Designation: E1335 – 08 (Reapproved 2017)

Standard Test Methods for Determination of Gold in Bullion by Fire Assay Cupellation Analysis[1]

This standard is issued under the fixed designation E1335; 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 These test methods cover cupellation analysis of bullion having chemical compositions within the following limits:

Element	Concentration Range, %
Gold	0.5 to 4.0 and 20.0 to 99.8
Silver	1.0 to 99.5
Total gold plus silver	75.0 to 100.0

1.2 These test methods appear in the following order:

	Sections
20.0 % – 99.0 % gold	10 – 17
0.5 % – 4.0 % gold	18 – 23
98.9 % – 99.8 % gold	24 – 30

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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 safety hazards, see Section 8.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B562 Specification for Refined Gold

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

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *anneal*—a thermal treatment to change the properties or grain structure of the product.

3.2.2 *cupel*—a small, shallow, porous cup, usually made of bone ash or from magnesium oxide.

3.2.3 *cupellation*—an oxidizing fusion of lead, sample base metals and gold, and silver in a cupel. The lead is oxidized to litharge (PbO); other base metals which may be present, such as copper and tin, are oxidized as well. The oxidized metals are absorbed into the cupel, leaving a gold and silver doré bead on the cupel surface.

3.2.4 *doré bead*—a gold and silver alloy bead which results from cupellation.

3.2.5 *inquartation*—the addition of silver to an assay sample to enable parting.

3.2.6 *part*—the separation of silver from gold by selectively dissolving the silver in acid, usually nitric acid (HNO_3).

3.2.7 *proof*—a synthetic standard having a composition similar to the test sample.

3.2.8 *proof correction*—analyzing the proof concurrently with the test sample and using the results to correct the final assay.

[1] These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.05 on Cu, Pb, Zn, Cd, Sn, Be, Precious Metals, their Alloys, and Related Metals.

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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] The last approved version of this historical standard is referenced on www.astm.org.

4. Significance and Use

4.1 These test methods are intended for the determination of the gold content of gold and silver bullion. It is assumed that all who use these test methods are trained assayers capable of performing common fire assay procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

5. Interferences

5.1 If the bullion contains any of the following elements in excess of the concentrations shown, the accuracy and precision requirements of these test methods may not be achieved.

Element	Maximum Level, %
Arsenic	2.0
Antimony	2.0
Bismuth	2.0
Iron	2.0
Nickel	2.0
Platinum group, total (Ir, Os, Pd, Pt, Rh, Ru)	0.01
Selenium	2.0
Tellurium	2.0
Thallium	2.0
Tungsten	0.5
Zinc	5.0

6. Apparatus

6.1 *Assay Furnace*—Capable of temperatures up to 1100 °C, accurate to ± 10 °C, with draft controls and fume hood.

6.2 *Hammer.*

6.3 *Hammering Block.*

6.4 *Rolling Mill.*

6.5 *Analytical Balance.*

6.5.1 For Test Methods A and B, capable of weighing to 0.01 mg.

6.5.2 For Test Method C, capable of weighing to 0.002 mg.

6.6 *Parting Basket*—Platinum basket or porcelain gooch crucibles in stainless steel basket/vessel.

6.6.1 *Gooch Porcelain Crucible*—13 mL capacity, bottom inside diameter (ID) 18 mn, top ID 29 mn.

6.6.2 *Stainless Steel Basket*—316 stainless steel.

7. Reagents[4]

7.1 *Copper Metal,* 99.9 % purity, minimum; 0.0005 % gold, maximum.

7.2 *Gold Metal,* 99.99 % purity, minimum.

7.2.1 Gold metal, 99.999 % purity, minimum for Test Method C only.

7.3 *Lead Foil,* 99.99 % purity, minimum (0.001 % silver, maximum; 0.0005 % gold, maximum).

7.4 *Silver Metal,* 99.9 % purity, minimum (0.0005 % gold, maximum).

7.5 *Nitric Acid,* 0.0002 % chloride, maximum.

7.6 *Cupels*—Magnesium oxide or bone ash.

8. Hazards

8.1 For precautions to be observed in the use of certain reagents and equipment in these test methods refer to Practices E50.

