



Designation: E 400 – 02

Standard Test Method for Analysis of Ores, Minerals, and Rocks by Fire Assay Preconcentration Atomic Emission Spectrometry¹

This standard is issued under the fixed designation E 400; 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 test method covers the spectrographic analysis of ores, minerals, and rocks for silver, palladium, platinum, gold and rhodium. The concentrations of precious metals which can be determined in the material being analyzed depend on the amount of sample assayed (Note 1). Concentration ranges for the lead fire assay beads are as follows:

Element	Concentration Range, %
Silver	0.028 to 1.40
Palladium	0.004 to 0.14
Platinum	0.004 to 0.14
Gold	0.003 to 0.14
Rhodium	0.004 to 0.07

NOTE 1—The amounts used are large enough to minimize weighing errors. A wide range of precious metal concentrations in rocks, minerals and ores can be covered by a modest range of percentages in the lead beads by regulating the weights of the initial sample and the lead bead. Also, both gold and silver can be determined in the lead bead. When either of these metals is used as a collector for the others in the assay, as is generally done, it cannot be determined without another assay.

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. Specific hazard statements are given in Section 9.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals³

E 115 Practice for Photographic Processing in Optical

Emission Spectrographic Analysis³

E 116 Practice for Photographic Photometry in Spectrochemical Analysis³

E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes³

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials³

E 172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis³

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory⁴

E 356 Practices for Describing and Specifying the Spectrograph⁴

3. Terminology

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

4. Summary of Test Method

4.1 The general procedure has been described in the literature (1-3).⁵ A sample of ore, mineral, or rock is fire assayed according to conventional practice (4-7), but the cupellation process is arrested before completion to provide a lead bead, weighing 50 to 100 mg, which contains the precious metals from the original sample. The lead bead is mounted on a copper rod which is made the lower electrode for an oscillatory self-initiating capacitor discharge as described in Practice E 172. The spectra are photographed on a calibrated emulsion, and intensity ratios of selected pairs of analytical lines and internal standard (lead) lines are determined photometrically. Concentrations of the elements are read from analytical curves relating log intensity ratio to concentration.

5. Significance and Use

5.1 This test method is a comparative method intended primarily to test materials for compliance with compositional specifications or monitoring for metal content. It is assumed that use this method will be trained analysts capable of

¹ This test method 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 *Annual Book of ASTM Standards*, Vol. 11.01.

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

⁴ *Annual Book of ASTM Standards*, Vol 03.06.

⁵ The boldface numbers in parentheses refer to the list of references at the end of this method.

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. Interferences

6.1 The elements ordinarily present do not interfere with this test method.

7. Apparatus

7.1 Sample Preparation Equipment:

7.1.1 *Silica Dishes*, 100 mm in diameter by 15 mm deep.

7.1.2 *Assay Furnace*,⁶ pyrometrically controlled, with provision for drawing air through the heating chamber, and capable of attaining 1200°C.

7.1.3 *Fire-Clay Crucibles*, 15-g and 30-g sizes.

NOTE 2—The crucible size, that is 15-g or 30-g capacity, indicates a crucible of sufficient volume to hold 15 g or 30 g of ore with the necessary flux.

7.1.4 *Asbestos Gloves or Mitts*.

7.1.5 *Face Shield or Safety Glasses*.

7.1.6 *Conical Iron Molds*.

7.1.7 *Cupels*, bone ash or magnesia, 50-g size.

7.1.8 *Graphite Crucibles*:

7.1.8.1 *Large*, 20 mm in inside diameter by 20 mm deep.

7.1.8.2 *Medium*, 12 mm in inside diameter by 5 mm deep.

7.1.8.3 *Small*, 5 mm in inside diameter by 5 mm deep.

7.1.9 *High-Silica Glass Crucibles*,⁷ 30 mL, equipped with Rose covers and inlet tubes.

7.1.10 *High-Silica Glass Tube*,⁷ 30 mm in diameter.

7.1.11 *Combustion Boat*, 110 by 20 mm by 10 mm deep.

7.1.12 *Steel Block*, suitable for clamping in a vise and having a 4.91-mm diameter hole drilled 20 mm deep in one side.

7.1.13 *Center Punch*, with sharp, pointed tip.

7.2 *Excitation Source*, providing a high-voltage spark discharge with the source parameters described in 13.1.

7.3 *Spectrograph*, having sufficient resolving power and linear dispersion to separate clearly the analytical lines from other lines in the spectrum of samples in the range from 2350 to 3400 Å. An instrument having a reciprocal linear dispersion of 5 Å/mm satisfies these conditions. (See Practices E 356.)

7.4 *Photographic Processing Equipment*, providing developing, fixing, washing, and drying operations and conforming to the requirements of Practice E 115.

7.5 *Microphotometer*, having a precision of $\pm 1.0\%$ for transmittance values between 10 and 90 %.

7.6 *Calculating Equipment*—A calculating board with emulsion calibration scales may be used to convert microphotometer readings to log intensity ratios and concentrations.

8. Reagents and Materials

8.1 *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 were such specifications are available.⁸ 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification D 1193.

8.3 *Calcium Fluoride* (fluorspar), native powder.

8.4 *Copper Rods, High-Purity*, 4.76 mm in diameter and 380 mm long.

8.5 *Flour*.

8.6 *Graphite Rods, High-Purity*, 6.15 mm in diameter.

8.7 *Hydrogen*.

8.8 *Lead*, granular, high-purity, free of gold and silver.

8.9 *Lead Monoxide* (litharge), assayer's grade.

8.10 *Metals*—The following high-purity metals shall be used: gold, palladium, platinum, rhodium, and silver (8).

8.11 *Nitrogen*.

8.12 *Silica Sand*, 80 (180- μ m) to 120 mesh (125- μ m).

8.13 *Sodium Borate* (borax glass), fused and ground.

8.14 *Sodium Carbonate* (soda ash), anhydrous.

8.15 *Photographic Emulsion*.⁹

8.16 *Photographic Processing Solutions*—The formulas for processing solutions are given in Practice E 115.

9. Hazards

9.1 For precautions to be observed in this method, refer to Practice E 50.

9.2 Safety precautions as practiced by fire assayers shall be observed when handling hydrogen or pouring molten lead.

10. Sampling and Sample Preparation

10.1 Crush and pulverize the sample to between 150 (106- μ m) and 200 mesh (75- μ m). Place 29.166 g (1 assay ton) of sample in a silica dish and heat in the assay furnace under oxidizing conditions for 1 h at 700°C to convert sulfide or arsenide minerals to oxides. Allow the sample to cool, then mix it intimately with a flux of the following composition (Note 3):

Sodium borate	30 g
Lead monoxide	40 g
Flour	5.5 g
Calcium fluoride	10 g
Silica	30 to 40 g
Sodium carbonate	40 g

NOTE 3—During fusion, the litharge is reduced by carbon from the flour so that droplets of lead form, collecting the precious metals and sinking to the bottom of the crucible. Beamish states that "the technique of a fire assay collection of the noble metals is largely an empirical process assisted to a degree by some fundamental principles" (5). The method detailed here has been found satisfactory for many rocks, ores, and

⁸ *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.

⁹ Kodak Spectrum Analysis No. 1 film or plate, or equivalent, has been found suitable.

⁶ A Lindberg Assay Furnace has been found suitable.

⁷ VYCOR Brand Glass, made by Corning Glass Works, Corning, NY, has been found suitable.