Designation: E2295 – 13

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 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 and health practices and determine the applicability of regulatory limitations prior to use.* (See Practices E50 and ISO Guide 35:1989.)

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E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry (Withdrawn 2004)[3]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 10378:1994 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 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 10378:1994, Bugbee,[5] and Smith[6]).

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.

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[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), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

[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.

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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 Weigh the fire assay slags and cupels (7.6) from the duplicate fusion and cuppellation processes for each test sample on a balance to 0.01 g. Record weight.

9.2 Place the weighed 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 Weigh 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 Weigh 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.

11.2 Round the doré correction to the nearest 0.001 mg in accordance with Practice E29.

11.3 To perform the doré correction on the original fire assay, add the average of the two doré corrections to the individual uncorrected doré masses for the fire assays of the

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[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 the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

test sample. The combined mass is then the corrected doré mass for final calculation of the gold plus silver in the test sample.

11.4 The gold must be determined in all doré beads and subtracted from each doré mass to obtain the silver mass in that doré bead.

NOTE 4—Gold can also be determined by first weighing the doré bead, parting the doré bead with nitric acid according to standard fire assay procedure, and weighing the resulting gold bead (see Test Methods E1335). The silver mass is determined by the difference between the doré bead and gold bead masses.

ATOMIC ABSORPTION SILVER CORRECTION METHOD

12. Procedure

12.1 Transfer duplicate 2.00 g portions of the retreatment sample (9.2) into 400-mL beakers and record the masses. Carry a blank beaker throughout the digestion procedures.

12.2 Add 5 mL of HCl and 15 mL of $HClO_4$ to the retreatment sample. Heat to $HClO_4$ fumes on a hot plate. (**Warning—**An $HClO_4$ fume hood should be used for all $HClO_4$ digestions.)

12.3 Cool. When cool to touch, add 40 mL HCl and 5 mL of NH_4Cl (7.4) solution.

12.4 Mix solution, then place into a 400-mL volumetric flask.

12.5 Dilute to volume with water. Mix. This is the analysis solution.

12.6 Set the atomic absorption spectrophotometer at 328.1 nm with a 10 cm path length burner head. Background correction must be used, (see Guide E1024 and ISO 10378:1994). Perform three measurements on calibration solutions, that would match the low, middle, and high values of expected measurements in μg/mL silver. Calculate, to three significant figures the mean, absorbance for each calibration solution, provided the precision of values does not exceed 10 % relative standard deviation (RSD). If this precision is exceeded, repeat the calibration. Prepare a calibration curve.

12.7 Read the blank and duplicate retreatment test solutions in reference to the calibration curve (12.6) and record the results μg/mL silver.

NOTE 5—During AAS determinations, the retreatment and calibration solutions should have the same temperatures as well as the same acid concentrations.

NOTE 6—The method of bracketing test solutions by calibration solutions to improve precision is recommended.

NOTE 7—Alternatively, an ICP atomic emission spectrometer can be used for the determination of silver at: 328.1 nm.

13. Calculations

13.1 Calculate the silver content of the retreatment sample as follows:

$$D = (C - B)400/W \qquad (2)$$

where:

D = concentration of silver in the retreatment sample, μg/g,
C = concentration of silver in the retreatment analysis solution, μg/mL,
B = concentration of silver in the blank solution, μg/mL,
400 = volume of the retreatment analysis solution, mL, and
W = mass of the retreatment sample, g.

13.2 Average the results for the duplicate retreatment samples.

13.3 Calculate the silver correction mass as follows:

$$\text{Silver Correction Mass, mg} = \frac{AD}{1000} \qquad (3)$$

where:

A = total slag and cupel mass, g, and
D = concentration of silver in the retreatment sample, μg/g.

13.4 Round the silver correction mass to the nearest 0.001 mg and record.

13.5 The silver correction mass is added to the milligrams of silver measured in the original fire assay silver determination to obtain the corrected silver fire assay.

14. Keywords

14.1 cupellation; fire assay; silver; silver correction

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