



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

This standard is issued under the fixed designation E 1805; 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 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.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals³

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials³

E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods⁴

E 882 Guide for Accountability and Quality Control in the Quality Control Laboratory²

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method⁴

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E 135.

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.

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 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.06.

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 $\pm 10^\circ\text{C}$, 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.⁵

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* (NH_4OH).

8.4 *Borax Glass* ($\text{Na}_2\text{B}_4\text{O}_7$).

8.5 *Lead Oxide*, Litharge, (PbO)—Containing less than 0.02 $\mu\text{g/g}$ gold and less than 0.40 $\mu\text{g/g}$ silver.

8.6 *Potassium Carbonate*, Potash (K_2CO_3).

8.7 *Potassium Nitrate*, Niter (KNO_3).

8.8 *Silica Sand* (SiO_2)—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* (Na_2CO_3), 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 ($\text{Na}_2\text{B}_4\text{O}_7$)	15 g
Lead oxide (PbO)	55 g
Potassium carbonate (K_2CO_3)	6 g
Potassium nitrate (KNO_3)	13 g
Silica (SiO_2)	6 g
Sodium carbonate (Na_2CO_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 KNO_3 and

⁵ *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.

try again. Increasing the KNO_3 produces a smaller lead button, and decreasing the KNO_3 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 Pass the laboratory sample through a No. 100 (150- μm) sieve, grind the oversize material in a ring mill until it also passes the sieve, and blend the prepared sample in a cube or cone blender. Obtain the test samples by incremental division by mixing the prepared sample and spreading it on a flat nonmoisture-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.

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 ($\frac{1}{2}$ 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 Au to Ag 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).

11.2 *Fusion*:

11.2.1 *Primary*—Place the prepared crucibles carefully into a room temperature fire assay furnace. Raise the temperature as rapidly as possible to 600°C and hold at that point until the salt cover melts. Elapsed time should be approximately 25 min.

11.2.2 *Secondary*—Increase temperature to 950°C and hold until slag formation is completed. Time required is about 10 min.

11.2.3 *Tertiary*—Increase temperature to 1100°C and let the fusion material liquify completely. Total time in the fire assay furnace for all three fusion stages should be about 45 min.

11.3 *Pouring*:

11.3.1 When the fusion is completed, remove the crucible with the crucible tongs. Slowly swirl the crucible several times, tap the crucible lightly on an iron plate or table, then pour the contents in the fusion assay mold.

NOTE 3—**Warning:** Rapidly cooling slag may eject sharp flying fragments. Cover the mold with a wire screen immediately after pouring.

11.3.2 Save the crucible from the fusion for the slag and