8.2 Use care when handling hot crucibles and operating furnaces to avoid personal injury by either burn or electrical shock.

8.3 Lead and litharge (PbO) are toxic materials and are volatile at low temperatures. Avoid inhalation, ingestion, or skin contact.

9. Sampling

9.1 Use shot or pin tube samples. Brush the samples to remove any adhering glass or flux.

9.2 Prepare shot samples from molten metal poured into water. Use only whole single pieces between 1 mm and 3 mm in diameter.

9.3 Pin tube samples are prepared from molten metal drawn into vacuum-evacuated glass tubes. Break the glass and inspect the samples to ensure that they are not hollow and that they are free from slag and inclusions.

9.3.1 Roll the samples lengthwise on a clean rolling mill to 0.127 mm, then clean them with alcohol.

9.3.2 Cut the strip into horizontal slices to obtain the desired sample weight.

9.4 Drillings are not usually as representative of a melt as pin tube or shot samples. If bar drillings are to be analyzed, obtain them as directed in Specification B562.

TEST METHOD A

10. Scope

10.1 This test method covers cupellation analysis of gold in bullion containing 20.0 % to 99.0 % gold and 1.0 % to 80.0 % silver.

10.2 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

11. Summary of Test Method

11.1 A preliminary assay is performed to estimate the approximate gold content and approximate gold plus silver content. Other methods such as X-ray fluorescence (XRF), inductively coupled plasma emission (ICP), direct current plasma emission (DCP), or atomic absorption spectroscopy (AAS) can also be used for a preliminary assay if they have been shown to have an accuracy of better than ± 1 % for gold and ± 2 % for silver. The sample is weighed and silver or copper, or both, added if necessary. The sample is wrapped and compacted in lead foil and cupelled to remove base metals,

[4] *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.

then parted in nitric acid. The insoluble portion is weighed to determine the gold content. Proof standards are used for correction of systematic gravimetric errors.

12. Approximate Assay

12.1 Perform a preliminary assay first on the test sample to establish a suitable composition for the proof correction standard and inquarting silver.

12.2 *Approximate Gold Plus Silver Content*—Weigh one 500 mg ± 2-mg sample to the nearest 0.1 mg. Weigh a portion of lead foil in accordance with the following:

Estimated Total Gold Plus Silver, %	Weight of Lead Foil, g
95.0 – 100.0	5.0
75.0 – 95.0	10.0

12.2.1 Wrap the sample in the lead foil.

12.2.2 *Cupellation*—The cupels are placed in rows in the section of the furnace having the most uniform temperature gradient. After the lead foil packets are prepared, place them in the assay furnace on cupels which have been preheated to 900 °C for 20 min with the draft slightly open. The furnace temperature is correct if the dark crust which forms over the melted lead packet disappears within a few minutes. A typical temperature to produce such reasonably rapid "opening up" of the samples is 900 °C.

12.2.3 After the lead packets have opened up adjust the airflow through the furnace. The temperature must be maintained high enough to prevent the button from freezing (the solidification of molten litharge on the button surface).

12.2.4 Keep the cupels in the furnace until all traces of lead have disappeared. This time depends on the amount of lead used, the furnace temperature, and the airflow (Note 1). Remove the cupels and slowly cool them to room temperature either by placing cupels at the entrance of the furnace with the door open or by placing a warmed metal spatula on top of the cupels.

NOTE 1—Occasionally at the end of the cupellation process, the beads will visibly brighten or "flash." This is a result of the sudden release of the latent heat of fusion as the lead-free bead solidifies.

12.2.5 Remove the test samples and any proof beads from the cupels, place them on edge and tap them lightly with a hammer to loosen any adhering cupel material. Remove the remaining traces of cupel material with a stiff brush.

12.2.6 Weigh the doré bead to the nearest 0.1 mg and calculate the approximate gold plus silver content as follows:

$$T_a = (D/V) \times 100 \quad (1)$$

where:

T_a = approximate total gold plus silver, %,
D = weight of the doré bead, g, and
V = weight of the sample, g.

