



Standard Test Method for Chemical Analysis of Refined Gold by Direct Current Plasma Emission Spectrometry¹

This standard is issued under the fixed designation E 1446; 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.

^{ε1} NOTE—E 1446 was reapproved with a title change in April 1997.

1. Scope

1.1 This test method covers the analysis of refined gold for the following elements having the following chemical composition limits:

Element	Concentration Range, ppm
Copper	17 to 300
Iron	6 to 150
Lead	17 to 100
Palladium	7 to 350
Silver	17 to 500

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.* For specific precautionary statements, see Section 8.

2. Referenced Documents

2.1 ASTM Standards:

- B 562 Specification for Refined Gold²
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁴
- E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals⁴
- E 691 Practice for Conducting An Interlaboratory Study to Determine the Precision of a Test Method³
- E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory⁵
- E 1097 Guide for Direct Current Plasma Emission Spectrometry Analysis⁵

3. Summary of Test Method

3.1 The sample is dissolved with acids. Calibration solu-

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 E01.03 on Precious Metals.

Current edition approved Jan. 15, 1992. Published June 1992.

² *Annual Book of ASTM Standards*, Vol 02.04.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.05.

⁵ *Annual Book of ASTM Standards*, Vol 03.06.

tions are prepared from pure reagents to match the sample matrix. Analysis is performed with the d-c argon plasma optical emission spectrometer. Element concentrations are measured by comparing emission intensities from the sample with those of the calibration solutions. Copper is measured at 327.3 nm or 324.7 nm; iron at 259.9 nm; lead at 405.7 nm; palladium at 340.4 nm; and silver at 328.0 nm or 338.3 nm.

4. Significance and Use

4.1 This test method for the analysis of fine gold is primarily intended to test such material for compliance with compositional specifications. It is assumed that all who use this test method will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

5. Interferences

5.1 Spectral line interferences and spectrochemical background effects are overcome by preparing a matrix-matched calibration standard series to approximate the prepared sample.

5.2 The analyte wavelengths mentioned herein have been previously evaluated for spectral line interferences and found to be the optimum emission wavelengths for refined gold sample testing. Alternative wavelengths, if shown to be free of interferences, may also be used.

6. Apparatus

6.1 *D-C Argon Plasma Optical Emission Spectrometer*—The instrument must be equipped with a sample nebulization system compatible with mineral acids and with test solutions containing 4 % total solids. Follow the manufacturer's instructions for installation and operation.

7. Reagents and Materials

7.1 *Argon*—Purity: 99.998 % minimum.

7.2 *Copper Standard Solution* (1 mL = 1.0 mg Cu)—Transfer 1.000 g of copper metal (purity: 99.9 % minimum) to a 400-mL beaker. Add 60 mL of HNO₃(1+1) in 10-mL increments and heat gently on a hot plate. When dissolution is complete, cool, then transfer to a 1-L volumetric flask. Dilute to volume with HNO₃(1+99) and mix. This solution is stable for at least six months.

7.3 *Iron Standard Solution* (1 mL = 1.0 mg Fe)—Transfer 1.000 g of iron metal (purity: 99.9 % minimum) to a 400-mL beaker. Add 50 mL of HCl plus HNO₃(4+1) and heat gently on a hot plate. When dissolution is complete, cool and then transfer to a 1-L volumetric flask. Dilute to volume with HCl (1+99) and mix. This solution is stable for six months.

7.4 *Lead Standard Solution* (1 mL = 1.0 mg Pb)—Transfer 1.000 g of lead metal (purity: 99.9 % minimum) to a 400-mL beaker. Add 50 mL of HNO₃(1+4) and heat gently on hot plate to dissolve and then cool. Transfer the lead solution to a 1-L volumetric flask, dilute to volume with water, and mix. This solution is stable for six months.

7.5 *Matrix Gold*—Gold purity must be a minimum 99.9999 % based on total metallic impurities.

7.6 *Palladium Standard Solution* (1 mL = 1.0 mg Pd)—Transfer 1.000 g of palladium metal (purity: 99.9 % minimum) to a 400-mL beaker. Add 50 mL of HCl plus HNO₃(4+1) and heat gently on a hot plate. When dissolution is complete, cool, then transfer to a 1-L volumetric flask and dilute to volume with HCl (1+99) and mix. This solution is stable for six months.

