



Designation: **E1600—07 E1600 – 13**

Standard Test Method for Determination of Gold in Cyanide Solutions by Flame Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation E1600; 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 determination of gold in ore processing cyanide solution in the range from 0.3 $\mu\text{g/mL}$ to 10.0 $\mu\text{g/mL}$ of gold by direct aspiration into an atomic absorption ~~spectrophotometer~~spectrometer.

1.1.1 This test method may also be applied to cyanide leach solutions from metallurgical evaluation procedures.

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.* Specific precautions are given in 8.1, ~~8.38.1.1~~, 8.5, ~~Section 9~~, and ~~10.4.1-11.2~~.

2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

D1293 Test Methods for pH of Water

D6888 Test Method for Available Cyanide with Ligand Displacement and Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection

D7237 Test Method for Free Cyanide with Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection

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E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)³

~~E663 Practice for Flame Atomic Absorption Analysis (Withdrawn 1997)³~~

~~E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory~~

~~E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry (Withdrawn 2004)³~~

E1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis (Withdrawn 1997)³

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 solution is collected and preserved with sodium hydroxide, if necessary, by careful adjustment of pH. The test solution is filtered and gold content is determined by flame atomic absorption ~~spectrophotometry~~spectrometry.

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 **E01.02** on Ores, Concentrates, and Related Metallurgical Materials.

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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.

5. Significance and Use

5.1 In primary metallurgical processes for gold bearing ores, gold is extracted with an alkaline cyanide solution. Metallurgical accounting, process control, and ore evaluation procedures depend on accurate, precise, and prompt measurements of the gold concentrations.

5.2 This test method is a comparative referee method for compliance with compositional specifications for metal content: concentration or to monitor processes. It is assumed that all who use this procedure 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 that proper waste disposal procedures will be followed. Follow under appropriate quality control practices such as those described in Guide E882, and that proper waste disposal procedures will be followed.

6. Interferences

6.1 Elements normally found in ore processing cyanide solutions do not interfere. Use of instrumental background correction is required to compensate for nonspecific absorption interferences in the flame.

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer, Spectrometer*, equipped with background correction and capable of measuring gold at the 242.8-nm wavelength using an air and acetylene flame over a linear range from 0.3 $\mu\text{g/mL}$ to 10.0 $\mu\text{g/mL}$ gold.

8. Reagents and Materials

8.1 *Gold Calibration Solutions (0.5, 1.0, 2.0, 5.0, 10.0) $\mu\text{g/mL}$* —In a fume hood, pipette 10 mL of Gold Standard Solution A (8.2) into a 1-L volumetric flask containing 100 mL of Sodium Cyanide-Sodium Hydroxide Solution (8.5). Dilute to volume and mix (10 $\mu\text{g/mL}$).

8.1.1 Pipette (5, 10, 20, and 50) mL of the 10 $\mu\text{g/mL}$ gold calibration solution into each of four 100-mL volumetric flasks, respectively. Add 10 mL of Sodium Cyanide-Sodium Hydroxide Solution (8.5), dilute to volume, and mix.

WARNING—Reaction of acid or chlorine and cyanide solutions releases toxic hydrogen cyanide or cyanogen chloride gases. Prepare in a fume hood.

8.2 *Gold Standard Solution A (1 mL – 1.0 $\mu\text{g Au}$)*—Weigh 1.000 g of gold metal (99.99 % minimum purity) and transfer to a 1-L beaker in a fume hood. Add 200 mL of water, 80 mL of HCl, and 50 mL of HNO_3 (1 + 1). Boil gently to expel NO_x fumes, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

8.2.1 A certified reference solution meeting these specifications may also be used.

8.3 *Reference Solution*—Dilute 100 mL of Sodium Cyanide-Sodium Hydroxide Solution (8.5), to 1 L with water.

8.4 *Sodium Cyanide.*

8.5 *Sodium Cyanide-Sodium Hydroxide Solution*—Dissolve 10 g of sodium hydroxide and 10 g of sodium cyanide in 1 L of water.

WARNING —The preparation, storage, use, and disposal of sodium cyanide solutions require special care and attention. Avoid any possibility of inhalation, ingestion, or skin contact with the compound, its solution, or its vapors. Work only in a well-ventilated hood.

8.6 *Sodium Hydroxide.*

8.7 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society werewhere such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.8 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by conforming to Type I or II of Specification D1193. Type III or IV may be used if they effect no measurable change in the blank or sample.

8.3 *Sodium Cyanide-Sodium Hydroxide Solution*—Dissolve 10 g of sodium hydroxide (NaOH) and 10 g of sodium cyanide (NaCN) in 1 L of water. (**Warning** —The preparation, storage, use, and disposal of NaCN solutions require special care and attention. Avoid any possibility of inhalation, ingestion, or skin contact with the compound, its solution, or its vapors. Work only in a well-ventilated hood. Refer to Practices E50.)

