ASTM **Designation: E 400 – 97**

Standard Test Method for
Analysis of Ores, Minerals, and Rocks by Fire Assay Preconcentration Optical Emission Spectrometry[1]

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 7.

2. Referenced Documents

2.1 *ASTM Standards:*
E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[2]
E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis[2]
E 116 Practice for Photographic Photometry in Spectrochemical Analysis[2]
E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes[2]

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[2]
E 172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis[2]
E 356 Practices for Describing and Specifying the Spectrograph[3]

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.*

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[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.
Currrent edition approved October 10, 1997. Published June 1998. Originally published as E 400 – 92. Last previous edition E 400 – 92a (1997)ε1.
[2] *Annual Book of ASTM Standards*, Vol 03.05.

[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 ±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.

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[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		

TABLE 2 Working Standards

Working Standard	Master Standard	Weight of Master Standard, mg	Final Weight of Working Standard, g
I	A	100.0	2.5000
	B	100.0	
	C	250.0	
II	A	50.0	2.5000
	B	50.0	
	C	100.00	
III	A	25.00	2.5000
	B	25.00	
	C	60.00	
IV	A	12.25	2.5000
	B	12.25	
	C	30.00	
V	D	25.00	2.5000
	F	20.00	
	C	15.00	
VI	E	10.00	2.5000
	G	75.00	
VII	H	20.00	2.5000

TABLE 3 Concentration of Precious Metals in Working Standards

Standard	Platinum, %	Palladium, %	Rhodium, %	Gold, %	Silver, %
I	0.140	0.140	0.070	0.140	1.40
II	0.070	0.070	0.035	0.070	0.560
III	0.035	0.035	0.018	0.035	0.340
IV	0.017	0.017	0.009	0.017	0.170
V	0.007	0.007	0.005	0.008	0.084
VI	0.004	0.004	0.004	0.003	0.030
VII	0.0028	0.0028	0.0028	0.0028	0.028

with a wing-tip.[6] Place several graphite crucibles in a combustion boat. Place the boat in the tube and flush with hydrogen *while the tube is cold*. Burn the hydrogen at the end of a Rose tube. Maintain a flow of hydrogen during melting and cooling to room temperature. Continue heating the standards for 20 min after the lead becomes molten. When cool, file fragments from each standard and remelt portions weighing approximately 50 mg in the small graphite crucible by the same procedure. Mount these 50-mg lead beads as described in 9.5. Prepare as many as required and store in labeled capsules or vials.

9. Preparation of Samples

9.1 Crush and pulverize the sample to between 150 and 200 mesh. 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 4):

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 4—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 minerals, but it must be emphasized that there is no universal method; the flux must be adjusted to suit the type of sample to be assayed. Such adjustments must usually be based on the judgment and experience of the assayer.

Experience, or trial and error, will give some indication of the sample grade. Thirty to sixty g of sample (1 or 2 assay tons) are usually sufficient for normal low-grade ores and similar materials. If the grade is suspected to be high, the amounts of sample and flux should be reduced accordingly. For rocks and very low-grade materials, it may be necessary to combine the lead buttons from several assays to obtain concentrations adequate for spectrochemical detection.

If the amount of nickel or copper is significant (for example, more than 4 g of nickel, 7.5 g of copper, or 3 g of nickel and copper together in a 29.166-g sample), small amounts of precious metals may be carried by them into the slag. It is necessary therefore to leach the oxidized sample with an acid (generally hydrochloric) to remove the base metals. The dissolved precious metals may be recovered by adding 1 g of copper as the chloride (if copper is not present in the sample) and co-precipitating the precious metals with copper by addition of zinc dust. The precipitated copper and the original residue are filtered, ignited, and mixed with a flux for crucible fusion.

9.2 Place the mixture in a 30-g fire-clay crucible and heat it for 1 h in the fire assay furnace at 1150°C. Pour the molten mixture into a conical iron mold; use tongs and wear a face shield or safety glasses. Allow the mixture to solidify and break the lead button away from the slag at the apex of the cone.

9.3 The lead button should weigh 25 to 35 g and should be "bright, soft and malleable" (**5**) (Note 5). If it is not clean, as is often the case, purification is required. Fuse a mixture of 60 g of lead monoxide and 6 g of silica in a 15-g fire-clay crucible for 15 min at approximately 900°C. Drop the lead button into the melt, maintaining the temperature at 900°C, and allow the melt to stand for 5 min. Pour the molten mixture into a conical iron mold, allow it to solidify, and break the lead button away from the slag (**9**).

NOTE 5—If the lead button is not within the 25 to 35-g range, the amount of flour may be increased for a heavier lead button and vice versa; however, other constituents in the sample may also influence the lead button weight.