12.3 *Approximate Gold Content*—Weigh one 500 mg ± 2-mg test sample to the nearest 0.1 mg. Add 1.25 g ± 0.05 g of inquarting silver and 0.05 g ± 0.010 g copper. Wrap the sample with additions in lead foil as directed in 12.2.

12.3.1 Cupel as directed in 12.2.2 – 12.2.4.

12.3.2 Remove the test samples and any proof beads from the cupels, place them on edge and tap them lightly with a hammer to loosen any adhering cupel material. Remove the remaining traces of cupel material with a stiff brush.

12.3.3 *Form Coronet*—Flatten the beads for the gold determination on an anvil with a hammer and taper the edges to facilitate rolling.

12.3.4 Anneal the flattened beads to a temperature of 650 °C to 700 °C. Pass the beads through a rolling mill to form an elongated strip about 10 cm long and 0.015 cm to 0.03 cm in thickness, maintaining a uniform thickness throughout the batch of samples. Reanneal the strips and then roll each into a loose spiral (or coronet) with the bottom side facing outward.

12.3.5 *Parting:*

12.3.5.1 Place each coronet in a suitable parting container (50-mL porcelain crucible, 50-mL Florence flask, or 50-mL Erlenmeyer flask). Add 25 mL of preheated HNO_3 (1 + 2) and heat at just below the boiling point for 45 min. The coronet must remain completely immersed throughout the parting process. Decant and discard the solution. If the coronet breaks apart, care must be taken not to lose any gold pieces.

NOTE 2—Parting baskets should not be used when determining the approximate gold content of multiple samples. If a coronet breaks apart, it will contaminate the other parted samples.

12.3.5.2 Add 25 mL of HNO_3 (2 + 1) and heat at just below the boiling point for 45 min. The coronet must remain completely immersed throughout the parting process. Decant and wash the gold three times with 25 mL of water.

12.3.5.3 Dry the gold on a hotplate, then anneal it to between 650 °C and 700 °C. Cool the gold and weigh to the nearest 0.1 mg.

12.3.5.4 Calculate the approximate gold content as follows:

$$G_a = (C/W) \times 100 \quad (2)$$

where:

G_a = approximate gold, %,
C = weight of gold, g, and
W = weight of sample, g.

12.4 *Approximate Silver Content*—Calculate the approximate silver content as follows:

$$S_a = T_a - G_a \quad (3)$$

where:

S_a = approximate silver, %,
T_a = approximate total gold plus silver, % (12.2.6), and
G_a = approximate gold, % (12.3).

12.5 *Approximate Base Metal Content*— Calculate the approximate base metal content, as follows:

$$M_a = 100 - T_a \quad (4)$$

where:

M_a = approximate base metal content, %, and
T_a = approximate gold plus silver, % (12.2.6).

13. Proof Standard Preparation

13.1 Prepare two proof standards, each containing gold, silver, and copper in the amounts listed as follows. Wrap each proof in lead foil in accordance with 12.2 and proceed to 12.2.2.

13.1.1 *Gold*—The weight of gold must be within ± 5 mg of the approximate gold content (12.3). Weigh the gold to the nearest 0.01 mg and use this weight for calculating the proof correction (15.3).

13.1.2 *Silver*—The weight of inquarting silver is 2.5 times the approximate gold content (12.3). Weigh the silver to the nearest 10 mg.

13.1.3 *Copper*—If the approximate base metal content (12.5) of the sample is less than 1 %, add 0.05 g ± 0.01 g of copper metal to each proof. If the approximate base metal content is greater than 1 %, the amount of copper is equal to the approximate base metal content. Weigh the copper to the nearest 10 mg.

14. Procedure

14.1 *Proof Corrected Assay*—This is the final assay for the gold, incorporating corrections for any material losses.

14.2 *Test Sample Preparation*—Weigh three 500 mg ± 2-mg test samples to the nearest 0.01 mg. Add weighed portions of inquarting silver as follows:

$$S_w = \left[\left(2.5 \times G_a\right) - S_a\right] \times X/100 \quad (5)$$

where:

S_w = weight of silver to be added, g ± 0.05 g,
G_a = approximate gold, % (12.3),
S_a = approximate silver, % (12.4), and
X = sample weight, g (14.2).