7.7 *Silver Standard Solution* (1 mL = 1.0 mg Ag)—Transfer 1.000 g of silver metal (purity: 99.9 % minimum) to a 400-mL beaker. Add 60 mL of HNO₃(1+1) and heat gently on a hot plate. When dissolution is complete, cool, then transfer to a 1-L volumetric flask, dilute to volume with HNO₃(1+99), and mix. This solution should be stored in an amber glass container or in the dark. This solution is stable for at least six months. Any chlorides must be kept away from this solution or the silver will be precipitated.

8. Hazards

8.1 For precautions to be observed in the use of certain reagents and equipment, refer to Practices E 50.

8.2 The ultraviolet radiation from the plasma must be shielded at all times to prevent eye damage.

8.3 All dissolution by acids must be performed under a hood with proper ventilation.

9. Sampling

9.1 For appropriate procedures on sampling the materials covered by this test method, refer to Specification B 562.

10. Calibration

10.1 Calibration Solutions:

10.1.1 *Master Analyte Solution*—Clean a 100-mL volumetric flask by boiling with 10 mL of HCl (1+1) for 10 min and rinsing with water. Add 30 mL of HCl and transfer by means of a pipet the amounts of standard solutions listed in Table 1. Dilute to the mark and mix. Silver will not precipitate as silver

chloride because of the presence of excess HCl. This solution is stable for no longer than five days.

10.1.2 *High Calibration Solution*—Weigh 2.000 ± 0.003 g of matrix gold and transfer to a 50-mL volumetric flask, previously cleaned by boiling with HCl (1+1) for 10 min and rinsing with water. Add 15 mL of HCl plus HNO₃(4+1) and heat gently until dissolved. If the gold does not completely dissolve, add 10 mL of HCl plus HNO₃(4+1) and heat gently to complete dissolution. Cool, then transfer by pipet 10 mL of master analyte solution and dilute to volume with HCl (1+2), and mix. This solution is equivalent to 300 ppm copper, 100 ppm iron, 100 ppm lead, 300 ppm palladium, and 500 ppm silver in the matrix gold.

10.1.3 *Medium Calibration Solution*—Weigh 2.000 ± 0.003 g of matrix gold and transfer to a 50-mL volumetric flask, previously cleaned by boiling with HCl (1+1) for 10 min and rinsing with water. Add 15 mL of HCl plus HNO₃(4+1) and heat gently until dissolved. If the gold does not dissolve completely, add 10 mL of HCl plus HNO₃(4+1) and heat gently to complete dissolution. Cool, then transfer by pipet 5 mL of the master analyte solution and dilute to volume with HCl (1+2), and mix. This solution is equivalent to 150 ppm copper; 50 ppm iron; 50 ppm lead; 150 ppm palladium; and 250 ppm silver in the matrix gold.

10.1.4 *Low Calibration Solution*—Weigh 2.000 ± 0.003 g of matrix gold and transfer to a 50-mL volumetric flask, previously cleaned by boiling with HCl (1+1) for 10 min and rinsing with water. Add 15 mL of HCl plus HNO₃(4+1) solution and heat gently until dissolved. If the gold does not completely dissolve, add 10 mL of HCl plus HNO₃(4+1) and heat gently to complete dissolution. Cool, then transfer by pipet 1 mL of master analyte solution and dilute to volume with HCl (1+2), and mix. This solution is equivalent to 30 ppm copper; 10 ppm iron; 10 ppm lead; 30 ppm palladium; and 50 ppm silver in the matrix gold.

10.1.5 *Blank Calibration Solution*—Weigh 2.000 ± 0.003 g of matrix gold and transfer to a 50-mL volumetric flask, previously cleaned by boiling with HCl (1+1) for 10 min and rinsing with water. Add 15 mL of HCl plus HNO₃(4+1) solution and heat gently until dissolved. If the gold does not completely dissolve, add 10 mL of HCl plus HNO₃(4+1) and heat gently to complete dissolution. Cool and dilute to volume with HCl (1+2) and mix. This solution is equivalent to 0 ppm copper; 0 ppm iron; 0 ppm lead; 0 ppm palladium; and 0 ppm silver in the matrix gold.