9.4 Preheat cupels at 1000°C for at least 30 min. Place the lead button on a heated cupel, using cupel tongs. Close the furnace door until the button is completely molten. Open the furnace door slightly and open the draft to supply air for oxidation of the lead (Note 6). Allow this cupellation to proceed until the lead bead remaining on the cupel appears to be 4 to 5 mm in diameter. Move the cupel near the furnace door. When the bead diameter is further reduced to 2 to 3 mm, remove the cupel from the furnace and place it under a gentle stream of nitrogen. The lead bead will freeze at a weight of approximately 50 to 100 mg (Note 7).

NOTE 6—The resulting lead oxide is absorbed by the cupel and partially volatilized as it is formed.

NOTE 7—It is not difficult to arrest a cupellation in the manner described. Some practice is advisable to acquire the technique but this may be obtained by observing the bead formed by melting 50 to 100 mg of lead on a cupel at temperature of 950 to 1000°C. Variable amounts of lead do not affect accuracy within reasonable limits. Satisfactory results can be obtained from beads weighing as little as 25 or 30 mg. However,

lead can be added (as in 8.2) if the cupellation is inadvertently carried too far.

9.5 Clamp the steel block (5.1.12) securely in the vise. Clean the copper rods (6.3) by dipping in dilute nitric acid. File or machine one end flat, perpendicular to the axis of the rod. Place a rod in the steel block. Fold aluminum foil into a shallow tray and make a small hole in the center; place this tray over the rod in such a manner that it will prevent loss of the bead should it roll off the rod during mounting. Make four holes, roughly 0.8 mm deep and evenly spaced, in the top of the rod with the center punch. Place the lead bead on the rod with forceps and center it. Hold a folded aluminum foil strip over the bead and flatten it with a small ball peen hammer. The end of the rod should be uniformly covered with lead and some lead forced into the holes to secure the bead firmly to the rod. Form the lead into a circular sheet just covering the rod, but not extending beyond the rim.

10. Preparation of Apparatus

10.1 *Electrode System*—Place the copper rod carrying the lead bead in the lower electrode clamp and make it the cathode. Use a graphite upper electrode (anode), Type C-5, as described in Practice E 130.

11. Excitation and Exposure

11.1 Produce and record the spectra in accordance with the following conditions:

11.1.1 *Electrical Parameters*:

Capacitance, µF	0.008
Inductance, µH	360
Resistance, Ω	residual
Peak secondary potential, V	20,000
R-f current, A	5
Discharges/s	240

11.1.2 *Exposure Conditions*:

Spectral region, Å	2100 to 3900
Slit width, µm	50
Slit length, mm	2
Preburn period, s	0
Exposure period, s	20
Filters	as noted in Table 4

11.1.3 *Exposure Index*—Maintain the transmittance of Pb 3118.92 Å at approximately 30 %.

11.1.4 *Replicate Exposures*—Make triplicate exposures for standards and duplicate exposures for samples.

12. Photographic Processing

12.1 Process the emulsion in accordance with Practice E 115.

13. Photometry

13.1 Measure the transmittance of the analytical and internal standard line pairs shown in Table 4 using a microphotometer. Correct the measurements for background in accordance with Practices E 116.

14. Calibration

14.1 *Emulsion Calibration*—Calibrate the emulsion in accordance with Practice E 116.

TABLE 4 Line Pairs and Analytical Ranges

Element	Analytical Line, Å	Internal Standard Line, Å	Concentration Range, %
Silver	Ag 2437.79 Ag 2357.92[A]	Pb 3118.92	0.025 to 1.50
Palladium	Pd 3242.70 Pd 3373.00 Pd 3027.92	Pb 3118.92	0.0025 to 0.150
Platinum	Pt 3064.71 Pt 3042.63	Pb 3118.92	0.0025 to 0.150
Gold	Au 2675.95 Au 2427.95[B] Au 3122.78	Pb 3118.92	0.0025 to 0.150
Rhodium	Rh 3396.85 Rh 3323.09 Rh 3283.57 Rh 3372.25	Pb 3118.92	0.0025 to 0.075

[A] Pt 2357.57 may interfere if Pt is high.
[B] Filter, 50 % transmittance.

14.2 *Preparation of Analytical Curves*—Make sufficient exposures of the standards to establish analytical curves. Convert the percent transmittances of the analytical and internal standard lines to log intensity ratios, using the emulsion calibration curve. Prepare analytical curves by plotting the log intensity ratio versus the log concentration for each of the respective elements in the standard samples.

15. Calculation (Note 8)

15.1 Determine the log intensity ratio for each analytical line pair from the emulsion calibration curve. Refer this ratio to the previously prepared analytical curve to obtain the percent concentration in the lead bead.

15.2 Calculate the concentrations of precious metals in the test samples using either equation, as follows:

15.2.1 Calculate the concentration in troy ounces per short ton as follows:

$$C_t = \frac{29.1667EW}{100S} \tag{1}$$

where:
C_t = concentration of element in test sample, troy ounces per short ton,
E = concentration of the element in lead bead, %,
W = weight of the lead bead, mg, and
S = weight of test sample, g.

