Designation: E1805 – 13

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

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[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.

not pass the laboratory sample through the 150-µm 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 g ± 0.001 g. 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 or perform equivalent flux mixing manually.

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 salt cover.

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 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).

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 min to 2 min.

TABLE 1 Troy Conversion

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

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 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.

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 HNO_3 (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 HNO_3 (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 NH_4OH (1 + 9) to dissolve any silver chloride ($AgCl_2$). Discard the liquid.

WARNING—Stronger NH_4OH 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:*

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

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[4] Bugbee, E. E., "A Textbook of Fire Assaying," John Wiley & Sons, Inc., Third ed., 1940.

11.8.2 Crush and grind the slag-cupel mixture for each test sample until 100 % passes a 250-µm sieve.

11.8.3 Weigh duplicate 14.583 g ± 0.001-g 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 8.2) to each crucible, mix and add silver solution (see 8.10) 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 – 11.7.

12. Calculation

12.1 Calculate the correction value as follows:

$$F = AD/2\left(B+C\right) \tag{1}$$

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:

$$H = 1000(E+F)/G \tag{2}$$

where:

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

TABLE 2 Statistical Information

Material	A	B	C
Laboratory	Au, µg/g	Au, µg/g	Au, µg/g
1	0.14	0.59	17.6
2	0.22	0.57	17.6
3	0.15	0.61	18.0
4	0.21	0.55	17.9
5	0.14	0.66	18.1
6	0.10	0.57	18.0
7	0.17	0.58	17.9
Mean	0.16	0.59	17.9
s_r[A]	0.02	0.05	0.15
r[B]	0.05	0.16	0.41
s_R[C]	0.04	0.06	0.23
R[D]	0.12	0.16	0.64
%R	75	27	3.6

[A] Repeatability standard deviation.
[B] Repeatability limit, based on 95 % confidence limit.
[C] Reproducibility standard deviation.
[D] Reproducibility limit, based on 95 % confidence limit.

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

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 as the average of the duplicates rounded to the nearest 0.1 µg/g Au in accordance with Practice E29, rounding method.

13.3 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 µg/g to 0.41 µg/g gold.

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

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[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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