



Designation: E1600 – 23

Standard Test Methods for Determination of Gold in Cyanide Solutions¹

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1. Scope

1.1 These test methods cover the determination of gold in ore processing cyanide solutions within the following ranges:

Method	Application Range, $\mu\text{g/mL}$
Inductively Coupled Plasma Mass Spectrometry	0.001 to 0.500
Flame Atomic Absorption Spectrometry	0.300 to 10.0

NOTE 1—The lower limit for the Inductively Coupled Plasma Mass Spectrometry Method, 0.001 $\mu\text{g/mL}$, was set following the guidance of Practice E1601. The reproducibility Index, R, was calculated using the total standard deviation for the lowest concentration Youden pair solution.

1.1.1 These test methods 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 The test methods appear in the following order:

Method	Sections
Flame Atomic Absorption Spectrometry	9 – 16
Inductively Coupled Plasma Mass Spectrometry	17 – 24

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautions are given in 11.1.1, 11.5 and 12.2.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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2. Referenced Documents

2.1 ASTM Standards:²

- D1193 Specification for Reagent Water
- D1293 Test Methods for pH of Water
- D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
- D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry
- D6888 Test Method for Available Cyanides with Ligand Displacement and Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection
- D7237 Test Method for Free Cyanide and Aquatic 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 1997)³
- E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
- 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 these test methods, refer to Terminology E135.

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.

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 levels.

4.2 These test methods are comparative referee methods for compliance with compositional specifications for metal amounts or to monitor processes. It is assumed that all who use these methods will be trained users capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory under appropriate quality control practices such as those described in Guide E882, and that proper waste disposal procedures will be followed.

5. Hazards

5.1 For precautions to be observed in these methods, refer to Practice E50.

5.2 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 conducted at high pH (greater than 11) to prevent generation of toxic cyanogen chloride gas.

5.3 See specific warnings in 11.1.1, 11.5 and 12.2.

6. Sampling and Sample Preparation

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

6.2 *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 NaOH, followed by mixing, until the pH is greater than 10.

6.3 Samples may be preserved at 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.

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

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where 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.

⁴ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall mean reagent water 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.

FLAME ATOMIC ABSORPTION SPECTROMETRY

8. Summary of Test Method

8.1 The sample solution is collected and preserved with NaOH, if necessary, by careful adjustment of pH. The test solution is filtered and gold content is determined by flame atomic absorption spectrometry.

9. Interferences

9.1 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.

NOTE 2—Alkaline soluble arsenic can cause low bias on Au by Flame AAS. Sample Dilution, matrix spikes, or Method of Standard Additions may be needed.

10. Apparatus

10.1 *Flame Atomic Absorption 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 µg/mL to 10.0 µg/mL gold.

11. Reagents and Materials

11.1 *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 (11.2) into a 1-L volumetric flask containing 100 mL of Sodium Cyanide-Sodium Hydroxide Solution (11.5). Dilute to volume and mix (10 µg/mL).

11.1.1 Pipette (5, 10, 20, and 50) mL of the 10 µg/mL gold calibration solution into each of four 100-mL volumetric flasks, respectively to make calibration solutions of (0.5, 1.0, 2.0, and 5.0) µg/mL. Add 10 mL of Sodium Cyanide-Sodium Hydroxide Solution (11.5), 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.

11.2 *Gold Standard Solution A (1 mL – 1.0 µg 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 HNO₃ (1 + 1). Boil gently to expel NO_x fumes, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

11.2.1 A certified reference solution, made by an accredited ISO 17034 producer, meeting these specifications may also be used.

NOTE 3—Commercially prepared Gold Cyanide reference solutions are best preserved in NaCN.

11.3 *Reference Solution*—Dilute 100 mL of Sodium Cyanide-Sodium Hydroxide Solution (11.5), to 1 L with water.

11.4 *Sodium Cyanide*.

11.5 *Sodium Cyanide–Sodium Hydroxide Solution*—Dissolve 10 g of NaOH, then 10 g of sodium cyanide in 1 L of water.

WARNING—The preparation, storage, use, and disposal of sodium cyanide solutions requires 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.

12. Preparation of Apparatus

12.1 Follow the instrument manufacturer's instructions to adjust the instrument for gold at 242.8 nm. Stabilize 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 wavelength. Ignite the burner, allow it to reach thermal equilibrium, and adjust the instrument to zero while aspirating water.

12.2 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 NaOH to the drain bottle to maintain the pH above eleven.

12.3 Determine if the instrument precision is acceptable as follows:

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

12.3.2 Aspirate the calibration solutions in order of increasing concentration, and select a calibration solution from 0.2 absorbance units (AU) to 0.4 AU.