15.2.2 Calculate the concentration in grams per metric ton as follows:

$$C_m = \frac{10EW}{S} \tag{2}$$

where:
C_m = concentration of element in test sample, grams per metric ton,
E = concentration of the element in lead bead, %,
W = weight of the lead bead, mg, and
S = weight of test sample, g.

Note 8—It is customary in fire assaying to use a factor weight of

sample (**5**). In countries where the metric system is used, values are reported in grams per metric ton. As this is the same as parts per million, the concentration obtained in the above formula can be converted (0.0001 % ≡ 1 ppm) and reported as grams per metric ton. In North America a sample weight is chosen so that each milligram of precious metal in the sample is equivalent to one troy ounce in one avoirdupois ton of ore. As one ton contains 29,166 troy ounces, the assay ton, containing 29.166 g, is the factor weight normally used. Milligrams per assay ton can then be reported as troy ounces per short ton of ore. If this system is used, the sample taken should be an assay ton or some multiple or fraction of an assay ton. One troy ounce per short ton is equivalent to 34.286 parts per million.

16. Precision and Bias

16.1 *Precision*:

16.1.1 In one study, one sample was analyzed repeatedly in a single laboratory to determine the within-laboratory precision for the method. Results from that study are shown in Table 5.

16.1.2 In a second study, two samples were analyzed within a single laboratory with this spectrographic and a spectrophotometric method for comparison. Results from that study are shown in Table 6.

16.1.3 In a third study, two samples were analyzed in four laboratories to determine between-laboratory precision for the method. Results from that study are shown in Table 7.

16.2 *Bias*—No information concerning the bias of this test method is available. However, the bias of the method may be

judged by comparing accepted reference values with the corresponding arithmetic averages obtained by interlaboratory testing.

TABLE 5 Precision Data for a Single Laboratory

Element	Mean Concentration		RSD^A, %	Number of Determinations
	troy ounces per short ton	grams per metric ton		
Silver	2.28	78.1	12.6	332
Palladium	0.173	5.93	5.7	359
Platinum	0.18	6.2	6.1	356
Gold	0.066	2.26	9.1	341
Rhodium	0.063	2.16	7.5	349

^{A} Relative Standard Deviation, RSD, %, calculated as follows:

$$RSD = (100/X) \sqrt{\Sigma d^2/(n-1)}$$

where:
X = mean concentration,
d = difference of the determination from the mean, and
n = number of determinations.

TABLE 6 Comparison of Spectrographic and Spectrophotometric Results Within a Single Laboratory

Sample	Element	Spectrographic Results		Spectrophotometric Results			
		troy ounces per short ton	grams per metric ton	troy ounces per short ton		grams per metric ton	
3572	Palladium	0.168	5.76	0.175	0.14	6.00	4.8
3573	Palladium	0.181	6.21	0.185	0.17	6.34	5.83
3572	Platinum	0.167	5.73	0.161	0.16	5.52	5.5
3573	Platinum	0.17	5.8	0.159	0.15	5.45	5.1
3572	Gold	0.059	2.02	0.054	0.058	1.85	1.99
3573	Gold	0.058	1.99	0.053	0.061	1.82	2.09
3572	Rhodium	0.065	2.23	0.061	0.062	2.09	2.13
3573	Rhodium	0.068	2.33	0.062	0.062	2.13	2.13

TABLE 7 Interlaboratory Precision Data

Laboratory	Silver		Gold		Platinum		Palladium		Rhodium	
	A	B	A	B	A	B	A	B	A	B
Sample 1										
1	0.081	2.78	0.003	0.1	0.0057	0.20	0.0029	0.099	0.0023	0.079
2	0.107	3.67	0.003	0.1	0.005	0.2	0.003	0.1	0.002	0.07
3	0.117	4.01	0.003	0.1	0.006	0.2	0.0035	0.12	0.0021	0.072
4	0.121	4.15	0.005	0.2	0.0068	0.23	0.0035	0.12	0.0025	0.086
Sample 2										
1	0.39	13	0.026	0.89	0.063	2.2	0.059	2.0	0.025	0.86
2	0.341	11.7	0.021	0.72	0.054	1.9	0.053	1.8	0.025	0.86
3	0.5	17	0.027	0.93	0.058	2.0	0.058	2.0	0.027	0.93
4	0.39	13	0.03	1	0.06	2	0.056	1.9	0.023	0.79

^{A} Troy ounces per short ton.
^{B} Grams per metric ton.

17. Keywords

17.1 analysis; fire assay; minerals; ores; precious metals; rocks; spectrographic

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(**9**) Guerin, B. D., "The Effect of Nickel on Fire Assaying Processes for the Determination of Silver," *The Australasian Institute of Mining and Metallurgy, Proceedings*, No. 193, March 1960, p. 1.

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