Designation: E2295 – 21

Standard Practice for Fire Assay Silver Corrections in Analysis of Metal Bearing Ores, Concentrates, and Related Metallurgical Materials by Silver Determination in Slags and Cupels[1]

This standard is issued under the fixed designation E2295; 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 silver corrections for fire assay of metal bearing ores, concentrates, and related metallurgical materials using the spent slags and cupels from the fire assay process, by gravimetry and atomic absorption spectrophotometry.

1.2 The test methods appear in the following order:

	Sections
Gravimetric Method	10–11
Atomic Absorption Method	12–13

1.3 *Units*—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.* (See Practices E50 and ISO Guide 35:1989.)

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]

D1193 Specification for Reagent Water

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

E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry (Withdrawn 2004)[3]

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

2.2 *ISO Documents:*[4]

ISO Guide 35:1989 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 slags and cupels are collected, retreated and silver is determined in them to provide a correction value for the fire assay determination of silver (see Guide E1024, Test Methods E1335, ISO 10378:2016, Bugbee,[5] and Smith[6]).

[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] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

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

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

5. Significance and Use

5.1 These methods are primarily intended to be used for the determination of silver correction in the fire assay silver determination. 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 method 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 0.01 g.

6.2 *Analytical Balance,* capable of weighing to 0.001 mg.

6.3 *Assay Furnace,* capable of temperatures up to 1100 °C, accurate to ± 5 °C.

6.4 *Atomic Absorption Spectrophotometry,* AAS.

6.5 *Ring Grinder,* 250 g capacity.

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.[7] 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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or II of Specification D1193. Type III or IV may be used if they effect no measurable change in the blank or sample.

7.3 *Borax,* sodium tetraborate ($Na_2B_4O_7$), technical grade.

7.4 *Ammonium Chloride Solution* (NH_4Cl 250g/L)—Add 250 g of ammonium chloride to 500 mL of water in a 1 L volumetric flask. Dilute to the mark and mix.

7.5 *Crucibles,* standard fire assay.

7.6 *Cupels,* magnesite ($MgCO_3$) or bone ash.

7.7 *Flour,* common baking grade.

7.8 *Litharge* (PbO), technical grade, silver free.

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

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

8. Hazards

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

8.2 All precautions and safe laboratory operating procedures should be followed when using $HClO_4$.

9. Sampling and Sample Preparation

9.1 Determine the mass of the fire assay slags and cupels (7.6) from the duplicate fusion and cupellation processes for each test sample on a balance to 0.01 g. Record mass.

9.2 Place the determined slags and cupels (7.6) into a ring grinder and pulverize for about 20 s. This should reduce the material to pass a 150 µm sieve. This is the retreatment sample that corresponds to the duplicate test sample.

NOTE 1—Longer grinding may cause caking of the ground material. Clean the ring grinder by grinding silica sand between each retreatment sample.

GRAVIMETRIC SILVER CORRECTION METHOD

10. Procedure

10.1 To the duplicate crucibles saved from the fire assay fusion of each test sample, add the following flux.

Crucible Fire Assay Flux

1. Litharge—50 g (7.8)
2. Sodium Carbonate—50 g (7.10)
3. Silica—50 g (7.9)
4. Borax—50 g (7.3)
5. Flour—Usually 4 g add or subtract to produce an approximately 30 g lead fire assay button (7.7)

10.2 Determine the mass of two portions of the retreatment sample into the pre-fluxed crucibles and record the masses.

Sample A = 14.583 g or ½ AT
Sample B = 29.167 g or 1 AT

NOTE 2—AT = Assay Ton, a fire assay mass system.

10.3 Mix retreatment samples and flux together in the crucibles.

10.4 Carry out the normal fire assay fusion and pour into assay molds. Separate the slag from the lead button (see Bugbee[5] and Smith[6]).

10.5 Place the lead button from the retreatment fusion into a new preheated cupel at 900 °C.

10.6 Cupel to finish (a lead free doré bead should be formed).

10.7 Discard the retreatment samples and crucibles when analysis and correction is completed.

NOTE 3—These materials contain lead wastes; dispose of properly.

10.8 Determine the mass of the duplicate retreatment doré beads to the nearest 0.001 mg and record the masses.

11. Calculation

11.1 Calculate the doré correction for each fusion as follows:

$$\text{Doré Correction, mg} = \frac{AB}{C} \quad (1)$$

where:

A = total slags and cupels mass, g,
B = doré bead mass from retreatment fusion, mg, and
C = mass of retreatment sample used in the fusion, g.

[7] *Reagent Chemicals, 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.