



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 E1601.

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

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

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.

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 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 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 E882.

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-g 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 ± 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 **D1193**.

8.3 *Ammonium Hydroxide* (NH₄OH).

8.4 *Borax Glass* (Na₂B₄O₇).

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* (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 **E50**.

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.

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 scoopfuls of approximately equal size from each segment from the full depth of the bed. Combine the scoopfuls 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 PbO on top of the mixed fire assay clay crucible. Top with 3 g NaCl.

11.2 Fusion:

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC, 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.org>.

TABLE 1 Troy Conversion

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

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 liquefy 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. (**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 cupel correction procedure (see 11.8.1).

TABLE 2 Statistical Information

Material	A		B		C	
Laboratory	Au, oz/ton	Au, µg/g	Au, oz/ton	Au, µg/g	Au, oz/ton	Au, µg/g
1	0.0040	0.14	0.0173	0.59	0.515	17.6
2	0.0063	0.22	0.0167	0.57	0.514	17.6
3	0.0043	0.15	0.0177	0.61	0.513	18.0
4	0.0060	0.21	0.0160	0.55	0.522	17.9
5	0.0040	0.14	0.0193	0.66	0.529	18.1
6	0.0030	0.10	0.0167	0.57	0.524	18.0
7	0.0050	0.17	0.0170	0.58	0.517	17.9
Mean	0.0047	0.16	0.0172	0.59	0.521	17.9
s_r^A	0.0005	0.02	0.0016	0.05	0.004	0.15
r^B	0.0014	0.05	0.0046	0.16	0.012	0.41
s_R^C	0.0013	0.04	0.0017	0.06	0.007	0.23
R^D	0.0035	0.12	0.0048	0.16	0.019	0.64

^A Repeatability standard deviation.

^B Repeatability limit, based on 95 % confidence limit.

^C Reproducibility standard deviation.

^D Reproducibility limit, based on 95 % confidence limit.

11.4 Lead Separation:

11.4.1 After cooling, separate the lead button from the slag by tapping with a steel hammer. Save the slag for correction procedure (see 11.8). Carefully hammer the lead button into a square shape, completely removing any adhering slag from the surface.

11.4.2 Save the slag from the fusion for the slag and cupel correction procedure (see 11.8.1).

11.5 Cupellation:

11.5.1 Place cupels into the furnace and preheat at 900 °C for 30 min.

11.5.2 Quickly place a lead button prepared as described in 11.4.1 onto each cupel. The lead button will melt into a bright lead puddle that will soon form a dark crust with the formation

of surface PbO. The furnace temperature must be greater than 888 °C, the melting point of PbO. This step should be accomplished within 1 to 2 min.

11.5.3 After 5 min, open the draft to the furnace, and decrease the temperature to 820 °C. The ventilation should be regulated. If the temperature and draft conditions are correct, a fringe of PbO crystals, known as “feathers” will appear around the upper edge of the bone ash cupel.

11.5.4 When the process of cupellation is near the end, oily appearing drops of PbO can be seen to collect on the surface of the remaining doré bead. These cause a rainbow color band to move swiftly over the surface of the doré bead giving the illusion that the bead is revolving on a shifting axis. When the last trace of PbO has been removed, this play of colors disappears and the doré bead becomes duller than a normal metallic luster. At this point remove the cupels from the furnace and allow them to cool. (**Warning**—Hot cupels emit lead fumes. Use proper ventilation.)

11.5.5 Remove the doré bead containing the precious metal from the cool cupel with bead pliers or tongs.

11.5.5.1 Save the cupel from the cupellation for the slag and cupel correction procedure (see 11.8.1).

11.5.6 Brush the doré bead with a bead brush to remove any adhering cupel material.

TABLE 3 Bias Information

Reference Material	Certified Au, µ g/g	Average Au, µg/g	Bias, µg/g
Canmet CCU-1a	5.69	5.55	0.14
Canmet CCU-1b	5.89	5.45	0.40

11.6 Parting:

11.6.1 Flatten the doré bead with a hammer, then place it in a 50-mL porcelain crucible.⁴

11.6.2 Add 25 mL HNO₃ (1 + 7) to the crucible with the doré bead. Place the crucible on a hot plate at 95 °C and heat for 30 min. Carefully decant the spent parting solution and dispose of properly.

11.6.3 Repeat the parting and decanting step of 11.6.2 a second time.

11.6.4 Add 25 mL of HNO₃ (3 + 1) to the crucible. Heat for 25 min. Carefully decant the spent parting solution and discard.

