Designation: E1446 – 13

Standard Test Method for Chemical Analysis of Refined Gold by Direct Current Plasma Atomic Emission Spectrometry [1]

1. Scope

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

Element	Content Range, µg/g
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

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]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 direct-current plasma atomic emission spectrometer. Element contents 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.

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[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.

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[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.

[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 Atomic 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 HNO_3 (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 HNO_3 (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 HNO_3 (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 HNO_3 (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 HNO_3 (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 HNO_3 (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 HNO_3 (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.

NOTE 1—Commercially available certified or traceable element standards may also be used for calibration.

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, R_2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. Likewise the Repeatability Index R_1 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 g ± 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 HNO_3 (4 + 1) and heat gently until dissolved. If the gold does not completely dissolve, add 10 mL of HCl plus HNO_3 (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 µg/g copper, 100 µg/g iron, 100 µg/g lead, 300 µg/g palladium, and 500 µg/g silver in the matrix gold.

12.1.3 *Medium Calibration Solution*—Weigh 2.000 g ± 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 HNO_3 (4 + 1) and heat gently until dissolved. If the gold does not dissolve completely, add 10 mL of HCl plus HNO_3 (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 µg/g copper; 50 µg/g iron; 50 µg/g lead; 150 µg/g palladium; and 250 µg/g silver in the matrix gold.

TABLE 1 Master Analyte Solution

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

12.1.4 *Low Calibration Solution*—Weigh 2.000 g ± 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 HNO_3 (4 + 1) solution and heat gently until dissolved. If the gold does not completely dissolve, add 10 mL of HCl plus HNO_3 (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 µg/g copper; 10 µg/g iron; 10 µg/g lead; 30 µg/g palladium; and 50 µg/g silver in the matrix gold.

12.1.5 *Blank Calibration Solution*—Weigh 2.000 g ± 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 HNO_3 (4 + 1) solution and heat gently until dissolved. If the gold does not completely dissolve, add 10 mL of HCl plus HNO_3 (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 µg/g copper; 0 µg/g iron; 0 µg/g lead; 0 µg/g palladium; and 0 µg/g silver in the matrix gold.

12.2 *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.

12.3 *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 12.2 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.

12.4 *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.

13. Procedure

13.1 *Test Solution:*

13.1.1 Weigh 1.000 g ± 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 HNO_3 (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 HNO_3 (4 + 1) and heat gently to complete the dissolution. Cool, then dilute to volume with HCl (1 + 2), and mix.

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

TABLE 3 Statistical Information

Element	Sample	Test Mean, µg/g[A]	r, µg/g[A]	R, µg/g[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 micrograms per gram (µg/g) to percent (%), divide the µg/g value by 10 000 and report in accordance with Practice E29.

13.1.2 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.

13.1.3 As an alternative to verifying calibration, the drift correction procedure as described in Guide E1097 may be used.

14. Calculation

14.1 This test method is designed so that direct reading instruments will provide results in µg/g content in the refined gold sample material.

15. Precision and Bias

15.1 *Precision*[4]—This test method was tested in an interlaboratory testing program with seven participating laboratories. Three samples were tested for the determination of copper, silver, palladium, and lead, and four samples were tested for the determination of iron. The results are summarized in Table 3.

15.2 *Bias*—No information on the accuracy of this test method is known because at the time it was tested, no certified reference materials were available. Users are encouraged to employ suitable reference materials if available to verify the accuracy of the method in their laboratory.

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-0300.

16. Keywords

16.1 DCP-AES; direct current argon atomic spectroscopy; gold; spectrometry

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