10.2 *Instrument Optimization*—Adjust all instrumental parameters according to the instrument manufacturer's recommended operating practices. Use the high calibration solution when aligning optics and entrance slit positions. The element emission wavelengths given in Table 2 have been shown to be linear and interference-free for refined gold matrices.

TABLE 1 Master Analyte Solution

Element	Standard Solution, mL	Master Analyte Solution Final Concentration, µg/mL
Copper	6	60
Iron	2	20
Lead	2	20
Palladium	6	60
Silver	10	100

TABLE 2 Element Emission Wavelengths

Element	Wavelength, nm	Alternate Wavelength, nm
Copper	327.3	324.7
Iron	259.9	...
Lead	405.7	...
Palladium	340.4	...
Silver	328.0	338.3

10.3 *Instrument Calibration*—Following the instrument manufacturer’s instructions, calibrate the spectrometer using the low-calibration solution and the high-calibration solution at the wavelengths listed in 10.2 and using three, 8-s integrations. If the instrument will only accept a high standard and a blank, use the high-calibration solution and the blank calibration solution. Rinse the aspiration system with HCl (1+2) for at least 15 s between calibration solutions.

10.4 *Calibration Verification*—Analyze the high, low, or blank and medium calibration solutions using the instrument manufacturer’s recommended procedure. If all values are not within 5 % of the expected value, repeat the calibration procedure.

11. Procedure

11.1 Test Solution:

11.1.1 Weigh 1.000 ± 0.001 g of the sample and transfer to a 25-mL volumetric flask, previously cleaned by boiling with HCl (1+1) for 10 min and rinsing with water. Add 7 mL of HCl plus HNO₃ (4+1) and heat gently until dissolved. If the sample does not completely dissolve or silver chloride is seen as a precipitate, add 5 mL of HCl plus HNO₃(4+1) and heat gently to complete the dissolution. Cool, then dilute to volume with HCl (1+2), and mix.

11.1.2 For the determination of each element, aspirate the test solution and record its concentration, using the instrument manufacturer’s recommended procedure. Verify the calibration after every four sample determinations. If the measured values for the calibration solutions are not within 5 % of expected values, repeat the calibration procedure and the sample measurement. Aspirate HCl (1+2) for at least 15 s after each test or calibration solution.

11.1.3 As an alternative to verifying calibration, the drift correction procedure as described in Guide E 1097 may be used.

12. Calculation

12.1 This test method is designed so that direct reading instruments will provide results in parts per million weight concentration in the refined gold sample material.

13. Precision and Bias

13.1 *Precision*⁶—This test method was tested in an inter-laboratory testing program with seven participating laboratories. Three samples were tested for the determination of silver, palladium, and lead, and four samples were tested for the determination of iron. The results are summarized in Table 3.

13.2 *Bias*—No information on the accuracy of this test method is known.

TABLE 3 Statistical Information

Element	Sample	Test Mean, ppm ^A	R1, ppm ^A	R2, ppm ^A
Copper	1A	111.2	3.83	6.30
	2A	311.4	7.73	8.56
	3A	8.0	0.95	1.08
Iron	1A	4.1	0.93	1.53
	2A	4.3	0.49	1.31
	3A	2.4	0.82	1.22
	4A	141.3	2.40	6.81
Lead	1A	31.2	0.78	1.49
	2A	72.1	2.84	3.29
	3A	18.8	0.67	1.33
Palladium	1A	339.1	10.97	15.83
	2A	106.9	3.93	4.57
	3A	12.4	0.65	0.83
Silver	1A	19.5	1.47	1.59
	2A	228.7	5.14	9.90
	3A	519.3	8.19	25.48

^A To convert parts per million (ppm) to percent (%), divide the ppm value by 10 000.

14. Keywords

14.1 direct current argon plasma spectroscopy; gold; spectrometry

⁶ Supporting data are available from ASTM Headquarters. Request RR: E01.0300.

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