If less than 1 % of base metals are present in the unknown, add 0.05 g ± 0.010 g of copper metal to each sample. Weigh three portions of lead foil in accordance with 12.2 and wrap each sample in a portion of the foil.

14.3 Cupel as directed in 12.2.2. Alternate the three test samples with the two proof standards in each row.

14.4 Remove the test samples and any proof beads from the cupels, place them on edge and tap them lightly with a hammer to loosen any adhering cupel material. Remove the remaining traces of cupel material with a stiff brush.

14.5 Form the coronets as directed in 12.3.3.

14.6 Part the test samples and proofs as directed in 12.3.5. For accurate results, the parting conditions for the proofs and samples must be as close as possible. To this end, use of a parting basket or use of individual Bunsen Burners to control the time and temperature of the parting is recommended.

15. Calculation (Proof-Corrected Assay)

15.1 Calculate the average percent gold. For each gold determination sample calculate as follows:

$$G_u = (O/Z) \times 100 \quad (6)$$

where:

G_u = gold, uncorrected, %,
O = weight of gold found, g, and
Z = weight of sample, g.

15.2 Average the three replicates $\bar{G}_u$.

15.3 Calculate the proof correction, P, for each gold determination proof standard as follows:

$$P = Q/O \quad (7)$$

where:

P = proof correction,
Q = weight of gold added, g, and
O = weight of gold found, g.

A successful analysis should have proof corrections falling between 0.99XX and 1.00XX. (See supporting data[5] and ASTM proficiency test program for gold in bullion.)

15.4 Average the two proof standard corrections, $\bar{P}$.

15.5 Calculate the proof-corrected gold percent as follows:

$$G_c = \bar{G}_u \times \bar{P} \quad (8)$$

where G_c = gold, corrected, %.

16. Precision and Bias

16.1 *Precision*—Ten laboratories cooperated in testing Samples 1 through 4 and seven laboratories in Samples 5 and 6.[5] Their data is summarized in Table 1. Testing and statistical analysis were performed in accordance with Practice E173.

16.1.1 *Reproducibility*—The interlaboratory test data show the error of this test method to be constant throughout its range. The best estimate of the reproducibility derived from all six test materials is $R_2 = 0.13$ % gold.

16.2 *Bias*—Proof correction is used to compensate for systematic gravimetric bias in this test method. No information on the absolute bias of this test method is available. Users may verify the accuracy of this procedure by the use of reference materials, if available.

17. Keywords

17.1 fire assay; gold and silver bullion; gold content

TEST METHOD B

18. Scope

18.1 This test method covers the determination of gold in bullion estimated to contain from 0.5 % to 4.0 % gold and 75.0 % to 99.5 % silver.

18.2 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1010.

TABLE 1 Statistical Information—Gold (Test Method A)[A]

Test Material	Gold Found, %	Repeatability, R_1	Reproducibility, R_2
1 (21.5 % Ag)	78.392	0.056	0.193
2 (25.8 % Ag)	73.831	0.073	0.083
3 (23.0 % Ag)	76.484	0.077	0.152
4 (34.0 % Ag)	65.744	0.066	0.123
5 (0.7 % Ag)	99.060	0.053	0.103
6 (67.0 % Ag)	26.350	0.025	0.089

[A] In accordance with Practice E173.

19. Summary of Test Method

19.1 The sample is weighed and cupelled to remove base metals and is then parted in nitric acid. The insoluble part is weighed to determine the gold content. Synthetic test standards prepared from pure metals are tested concurrently to monitor systematic gravimetric errors.

20. Procedure

20.1 *Sample Weighing:*

20.1.1 Weigh three replicates of each test sample to the nearest 0.01 mg and in accordance with the following:

Estimated Gold, %	Sample Weight, g
0.5–2.0	2.98–3.02
2.0–4.0	1.98–2.02

20.1.2 Wrap each replicate in 10 g of lead foil and seal it securely.

20.2 *Test Standard Weighing*—For each batch of test samples assayed, prepare a minimum of two test standards with pure gold and silver, at concentration ratios within the range of the test samples. Weigh these test standards as directed in 20.1.1 to the nearest 0.01 mg and treat them the same as test samples throughout the analysis.

