Designation: E 2294 – 03

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 E 2294; 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 standard practice covers the determination of fire assay correction for silver, utilizing proof silver, for ores, concentrates and related metallurgical materials.

1.2 *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 Method E 1335, Practices E 50, Guide E 882, and ISO Guide 35).

2. Referenced Documents

2.1 *ASTM Standards:*

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications[2]

E 50 Standard Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[3]

E 135 Standard Terminology relating to Analytical Chemistry for Metals, Ores, and Related Materials[2]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis of Metals[2]

E 1335 Test Methods for Determination of Gold in Bullion by Cupellation[2]

2.2 *Other Documents*

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

Bugbee, Edward, Textbook of Fire Assaying[4]

Smith, E.A., The Sampling and Assay of Precious Metals[5]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this Practice, refer to Terminology E 135.

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 Method E 1335, Method ISO 10378, Bugbee, , Smith).

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 E 882.

6. Apparatus

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

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

[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 .

Current edition approved June 10, 2003. Published July 2003.

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

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

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

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

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

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 Practice E 50.

9. Procedure

9.1 Prepare samples according to normal fire assay procedures, (Bugbee, , or Smith).

9.2 Weigh two proof silver samples (99.99 % pure silver foil), to match the typical weight of the expected dore' bead. If the weight of the dore' bead is unknown, weigh two proof silver samples, approximately 75-150 mg and 250-350 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. 4-5 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 E 29.

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 Silver; silver correction; fire assay; cupellation

[6] *Reagent Chemical, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole Dorset, U. K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc., (USPC), Rockville, MD.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).