



Designation: E 400 – 97

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

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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 7.

2. Referenced Documents

2.1 ASTM Standards:

- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²
- E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis²
- E 116 Practice for Photographic Photometry in Spectrochemical Analysis²
- E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes²

¹ This test method is under the jurisdiction of ASTM Committee E-1 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 03.05.

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials²

E 172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis²

E 356 Practices for Describing and Specifying the Spectrograph³

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).⁴ 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. Apparatus

5.1 Sample Preparation Equipment:

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

5.1.2 *Assay Furnace*,⁵ pyrometrically controlled, with provision for drawing air through the heating chamber, and capable of attaining 1200°C.

5.1.3 *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.

5.1.4 *Asbestos Gloves or Mitts*.

5.1.5 *Face Shield or Safety Glasses*.

5.1.6 *Conical Iron Molds*.

³ *Annual Book of ASTM Standards*, Vol 03.06.

⁴ The boldface numbers in parentheses refer to the list of references at the end of this method.

⁵ A Lindberg Assay Furnace has been found suitable.

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

5.1.8 *Graphite Crucibles*:

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

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

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

5.1.9 *High-Silica Glass Crucibles*,⁶ 30 mL, equipped with Rose covers and inlet tubes.

5.1.10 *High-Silica Glass Tube*,⁶ 30 mm in diameter.

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

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

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

5.2 *Excitation Source*, providing a high-voltage spark discharge with the source parameters described in 11.1.

5.3 *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.)

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

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

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

6. Reagents and Materials

6.1 *Purity and Concentration of Reagents*—The purity and concentration of the chemical reagents shall conform to the requirements prescribed in Practices E 50.

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

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

6.4 *Flour*.

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

6.6 *Hydrogen*.

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

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

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

6.10 *Nitrogen*.

6.11 *Silica Sand*, 80 to 120 mesh.

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

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

6.14 *Photographic Emulsion*.⁷

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

7. Hazards

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

⁶ VYCOR Brand Glass, made by Corning Glass Works, Corning, NY, has been found suitable.

⁷ Kodak Spectrum Analysis No. 1 film or plate, or equivalent, has been found suitable.

8. Preparation of Standards

8.1 *Master Standards*—Prepare a series of master standards as shown in Table 1. Weigh and transfer the lead into a large graphite crucible together with the required amounts of the other metals (Note 2). Place the graphite crucible inside a 30-mL high-silica glass crucible.⁶ Arrange the Rose crucible cover and inlet tube such that a gentle stream of hydrogen is directed at the melt. Light the hydrogen (Note 3). Heat over a Meker burner until the melt appears clean and shiny; the platinum metals will appear as floating specks until they have dissolved completely. Allow the melt to cool to room temperature under a hydrogen atmosphere. Assign a small triangular file to each standard; keep each file in a clean, labeled container. File fragments from each master standard for dilution.

NOTE 3—A Rose tube must be used; a larger bore tube may allow the hydrogen to explode.

8.2 Working Standards:

8.2.1 Prepare working standards by mixing and diluting the master standards with lead to obtain the desired concentrations as shown in Table 2. Concentrations of precious metals in the set of working standards are shown in Table 3.

8.2.2 Melt the working standards in the same manner as the master standards. Use the medium graphite crucibles and melt several standards simultaneously by use of a large high-silica glass tube mounted horizontally over a Meker burner fitted

TABLE 1 Master Standards

Standard	Element	Weight, mg	Percent Element in Standard	Weight of Standard, g
A	Platinum	35.0	3.50	1.0000
	Palladium	35.0	3.50	
	Rhodium	17.5	1.75	
	Lead	912.5		
B	Gold	35.0	3.50	1.0000
	Lead	965.0		
C	Silver	140.0	14.00	1.0000
	Lead	860.0		
D	Platinum	14.0	0.70	2.0000
	Palladium	14.0	0.70	
	Rhodium	10.0	0.50	
	Lead	1962.0		
E	Platinum	20.0	1.00	2.0000
	Palladium	20.0	1.00	
	Rhodium	20.0	1.00	
	Gold	15.0	0.75	
	Lead	1925.0		
F	Gold	10.0	1.00	1.0000
	Lead	990.0		
G	Silver	20.0	1.00	2.0000
	Lead	1980.0		
H	Platinum	7.0	0.35	2.0000
	Palladium	7.0	0.35	
	Rhodium	7.0	0.35	
	Gold	7.0	0.35	
	Silver	70.0	3.50	
	Lead	1902.0		