ASTM

Designation: E 1600 – 96

Standard Test Method for
Determination of Gold in Cyanide Solutions by Atomic Absorption Spectrometry[1]

This standard is issued under the fixed designation E 1600; 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 to 10.0 µg/mL of gold by direct aspiration into an atomic absorption spectrophotometer.

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

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 precautions are given in Section 8 and in specific warning notes throughout.

2. Referenced Documents

2.1 *ASTM Standards:*
D 1293 Test Methods for pH of Water[2]
E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[3]
E 50 Practices for Apparatus, Reagents and Safety Precautions for Chemical Analysis of Metals[4]
E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[4]
E 663 Practice for Flame Atomic Absorption Analysis[5]
E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[5]
E 1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry[5]
E 1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis[5]

3. Summary of Test Method

3.1 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.

4. Significance and Use

4.1 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.

4.2 This test method is a referee method for compliance with compositional specifications for metal content. It is assumed that all who use this procedure 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 appropriate quality control practices such as those described in Guide E 882.

5. Interferences

5.1 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.

6. Apparatus

6.1 *Atomic Absorption Spectrophotometer*, 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 to 10.0 µg/mL gold.

7. Reagents

7.1 *Sodium Cyanide–Sodium Hydroxide Solution*—Dissolve 10 g of sodium hydroxide (NaOH) and 10 g of sodium cyanide (NaCN) in 1 L of water.

NOTE 1—**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 E 50.

7.2 *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 $HNO_3(1 + 1)$. Boil gently to expel NO_x fumes, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

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

7.3 *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).

NOTE 2—**Warning:** Reaction of acid or chlorine and cyanide solutions releases toxic hydrogen cyanide or cyanogen chloride gases. Prepare in a fume hood.

7.3.1 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.

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

8. Hazards

8.1 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. Procedure

9.1 Collect, store, and dispose of the sample in accordance with Practices E 50.

9.2 Determine the pH of the solution immediately after sampling in accordance with Test Method D 1293. 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.

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

9.4 *Photometry*:

9.4.1 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.

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

NOTE 3—**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 11.

9.4.2 *Precision of Measurements*—Use Practice E 663 to determine if the instrument precision is acceptable as follows:

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

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

9.4.2.3 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.

9.4.2.4 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.

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

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

9.4.3 *Linearity of Instrument Response*—Use Practice E 663 to determine if the instrument response is acceptable as follows:

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

9.4.3.2 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 $\frac{1}{50}$ of the difference between them.

9.4.3.3 Adequate linearity is confirmed if the slope of the calibration curve between the 5 and 10-µg/mL calibration solutions is at least 90 % of the slope between the reference solution and the 0.5-µg/mL calibration solution.

9.4.4 *High-Precision Method*:

9.4.4.1 Calibrate the instrument in accordance with the manufacturer's instructions in absorbance or gold concentration.

9.4.4.2 Adjust the instrument to zero with the reference solution and measure the test sample solution to determine its place in the order of increasing concentration of the calibration solutions.

9.4.4.3 Aspirate the test solution and the closely bracketing calibration solutions in order of increasing absorbance or concentration without intervening water aspirations. Repeat three times and calculate the average absorbance or concentration value for each of the three solutions.

9.4.5 *Linear Curve Method*:

9.4.5.1 Calibrate the instrument in accordance with the manufacturer's instructions in absorbance or gold concentration.

9.4.5.2 Record the reference solution and calibration solution readings before and after each test sample solution, selecting a different calibration solution after each test solution.

9.4.5.3 Continue recording measurements until at least three readings have been recorded for all test sample solutions and at least one reading has been recorded for each calibration solution. Calculate the average reading for each of the solutions.

10. Calculation

10.1 *High-Precision Method*—The gold concentration of

the test solution is calculated in accordance with Guide E 1024 as follows:

$$C_t = \frac{A_t(C_h - C_1)}{(A_h - A_1)} \qquad (1)$$

where:
C_t = concentration of gold in the test solution, µg/mL,
C_h = concentration of gold in the higher calibration solution, µg/mL,
C_1 = concentration of gold in the lower calibration solution, µg/mL,
A_t = average absorbance or concentration reading of the test solution,
A_h = average absorbance or concentration reading of the higher calibration solution, and
A_1 = average absorbance or concentration of the lower calibration solution.

10.2 *Linear Curve Method*—Calculate the gold concentration of each test sample solution in micrograms per millilitre using the graphical method in accordance with Guide E 1024, by simple linear regression, or by an equivalent computer method.

10.3 Average the results of the duplicate test sample solutions and round the results to the nearest 0.1 µg/mL in accordance with Practice E 29.

11. Precision and Bias

11.1 *Precision*—An interlaboratory study was undertaken to test the precision of this test method in accordance with Practice E 173 on six solutions in eight laboratories. The results from the study are summarized in Table 1. Since as few as three laboratories returned results for some of the materials, Practice E 1060 was used to estimate the precision. The base

TABLE 1 Gold in Cyanide Solutions—Statistical Information

Solutions	Mean, Au, µg/mL	R1 (Practice E 1060), Au, µg/mL	R2 (Practice E 1060), Au, µg/mL
1, 4	2.19	0.10	0.18
2, 5	0.19	0.05	0.21
3, 6	0.96	0.02	0.05
7, 10	4.87	0.17	0.22
8, 11	5.97	0.27	0.69
9, 12	10.7	0.15	1.28

data and statistics are documented.[6]

NOTE 4—Solutions 1 through 6 were analyzed by more laboratories than Solutions 7 through 12.

11.1.1 *Repeatability*—The repeatability standard deviation (s_w) ranged from 0.01 to 0.12 µg/mL gold over the range of the materials tested. The R1 value in Table 1 for each of the materials tested indicates the maximum difference expected between results in a single laboratory at 95 % confidence.

11.1.2 *Reproducibility*—The reproducibility standard deviation (s_{sr}) ranged from 0.01 to 0.15 µg/mL gold over the range of the materials tested. The R2 value in Table 1 for each of the materials tested indicates the maximum difference expected between results in different laboratories at 95 % confidence.

11.2 *Bias*—No information on the bias of this test method is known. Accepted reference materials were not included in the materials used in the interlaboratory study. Users of this test method are encouraged to employ accepted reference materials, if available, and to judge the bias of the test method from the difference between the accepted value for the gold concentration and the mean value from interlaboratory testing of the reference material.

12. Keywords

12.1 cyanide solutions; gold content

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[6] Supporting data are available from ASTM Headquarters. Request RR:E01-1013.