Designation: E 1335 – 96 (Reapproved 2000)ε1

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

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.

ε[1] NOTE—Editorial corrections were made throughout in November 2000.

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
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
10-1620.0–99.0 % gold	
0.5–4.0 % gold	17-21

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 safety hazards, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

B 562 Specification for Refined Gold[2]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[3]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[4]

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[4]

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[5]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[6]

3. Terminology

3.1 *Definitions:*

3.1.1 *annealing*—a thermal treatment to change the properties or grain structure of the product.

3.1.2 *cupel*—a small, shallow, porous cup, usually made of bone ash or magnesite.

3.1.3 *cupellation*—an oxidizing fusion of lead, 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 *doré bead*—a gold and silver alloy bead which results from cupellation.

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

3.1.8 *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 E 135.

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.

[1] These test methods are under the jurisdiction of ASTM Committee E-1 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] *Annual Book of ASTM Standards*, Vol 02.04.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 03.05.

[5] *Annual Book of ASTM Standards*, Vol 03.06.

[6] Supporting data are available from ASTM Headquarters. Request RR:E01-1010.

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.

6.2 *Cupels*—Magnesite ($MgCO_3$) or bone ash.

6.3 *Hammer*.

6.4 *Hammering Block*.

6.5 *Rolling Mill*.

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

7. Reagents

7.1 *Copper Metal*, 99.9 % purity, minimum.

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

7.3 *Lead Foil*, 99.99 % purity, min (0.001 % silver, maximum).

7.4 *Silver Metal*, 99.9 % purity, min (0.001 % gold, maximum).

8. Hazards

8.1 For precautions to be observed in the use of certain reagents and equipment 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 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.), 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 representative of a melt. 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. The sample is weighed and silver or copper, or both, added if necessary. The sample is wrapped 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.

12.2 *Approximate Gold Plus Silver Content*—Weigh one 500 ± 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
90.0–95.0	10.0

12.2.1 Wrap the sample in the lead foil.

12.2.2 *Cupellation*—After the lead foil packets are prepared, place them in the assay furnace on cupels which have been preheated to 900°C for 10 min with the draft slightly open. For proof-corrected assays, alternate samples and the corresponding proofs. 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 cool them to room temperature.

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.

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

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