Designation: E2296 – 21

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

This standard is issued under the fixed designation E2296; 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 silver loss correction, utilizing slag recycling and cupellation of proof silver during the fire assay of metal bearing ores, concentrates, and related metallurgical materials.

1.2 *Units*—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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* (See Practices E50 and ISO 35:2006.)

1.4 *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]

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

2.2 *ISO Documents:*[3]

ISO 35:2006 Certification of Reference Materials—General and Statistical Principles

ISO 10378:2016 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 fusion, the slag is retained from the initial fusion and reprocessed through the fusion procedure. The resulting lead button is combined with the preliminary lead button during cupellation. Proof silver is carried through the cupellation procedure to determine the silver losses. (See ISO 10378:2016, 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 contents are determined by fire assay for the purpose of metallurgical exchange between buyer and seller. 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.

[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 Nov. 15, 2021. Published December 2021. Originally approved in 2003. Last previous edition approved in 2013 as E2296 – 03(2013). DOI: 10.1520/E2296-21.

[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), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

[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.

TABLE 1 Slag Re-Fusion Flux

litharge	55.4 g
sodium carbonate	9.3 g
silica sand	9.4 g
potassium bitartrate	7.3 g
borax	18.7 g

6. Apparatus

6.1 *Assay Furnace,* capable of temperatures up to 1100 °C, accurate to ± 5 °C with draft controls.

6.2 *Analytical Balance,* capable of determining mass to 0.001 mg.

6.3 *Hammer,* blacksmith type.

6.4 *Hammering Block,* flat steel or iron.

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 *Crucibles,* standard fire assay clay.

7.4 *Cupels,* magnesite ($MgCO_3$) or bone ash, size determined by lead button mass.

NOTE 1—Cupel size is determined by the mass of the lead button. Cupels must be large enough to hold all the molten lead from the primary fire assay fusion and slag re-fusion.

7.5 *Lead Foil* (99.99 % purity min; 1 µg/g silver max).

7.6 *Litharge* (PbO), technical grade, precious metal free.

7.7 *Potassium Bitartrate* ($K_2C_4H_4O_6$), technical grade.

7.8 *Silica Sand* (SiO_2), technical grade.

7.9 *Silver Metal* (99.99 % purity), foil.

7.10 *Sodium Carbonate* (Na_2CO_3), technical grade.

7.11 *Wax Paper Flux Bag,* 25 cm^2 by 25 cm^2.

8. Hazards

8.1 For precautions to be observed in this method, refer to Practice E50.

9. Procedure

9.1 Perform the initial fire assay according to proper accepted procedures to produce the initial lead button. (See Bugbee[4] and Smith[5].)

9.2 Collect the fire assay fusion slag from the original fusion and place back into the original fire assay crucible.

9.3 Add 50 g of the slag re-fusion flux to the crucible. The composition is shown in Table 1.

9.4 Place crucible in the fire assay furnace. Raise the temperature to 1060 °C. After the furnace obtains that temperature, hold for at least 30 min. In most instances, the total furnace time will be approximately 1 h. Fusion must be in a liquid state.

NOTE 2—In most cases, the best way to add re-fusion flux is with a wax paper bag. Place the re-fusion flux into the bag, twist the top, and place on top of the original fusion slag material in the original fire assay crucible.

9.5 After 1 h, carefully pour the melt into fire assay moulds, taking care to ensure that no portion is lost.

9.6 After cooling, break off slag and retain lead button.

9.7 Place the re-fusion lead button with the original fusion lead button for each test sample.

9.8 Prepare two proof silver foil samples to match the typical mass of the expected doré bead and record the mass. If the mass of the doré bead is unknown, prepare two proof silver samples approximately 75 mg to 100 mg and 250 mg to 350 mg.

9.9 Wrap the proof silver beads in lead foil, and pound into a lead square with a hammer.

NOTE 3—The mass of the lead foil should approximate the mass of the combined lead buttons collected in 9.1 and 9.6.

9.10 Transfer the lead square (9.9) and the combined lead buttons (9.7) into the cupellation furnace into three cupels with the silver proofs on two sides of the combined test sample beads.

9.11 Cupel to obtain doré beads by normal fire assay cupellation procedures.

9.12 After cupellation, determine the mass of the silver proofs on the balance and record the mass.

NOTE 4—The loss of silver due to the original fire assay fusion technique is recovered by the slag recycling and re-fusion steps.

10. Calculation

10.1 Calculate the silver ratio as follows:

$$\text{Silver Ratio} = \frac{B}{A} \tag{1}$$

where:

A = initial mass of proof silver, mg, and
B = final mass of proof silver, mg.

10.2 Round the silver ratio to the nearest 0.0001 mg.

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

10.4 Rounding of test results obtained using this test method shall be performed as directed in Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification.

[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.