12.3.3 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.

12.3.4 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.

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

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

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

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

12.4.2 Adequate instrument response is obtained if the difference between the 5- $\mu\text{g/mL}$ calibration solution is suffi-

cient to permit estimation of $\frac{1}{50}$ of the difference between absorbance measurements (0.1 $\mu\text{g/mL}$).

12.4.3 Adequate linearity is confirmed if the slope of the calibration curve between the 5 $\mu\text{g/mL}$ and 10 $\mu\text{g/mL}$ calibration solutions is at least 90 % of the slope between the reference solution and the 0.5- $\mu\text{g/mL}$ calibration solution.

13. Calibration

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

14. Procedure

14.1 *High-Precision Method*:

14.1.1 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.

14.1.2 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.

14.2 *Linear Curve Method*:

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

14.2.2 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.

15. Calculation

15.1 *High-Precision Method*—The gold concentration of the test solution is calculated as follows:

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

where:

- C_t = concentration of gold in the test solution, $\mu\text{g/mL}$,
- C_h = concentration of gold in the higher calibration solution, $\mu\text{g/mL}$,
- C_1 = concentration of gold in the lower calibration solution, $\mu\text{g/mL}$,
- 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.

15.2 *Linear Curve Method*—Calculate the gold concentration of each test sample solution in $\mu\text{g/mL}$ using the graphical method, by simple linear regression, or by an equivalent computer method.

15.3 Average the results of the duplicate test sample solutions and round the results to the nearest 0.1 $\mu\text{g/mL}$ in

TABLE 1 Gold in Cyanide Solutions—Statistical Information

Solutions	Mean, Au, $\mu\text{g/mL}$	R1 (Practice E173), Au, $\mu\text{g/mL}$	R2 (Practice E173), Au, $\mu\text{g/mL}$
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

accordance with Practice E29, unless an alternative rounding method is specified by the customer or applicable material specification.

16. Precision and Bias⁵

16.1 *Precision*—An interlaboratory study was conducted to test the precision of this test method in accordance with Practice E1060 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 E173 was used to estimate the precision. The base data and statistics are documented.⁵

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

NOTE 5—The reproducibility, R2, of Practice E173 corresponds to the reproducibility index, R, of Practice E1601 and the repeatability, R1, of Practice E173 corresponds to the repeatability index, r, of Practice E1601.

16.1.1 *Repeatability*—The repeatability standard deviation (s_w) ranged from 0.01 $\mu\text{g/mL}$ to 0.12 $\mu\text{g/mL}$ 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.

16.1.2 *Reproducibility*—The reproducibility standard deviation (s_{sp}) ranged from 0.01 $\mu\text{g/mL}$ to 0.15 $\mu\text{g/mL}$ 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.

16.2 *Bias*—No information on the bias of this test method is known, because at the time of the interlaboratory study suitable reference materials were not available. The user of this method is encouraged to employ accepted reference materials, if available, to determine the presence or absence of bias.

INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY

17. Summary of Test Method

17.1 This test method describes the determination of trace gold concentrations by inductively coupled plasma—mass spectrometry (ICP-MS) based on Method D5673. Sample material in solution is introduced by pneumatic nebulization into a radiofrequency plasma where energy transfer processes cause desolvation, atomization, and ionization. The ions are

extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer. The ions transmitted through the quadrupole are detected by a continuous dynode electron multiplier assembly and the ion information processed by a data handling system. Interferences relating to the technique must be recognized and corrections applied (see Section 18 on interferences). Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents, or sample matrix. Instrumental drift and suppressions or enhancements of instrument response caused by the sample matrix must be corrected for by the use of internal standardization.

18. Interferences

18.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. These interferences can be summarized as follows:

18.1.1 *Abundance Sensitivity*—Abundance sensitivity is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

18.1.2 *Isobaric Polyatomic Ion Interferences*—Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom that have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified, and these are listed in Table 2 with the element affected. Such interferences must be recognized, and when they cannot be avoided by the selection of an alternative analytical isotope, appropriate corrections must be made to the data. Equations for the correction of data should be established at the time of the analytical run sequence as the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions.

18.1.3 *Physical Interferences*—Physical interferences are associated with the physical processes that govern the transport of the sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma—mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration solutions. Physical interferences may occur in the transfer of solution to the nebulizer (for example, viscosity effects), at the point of aerosol formation and transport to the plasma (for example, surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction, or skimmer cones, or both, reducing the effective diameter of the orifices and, therefore, ion transmission. Dissolved solids levels not exceeding 0.2 % (m/v) have been recommended to reduce such effects. Internal

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report: RR:E01-1013.