20.3 *Cupellation:*

20.3.1 Place the lead packets containing the test samples and test standards in an assay furnace, onto cupels that have been preheated for at least 20 min at 900 °C. Do not use the row of cupels in the front of the furnace. The furnace temperature is correct if the dark crust which forms over the melted lead packet disappears within a few minutes. A temperature of 900 °C is typical to produce a reasonably rapid "opening up" of the lead packets.

20.3.2 After the lead packets have "opened" (the dark crust has disappeared), adjust the airflow through the furnace. The temperature must be maintained high enough to prevent the button from "freezing" (the solidifying of molten litharge on the button surface).

20.3.3 Keep the cupels in the furnace until all traces of lead have disappeared. A typical cupellation time is 15 min to 20 min. Occasionally at the end of cupellation, the beads will visibly brighten or "flash." This results from the sudden release of the latent heat of fusion as the lead-free bead solidifies. Remove the cupels from the furnace and cover the samples while cooling. Tapping the table onto which the cupels have been placed until they cool may prevent "sprouting" (the formation of a surface growth on the bead due to the expulsion of dissolved oxygen). If a sample sprouts while cooling, repeat the analysis starting at 20.1.

20.4 *Parting:*

20.4.1 Remove the cooled beads from the cupels, and clean them with a stiff brush to remove any adhering material.

20.4.2 Place each bead into a suitable parting container (such as a 50-mL porcelain crucible, Florence flask, or Erlenmeyer flask). Add 20 mL of HNO_3 (1 + 3), and heat slowly to begin dissolving the silver. Control the acid temperature to allow for a slow reaction, which prevents the gold particles from becoming too small. Keep the bead in this solution until all of the silver has dissolved (1 h to 1.5 h). Carefully decant the supernatant solution, ensuring that all the gold particles remain in the container.

20.4.3 Add 25 mL of HNO_3 (2 + 1), and heat to approximately 90 °C for 1 h. Decant the acid and wash the gold three times with 25 mL of 90 °C deionized water.

20.4.4 Transfer the gold under water to an annealing cup and carefully decant the excess water. Dry the cup and gold at 100 °C to remove all residual moisture, then heat the cup and the gold to 650 °C to 700 °C to anneal the gold. Cool the cup with the gold and weigh the gold to the nearest 0.01 mg.

21. Calculation

21.1 Calculate the percent gold as follows:

$$\text{Gold, \%} = 100(A/B) \tag{9}$$

where:

A = weight of gold found, g, and
B = weight of sample, g.

Average the three replicates.

21.2 Calculate the standard ratios for each of the test standards as follows:

$$\text{Standard ratio} = C/D \tag{10}$$

where:

C = weight of gold found, g, and
D = weight of gold at start, g.

A successful analysis should have standard ratios falling between 0.997 and 1.003. If they are outside this range, repeat the analysis starting at 20.1. Do not apply the standard ratios as proof corrections.

22. Precision and Bias

22.1 *Precision*—Seven laboratories cooperated in testing samples identified as SMAAG 1 to 3.[6] Their data are summarized in Table 2. Testing and statistical analysis were performed in accordance with Practice E173.

22.2 *Bias*—No information on the bias of this test method is available. Test standards are used to monitor for systematic gravimetric bias. Users may verify the accuracy of this procedure by the use of reference materials if available.

23. Keywords

23.1 fire assay; gold and silver bullion; gold content

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1011.

TABLE 2 Statistical Information—Gold (Test Method B)[A]

Test Material	Gold Found, %	Repeatability, R_1	Reproducibility, R_2
SMAAG 1	0.511	0.0043	0.0054
SMAAG 2	1.104	0.0057	0.0102
SMAAG 3	3.074	0.0103	0.0248

[A] In accordance with Practice E173.

TEST METHOD C

24. Scope

24.1 This test method covers the cupellation analysis of gold in grade 99.5 refined gold.

24.2 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

25. Summary of Test Method

25.1 The sample is weighed and silver and copper are added. The sample is wrapped in lead foil and cupelled to remove base metals, then parted in HNO_3. The insoluble portion is weighed to determine the gold content. Proof standards are used for correction of systematic gravimetric errors.