⁴ *Reagent Chemical, American Chemical Society Specifications*, ; American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Reagent Chemicals and Standards*, by Joseph Rosin, D. Nostrand Co., Inc., New York, NY, and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc., (USPC), Rockville, MD.

8.4 *Gold Standard Solution A*—Weigh 1.000 g of gold metal (99.99 % min purity) and transfer to a 1-L beaker in a fume hood. Add 200 mL of water, 80 mL of HCl, and 50 mL of HNO₃ (1 + 1). Boil gently to expel NO_x fumes, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

8.4.1 A certified reference solution meeting these specifications may also be used.

8.5 *Gold Calibration Solutions* (0.5, 1.0, 2.0, 5.0, 10.0 µg/mL)—In a fume hood, pipette 10 mL of gold standard Solution A into a 1-L volumetric flask containing 100 mL of sodium cyanide-sodium hydroxide solution. Dilute to volume and mix (10 µg/mL). (**Warning**—Reaction of acid or chlorine and cyanide solutions releases toxic hydrogen cyanide or cyanogen chloride gases. Prepare in a fume hood.)

8.5.1 Pipette 5, 10, 20, and 50 mL of the 10 µg/mL gold calibration solution into each of four 100-mL volumetric flasks, respectively. Add 10 mL of sodium cyanide-sodium hydroxide solution, dilute to volume, and mix.

8.6 *Reference Solution*—Dilute 100 mL of sodium cyanide-sodium hydroxide solution to 1 L with water.

9. Hazards

9.1 For precautions to be observed in this method, refer to Practice E50.

9.2 Hydrogen cyanide and alkali cyanide are very toxic substances. Use an efficient fume hood. Cyanide must be disposed of with care, avoiding contact with acid that releases hydrogen cyanide gas. Oxidation of cyanide with chlorine or hypochlorite must be carried out at high pH (greater than 11) to prevent generation of toxic cyanogen chloride gas.

9.3 See specific warnings in 8.1, 8.1.1, 8.5, and 11.2.

10. Sampling and Sample Preparation

10.1 Collect, store, and dispose of the sample in accordance with Practices E50.

10.2 *Preservation*—Determine the pH of the solution immediately after sampling in accordance with Test Method D1293. If the pH of the sample is less than 10, adjust the pH with small additions of solid sodium hydroxide, followed by mixing, until the pH is greater than 10.

10.3 Samples may be preserved to pH 11 or higher if they are also being tested for free and weak acid dissociable cyanide in accordance with Test Methods D6888 or D7237.

10.4 *Test Solutions*—Filter two 50-mL portions of preserved sample solution through a coarse-porosity filter paper.

11. Preparation of Apparatus

11.1 Follow the instrument manufacturer's instructions to adjust the instrument for gold at 242.8 nm. Warm up the instrument with background correction applied in accordance with the manufacturer's instructions. With the gold hollow cathode lamp in position, energized and stabilized, adjust the wavelength to maximize the energy response of the 242.8-nm line. Light the burner, allow it to reach thermal equilibrium, and adjust the instrument to zero while aspirating water.

11.2 The use of an air-acetylene, lean, blue flame and caustic stabilized drain bottle is required.

WARNING—Reaction of acid and cyanide solutions in the burner chamber drain bottle may release toxic hydrogen cyanide gas. Add an excess of sodium hydroxide to the drain bottle to maintain the pH above eleven.

11.3 Determine if the instrument precision is acceptable as follows:

11.3.1 Calibrate the instrument in accordance with the manufacturer's instructions in absorbance. Set the absorbance to zero while aspirating the reference solution.

11.3.2 Aspirate the calibration solutions in order of increasing concentration, and select a calibration solution in the absorbance range from 0.2 to 0.4.

11.3.3 Alternate readings on the selected calibration solution and reference solution, and calculate the standard deviation of the readings on the selected calibration solution using accepted statistical methods. Measure the standard deviation in this way at increased measurement integration times until a relatively constant value is achieved.

11.3.4 If the standard deviation under these conditions is greater than 1 % of the average absorbance, determine the cause of the variability (for example, deposits in the burner or clogged capillary), and take corrective action.

11.3.5 If the minimum requirements are not met, do not use the instrument with this test method until the required stability is obtained.

11.3.6 Collect all instrumental measurements for the test method using the instrumental settings which gave the optimum precision of measurement on the selected calibration solution.

11.4 *Linearity of Instrument Response*—Determine if the instrument response is acceptable as follows:

11.4.1 Record absorbance measurements for each of the calibration solutions and the reference solution, prior to determining samples.

11.4.2 Adequate instrument response is obtained if the difference between the 5-µg/mL calibration solution and the 10-µg/mL calibration solution is sufficient to permit estimation of 1/50 of the difference between them.