TABLE 2 Common Molecular Ion Interferences

Molecular Ion	Mass, atomic mass units (amu)	Element Interference ^A
Background Molecular Ions		
NH ⁺	15	...
OH ⁺	17	...
OH ₂ ⁺	18	...
C ₂ ⁺	24	...
CN ⁺	26	...
CO ⁺	28	...
N ₂ ⁺	28	...
N ₂ H ⁺	29	...
NO ⁺	30	...
NOH ⁺	31	...
O ₂ ⁺	32	...
O ₂ H ⁺	33	...
³⁶ ArH ⁺	37	...
³⁶ ArH ⁺	39	...
⁴⁰ ArH ⁺	41	...
CO ₂ ⁺	44	...
CO ₂ H ⁺	45	Sc
ArC ⁺ , ArO ⁺	52	Cr
ArN ⁺	54	Cr
ArNH ⁺	55	Mn
ArO ⁺	56	...
ArOH ⁺	57	...
⁴⁰ Ar ³⁶ Ar ⁺	76	Se
⁴⁰ Ar ³⁸ Ar ⁺	78	Se
⁴⁰ Ar ₂ ⁺	80	Se
¹⁸¹ Ta ¹⁶ O ⁺	197	Au
Matrix Molecular Ions		
Chloride		
³⁵ ClO ⁺	51	V
³⁵ ClOH ⁺	52	Cr
³⁷ ClO ⁺	53	Cr
³⁷ ClOH ⁺	54	Cr
Ar ³⁵ Cl ⁺	75	As
Ar ³⁷ Cl ⁺	77	Se
Sulphate		
³² SO ⁺	48	...
³² SOH ⁺	49	...
³⁴ SO ⁺	50	V, Cr
³⁴ SOH ⁺	51	V
SO ₂ ⁺ , S ₂ ⁺	64	Zn
Ar ³² S ⁺	72	...
Ar ³⁴ S ⁺	74	...
Phosphate		
PO ⁺	47	...
POH ⁺	48	...
PO ₂ ⁺	63	Cu
ArP ⁺	71	...
Group I, II Metals		
ArNa ⁺	63	Cu
ArK ⁺	79	...
ArCa ⁺	80	...
Matrix Oxides ^B		
TiO	62 to 66	Ni, Cu, Zn
ZrO	106 to 112	Ag, Cd
MoO	108 to 116	Cd

^A Elements or internal standards affected by molecular ions.

^B Oxide interferences will normally be very small and will only impact the elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the user should be aware. It is recommended that Ti and Zr isotopes be monitored if samples are likely to contain high levels of these elements. Mo is monitored as an analyte.

standardization may be effectively used to compensate for many physical interference effects. Internal standards should have similar analytical behavior to the elements being determined.

18.1.4 *Memory Interferences*—Memory interferences result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can

TABLE 3 Preparation of Gold Stock Solution^A

Element or Compound	Mass, g	Solvent
Au	0.1000	20 mL H ₂ O, add 8 mL of HCl + 5 ml HNO ₃ (1 + 1)

^A Gold stock solutions, 1.00 mL = 1000 µg of Au. Dissolve the listed masses of gold as specified in Table 3, then dilute to 100 mL with water. The metals may require heat to increase rate of dissolution. Commercially available reference solutions of known purity may be used. Alternate salts or oxides may also be used.

result from sample deposition on the sampler and skimmer cones, and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank consisting of HNO₃ (1 + 49) in water between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a solution containing elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of ten of the method detection limit should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the user should be alerted to the possibility of a memory effect and should examine the analyte concentration in the previous sample to identify if this was high. If a memory interference is suspected, the sample should be re-analyzed after a long rinse period.

19. Apparatus

19.1 *Block Digester, Hot Plate or Steam Bath*—Suitable for reducing acidified sample volume from 103 mL to less than 25 mL.

19.2 *Inductively Coupled Plasma–Mass Spectrometer*—Instrument capable of scanning the mass range of 5 amu to 250 amu with a minimum resolution capability of 1 amu peak width at 5 % peak height. The instrument may be fitted with a conventional or extended dynamic range detection system. See manufacturers' instruction manual for installation and operation.

20. Reagents

20.1 *Argon*—High purity grade (99.99 %).

20.2 *Gold Stock Solution*—Preparation procedures for the gold stock solution is listed in Table 3.

NOTE 6—Commercially prepared certified gold reference solutions in a HCl matrix are available as an alternative to dissolving gold metal.

20.3 *Gold Standard Solution*—Prepare standard solutions by combining appropriate volumes of the stock solution in volumetric flasks. Prior to preparing standard solutions, the stock solution must be analyzed separately to determine possible interferences on the other analytes or the presence of