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Standard Practice for Proof Silver Corrections in Metal Bearing Ores, Concentrates, and Related Materials by Fire Assay Gravimetry[1]

This standard is issued under the fixed designation E2294; 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 practice covers the determination of fire assay correction for silver, utilizing proof silver, ores, concentrates, and related metallurgical materials.

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.* (See Test Methods E1335, Practices E50, Guide E882, and ISO Guide 35: 1989.)

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

E1335 Test Methods for Determination of Gold in Bullion by Fire Assay Cupellation Analysis

2.2 *Other Documents:*[3]

ISO Guide 35: 1989 Certification of Reference Materials-General and Statistical Principles

ISO 10378: 1994 Copper Sulfide Concentrates–Determination of Gold and Silver Contents–Fire Assay Gravimetric and Atomic Absorption Spectrometric Method

3. Terminology

3.1 *Definitions*—For definitions of terms used in this Practice, refer to Terminology E135.

4. Summary of Practice

4.1 In the process of fire assay, silver losses occur. Proof silver is carried through the assay fusion and cupellation procedures to determine losses that can provide the fire assay silver correction values (see Test Methods E1335, ISO 10378: 1994, Bugbee,[4] and Smith[5]).

5. Significance and Use

5.1 This practice is primarily intended to be used for the correction of silver loss in the fire assay process. Silver assays are determined by fire assay for the purpose of metallurgical exchange between seller and buyer.

5.2 It is assumed that all who use this practice will be trained analysts capable of performing skillfully and safely. It is expected that work will be performed in a properly equipped laboratory under appropriate quality control practices such as those described in Guide E882.

6. Apparatus

6.1 *Analytical balance*—Capable of weighing to 0.001 mg.

[1] This practice 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.

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

[4] Bugbee, E. E., *A Textbook of Fire Assaying*, Third Ed., John Wiley and Sons, Inc., Hoboken, NJ, 1946.

[5] Smith, E. A.., *The Sampling and Assay of the Precious Metals*, Second Ed., Charles Griffin and Co., Ltd., 1947.

6.2 *Assay furnace*—Capable of temperatures up to 1100 °C, accurate to ± 5 °C.

6.3 *Hammer*—Blacksmith type.

6.4 *Hammering block*—Flat Steel plate.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Borax*—sodium tetraborate—($Na_2B_4O_7$)—technical grade.

7.3 *Cupels*—magnesite ($MgCO_3$) or bone ash.

7.4 *Crucibles*—Standard Fire Assay clay.

7.5 *Flour*—common baking grade.

7.6 *Lead Foil*, 99.99 % purity, min (1 ug/g silver max).

7.7 *Litharge (PbO)*—technical grade—precious metal free.

7.8 *Potassium Carbonate (K_2CO_3)*—technical grade.

7.9 *Silica Sand (SiO_2)*—technical grade.

7.10 *Silver Metal*, 99.99 % purity.

7.11 *Sodium Carbonate (Na_2CO_3)*—technical grade.

8. Hazards

8.1 For precautions to be observed in this practice, refer to Practices E50.

9. Procedure

9.1 Prepare samples according to normal fire assay procedures (Bugbee[4] or Smith[5]).

9.2 Weigh two proof silver samples (99.99 % pure silver foil), to match the typical weight of the expected doré bead. If the weight of the doré bead is unknown, weigh two proof silver samples, approximately 75 mg to 150 mg and 250 mg to 350 mg.

9.3 Fill an assay clay crucible with the same flux used in the test samples. A typical flux is:

Typical Fire Assay Flux

1. 30.5 g of sodium carbonate
2. 14.5 g potassium carbonate
3. 6.5 g silica sand
4. 4 g to 5 g flour
5. 60 g litharge
6. flux cap (15 g litharge and 3 g borax)

9.4 Mix the flux mixture first. For the best and consistent results, transfer the weighed silver samples and lead foil packet on the top of the mixture, then cover with the flux cap mixture.

9.5 Place crucibles in fire assay furnace and proceed with fusion and cupellation steps required for test samples.

9.6 After cupellation, the silver proof is weighed on the microbalance. The weight after the fire assay is compared to the original silver proof weight.

10. Calculation

10.1 Calculate the silver ratio as follows:

$$\text{Silver Ratio} = B/A \quad (1)$$

Where:

A = Initial Weight of Proof Silver, mg, and
B = Final Weight of Proof Silver, mg.

10.2 Round the silver ratio to the nearest 0.0001 in accordance with Practice E29.

10.3 To correct a silver fire assay result for test samples, divide the weight of the silver in the test sample, determined by the difference in weight before and after parting, by the average silver ratio for the two proofs to obtain the corrected silver weight.

11. Keywords

11.1 cupellation; fire assay; silver; silver correction

[6] *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.