11.6.5 Wash the bead twice with NH₄OH (1 + 9) to dissolve any silver chloride (AgCl₂). Discard the liquid. (**Warning**—Stronger NH₄OH solutions left in the porcelain crucible to “soak” the bead can form ammonium nitrates which may explode under certain conditions.)

11.6.6 Dry beads in porcelain cups on a hot plate.

11.6.7 Anneal the remaining gold at 650 °C, then cool. Black or other colored deposits remaining inside the crucible indicate inadequate washing. Off-colored gold indicates the presence of impurities.

11.7 *Weighing*—Weigh the remaining gold bead to the nearest 0.001 mg and record.

11.8 Corrections:

⁴ Bugbee, E. E., “A Textbook of Fire Assaying,” John Wiley & Sons, Inc., Third ed., 1940.

11.8.1 Combine the slag and cupels from the duplicate fusions for each test sample, weigh the total, and record.

11.8.2 Crush the slag-cupel mixture for each test sample until 100 % passes a No. 60 (250- μ m) sieve.

11.8.3 Weigh duplicate 14.583 g $\pm$ 0.001-g ($\frac{1}{2}$ -assay ton) test samples of the mixture from 11.8.2 into the same two fire assay clay crucibles (see 11.3.2) used in the initial fusion for each test sample.

11.8.4 Add approximately 100 g of cupel correction flux (see 8.13) to each crucible, mix and add silver solution (see 8.11) as described in 11.1.3 and 11.1.4. Cap the mixture with 3 g of NaCl.

11.8.5 Carry out the fire assay as described in 11.2-11.4.

11.8.6 Combine the resulting lead buttons and carry through the cupellation, parting, and weighing operations. Follow the procedure in 11.5 and 11.6.

12. Calculation

12.1 Calculate the correction value as follows:

$$F = AD/2 (B+C) \quad (1)$$

where:

- A = total weight of cupel and slag, g,
- B = weight of the first correction sample weight, g,
- C = weight of the second correction sample weight, g,
- D = weight of correction samples' gold bead, mg, and
- F = correction value, mg.

12.2 Calculate the corrected gold concentration in the original sample as follows:

$$H = 1000(E+F)/G \quad (2)$$

where:

- E = weight of gold from step 11.7, mg,
- F = correction value from 12.1, mg,
- G = original sample weight from 11.1, g, and
- H = gold content, μ g/g.

13. Report

13.1 If the difference between duplicate samples is greater than the maximum allowable values given in the following table, then the results are invalid and the gold determination must be repeated.

Gold Bead Weight Range, mg	Maximum Allowable Difference, for Duplicate Bead Weights, mg
0.001 to 0.010	0.001
0.010 to 0.050	0.005
0.050 to 0.100	0.010

13.2 Report results that are less than the lower scope limit of 0.20 μ g/g as approximate values (that is, $\sim$ 0.0, $\sim$ 0.1, or $\sim$ 0.2 μ g/g Au as appropriate).

13.3 Report results that are within the range in accordance with 1.1 as the average of the duplicates rounded to the nearest 0.1 μ g/g Au in accordance with Practice E29, rounding method.

13.4 Conversion to other measurement units can be made by multiplying the result by the appropriate number using Table 1. Troy ounce per ton results are rounded to the nearest 0.001 oz/short ton, in accordance with Practice E29, rounding method.

14. Precision and Bias ⁵

14.1 *Precision*—An interlaboratory study on this test method was conducted with three materials analyzed by seven cooperating laboratories. The interlaboratory test data was evaluated using Practice E691 and is summarized in Table 2.

14.1.1 Repeatability increases with gold content for the three materials tested, in the range from 0.05 to 0.41 μ g/g gold.

14.1.2 Reproducibility also increases with the gold content for the three materials tested, in the range from 0.12 to 0.64 μ g/g gold. The lower scope limit for the method was set at two times R for Material A, at 0.24 μ g/g gold.

14.2 *Bias*—The bias of this test method was determined by analysis of two certified reference materials in a single laboratory on two separate days, and the results are shown in Table 3. The average values determined were slightly lower than the certified values of the reference materials tested, but the differences were within the reproducibility limits of the method determined in the interlaboratory study, thus no significant bias was measured.

15. Keywords

15.1 copper concentrates; fire assay; gold content

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1034.

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