



Designation: E1446 – 05

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

This standard is issued under the fixed designation E1446; 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 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

[B562 Specification for Refined Gold](#)

[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](#)

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 E01.05 on Cu, Pb, Zn, Cd, Sn, Be, Precious Metals, their Alloys, and Related Metals.

Current edition approved Oct. 1, 2005. Published December 2005. Originally approved in 1992. Last previous edition approved in 1997 as E1446 – 92 (1997)^{ε1}. DOI: 10.1520/E1446-05.

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.

[E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals \(Withdrawn 1998\)](#)³

[E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory](#)

[E1097 Guide for Determination of Various Elements by Direct Current Plasma Atomic Emission Spectrometry](#)

[E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method](#)

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology [E135](#).

4. Summary of Test Method

4.1 The sample is dissolved with acids. Calibration solutions 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.

5. Significance and Use

5.1 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 and operated in accordance with Guide [E882](#).

6. Interferences

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

³ The last approved version of this historical standard is referenced on www.astm.org.

6.2 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.

7. Apparatus

7.1 *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.

8. Reagents and Materials

8.1 *Argon*—Purity: 99.998 % minimum.

8.2 *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.

8.3 *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.

8.4 *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.

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

8.6 *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.

8.7 *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.

9. Hazards

9.1 For precautions to be observed in the use of certain reagents and equipment, refer to Practices E50.

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

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

10. Sampling

10.1 For appropriate procedures on sampling the materials covered by this test method, refer to Specification B562.

11. Interlaboratory Studies

11.1 This test method was evaluated in accordance with Practice E173. Practice E173 has been replaced by Practice E1601. The Reproducibility Index, R₂ of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. Likewise the Repeatability Index R₁ of Practice E173 corresponds to Repeatability Index r of Practice E1601.

12. Calibration

12.1 *Calibration Solutions:*

12.1.1 *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.

12.1.2 *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.

12.1.3 *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.

TABLE 1 Master Analyte Solution

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