



Designation: ~~E1335-04~~^{ε1} Designation: E 1335 - 08

Standard Test Methods for Determination of Gold in Bullion by Fire Assay Cupellation Analysis¹

This standard is issued under the fixed designation E 1335; 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.

~~ε¹Note~~ Editorial changes were made throughout the text in February 2006.

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 99.0
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	10-16-20.0% - 99.0% gold
0.5-4.0% gold	17-21-0.5% - 4.0% gold
98.9-99.8% gold	22-28-98.9% - 99.8% gold

1.3

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.

2. Referenced Documents

2.1 *ASTM Standards*:²

B 562 Specification for Refined Gold

~~E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications~~ astm-e1335-08

E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

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

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals

~~E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory~~

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

~~E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods~~ Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

~~3.1 Definitions:~~

~~3.1.1 annealing~~

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 E01.03 on Precious 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 Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology E 135.

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.

3.1.2

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

3.1.3.2.3 *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.

3.1.4

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

3.1.5

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

3.1.6 *parting*—separating silver from gold by selectively dissolving the silver in acid, usually nitric acid.

3.1.7—*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 (HNO₃).

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

3.1.8

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

3.1.9 For definitions of other terms, refer to Terminology E135.

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

6.2 *Cupels*—Magnesite (MgCO₃) or bone ash.

6.3 *Hammer.*

6.4

6.3 *Hammering Block.*

6.5

6.4 *Rolling Mill.*

6.6

6.5 *Analytical Balance*—Capable of weighing to 0.01 mg.

6.7

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.

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

6.7.2.6.2 *Stainless Steel Basket*—316 stainless steel

6.7.2.1 parting basket (9" X 6" X 1.25")

6.7.2.2 parting vessel (10" X 6.75" X 4")

6.7.2.3 parting vessel lid —316 stainless steel.

7. Reagents ⁴

- 7.1 Copper Metal, 99.9% purity, minimum, 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, min (0.001% silver, maximum), 99.99 % purity, minimum (0.001 % silver, maximum; 0.0005 % gold, maximum).
- 7.4 Silver Metal, 99.9% purity, min (0.001% gold, maximum), 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 E 50.
- 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 (0.005 in.); 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 B 562.

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.

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 $\pm 1\%$ for Ag-gold and $\pm 2\%$ for Ag-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, 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 inquiring 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.

⁴Supporting data are available from ASTM International Headquarters. Request RR:E01-I010.

⁴ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC, <http://www.chemistry.org>. For suggestions on the testing of reagents not listed by the American Chemical Society, see Annual Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., <http://uk.vwr.com>, and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, <http://www.usp.org>.

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 sample doré beads from the cupels and clean any adhering cupel material from them with a stiff brush. (see 14.4.)~~

~~12.2.6~~

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 \pm 2-mg test sample to the nearest 0.1 mg. Add 1.25 g \pm 0.05 g of inquarting silver and 0.05 g \pm 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 0.005-0.015 cm to 0.01 cm-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 HNO₃(1+2) and heat at just below the boiling point for 45 min, or until the evolution of NO_x fumes has ceased. The coronet must remain completely immersed throughout the parting process. Decant and discard the solution.~~ (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 HNO₃ (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:

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