Designation: E1335 – 24

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	Mass Fraction 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
Test Method A	20.0 % – 99.0 % gold	10 – 16
Test Method B	0.5 % – 4.0 % gold	17 – 21
Test Method C	98.9 % – 99.8 % gold	22 – 28

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, health, and environmental 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

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

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1997)[3]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 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.

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 levels shown, the accuracy and precision requirements of these test methods may not be achieved.

[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 www.astm.org/contact. 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.

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 mm, top ID 29 mm.

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 *Cupels*—Magnesium oxide or bone ash.

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

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

7.4 HNO_3—0.0002 % chloride, maximum.

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

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

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 and Rounding Results

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

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

9.3 Pin tube samples are prepared by drawing molten metal 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 mass.

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.

9.5 Rounding of test results obtained using this Test Method shall be performed in accordance with Practice E29, Rounding Methods, unless an alternative rounding method is specified by the customer or by an applicable material specification.

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 Spectrometry (XRFS), inductively coupled plasma atomic emission spectrometry (ICP-AES), direct current plasma atomic emission spectrometry (DCP-AES), or atomic absorption spectrometry (AAS) can also be used for a preliminary assay if they have been shown to have an accuracy 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, then parted in HNO_3. The insoluble portion is weighed to determine the gold content. Proofs are used for correction of systematic gravimetric errors.

[4] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC, www.acs.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. www.usp.org.