.
- 7.8 *Fire Assay Bead Pliers*.

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

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

7.14 *Jaw Crusher*, capable of reducing cupels and slag to 60 mesh.

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

7.16 *Steel Hammer*.

8. Reagents and Materials

8.1 *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

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type T of Specification D 1193.

8.3 *Ammonium Hydroxide* (NH₄OH).

8.4 *Borax Glass* (Na₂B₄O₇).

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

8.6 *Potassium Carbonate*, Potash (K₂CO₃).

8.7 *Potassium Nitrate*, Niter (KNO₃).

8.8 *Silica Sand* (SiO₂)—95 % minimum purity, particle size less than 80 mesh.

8.9 *Sodium Chloride*, salt (NaCl).

8.10 *Silver Foil*, 99.9 % purity with less than 0.10-ppm gold content.

8.11 *Silver Solution* (1 g/L)—Add 1.557 g silver nitrate to 1000 mL of water containing 5 mL of nitric acid. Store in a dark bottle.

8.12 *Sodium Carbonate*, *Soda Ash* (Na₂CO₃), anhydrous technical grade.

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

Borax glass	15 g
Flour, white wheat	2 g
Lead oxide	30 g
Potassium carbonate	45 g
Silica	12 g

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

Borax glass (Na ₂ B ₄ O ₇)	15 g
Lead oxide (PbO)	55 g
Potassium carbonate (K ₂ CO ₃)	6 g
Potassium nitrate (KNO ₃)	13 g
Silica (SiO ₂)	6 g
Sodium carbonate (Na ₂ CO ₃)	20 g

NOTE 2—Perform a preliminary fusion to determine lead button weight. If a 30 to 40-g lead button is not obtained, adjust the amount of KNO₃ and try again. Increasing the KNO₃ produces a smaller lead button, and decreasing the KNO₃ produces a larger one.

9. Hazards

9.1 For precautions to be observed in the use of certain reagents in this test method, refer to Practice E 50.

10. Sampling and Sample Preparation

10.1 Collect, store, and handle gross samples in accordance with the safety and materials guidelines in Practice E 50. Gross samples must be free of all extraneous materials.

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

10.3 Grind the laboratory sample in a ring mill so that 100% passes through a No. 100 (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 scoopfulls of approximately equal size from each segment from the full depth of the bed. Combine the scoopfulls to form the test sample.

NOTE 3—Verify the adequacy of the grind on a separate sub-sample, do not pass the laboratory sample through the No. 100 sieve.

11. Procedure

11.1 *Crucible Preparation:*

11.1.1 Add 106 g of fire assay flux mixture to each clay fire assay crucible.

11.1.2 Weigh duplicate test samples: 14.583 ± 0.001 g (½ assay ton). Record the test sample weights. Transfer the test samples to the fire-assay crucibles.

11.1.3 Mix the contents of the crucible for 2 min in a crucible tumble mixer.

11.1.4 *Inquartation*—Based on the preliminary assay or an estimate, dispense the silver solution over the top portion of the mixed fire assay clay crucible to achieve an Ag to Au ratio of 10 to 1. Alternatively, add silver foil in small chips to make the same ratio.

11.1.5 Place 55 g of litharge (PbO) on top of the mixed fire assay clay crucible. Top with 3 g salt, (NaCl).

TABLE 1 Troy Conversion

Desired Units	Conversion Factor
ppm, mg/kg or g/metric ton	1
Troy oz/short ton	0.02917

11.2 *Fusion:*

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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.