26. Preparation of Proof Standard

26.1 Prepare two proof standards, each containing gold, silver and copper in the amounts listed as follows:

26.1.1 *Gold*—Weigh 497.5 mg ± 0.5 mg of gold and use this weight for calculating the proof correction (Section 27).

26.1.2 *Silver*—Weigh 1250 mg ± 10 mg of silver.

26.1.3 *Copper*—Weigh 20 mg ± 2 mg of copper.

26.1.4 Wrap each proof in lead foil in accordance with 12.2 and proceed to 27.3.

26.1.5 Cupel the proof standards together with the test samples (27.2) as directed in 12.2.2.

27. Procedure

27.1 *Proof Corrected Assay*—This is the final assay for the gold, incorporating corrections for any material losses or gains.

27.2 *Test Sample Preparation:*

27.2.1 Weigh three 500 mg ± 2-mg test samples to nearest 0.002 mg. Add 1250 mg of inquarting silver. Add 20 mg of copper metal to each sample. Wrap each sample plus the inquarting silver and copper in a 2.5 gram piece of the foil.

27.3 *Cupellation:*

27.3.1 Cupel as directed in 12.2.2 through 12.2.4. Alternate the test samples with the proof standards as directed in 12.2.2.

27.3.2 Remove test sample and proof beads from the cupels, place them on edge and tap them lightly with a hammer to loosen any adhering cupel material. Remove the remaining traces of cupel material with a stiff brush.

27.4 *Form Coronets:*

27.4.1 Flatten the cleaned beads on an anvil with a hammer, tapering the edges to facilitate rolling.

27.4.2 Anneal to a temperature of 650 °C to 700 °C and pass the beads through a rolling mill to form an elongated strip about 0.1 cm in thickness. Maintain a uniform thickness throughout the batch of samples and proofs. Reanneal the strips and pass the beads through a rolling mill to form an elongated strip about 0.005 cm to 0.01 cm in thickness, maintaining a uniform thickness throughout the batch of samples and proofs. Reanneal the strips and then roll each into a loose spiral (coronet) with the bottom side facing outward. It is important that all rolled strips are rolled to the same length, width and thickness.

27.5 *Parting:*

27.5.1 Add HNO_3 (2 + 1) to a suitable container for the parting basket and heat to boiling (110 °C). Add all the samples and proofs to a suitable parting basket. Maintain the order of coronets in compartments of the platinum basket or in the porcelain gooch crucibles in stainless steel basket. Slowly place parting basket into acid and heat for 45 min or until the evolution of NO_x fumes has ceased. The coronets must remain completely immersed throughout the parting process. Remove the basket and rinse with hot deionized water.

27.5.2 Add HNO_3 (1 + 2) to container for the parting basket and heat to boiling (110 °C). Place parting basket into acid and heat for 45 min. The coronet must remain completely immersed throughout the parting process. Remove the basket and rinse four times with hot deionized water.

27.5.3 Place the basket (still containing coronets) on paper towels to absorb excess water and then on the hot plate and dry. Then anneal platinum basket or individual porcelain gooch crucibles to between 650 °C and 700 °C. Cool the gold and weigh to the nearest 0.002 mg.

28. Calculation

28.1 Calculate the average percent gold as directed in 15.1 to 15.5.

29. Precision and Bias

29.1 *Precision*—Seven laboratories cooperated in testing samples identified as AU1, AU2, and AU3.[7] Their data are summarized in Table 3. Testing and statistical analysis were performed in accordance with Practice E1601.

29.2 *Bias*—Proof correction is used to compensate for systematic gravimetric bias in this test method. No information on the absolute bias of this test method is available. Users may verify the accuracy of this procedure by use of reference materials, if available.

30. Keywords

30.1 fire assay; gold and silver bullion; gold content

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1039.

TABLE 3 Statistical Information—Gold (Test Method C)[A]

Test Material	Gold Found, %	r	R
AU1	98.9896	0.0125	0.0350
AU2	99.4847	0.0115	0.0321
AU3	99.7854	0.0193	0.0541

[A] In accordance with Practice E1601.