



Designation: ~~C760 – 90 (Reapproved 2007)~~ C760 – 90 (Reapproved 2015)

Standard Test Methods for Chemical and Spectrochemical Analysis of Nuclear-Grade Silver-Indium-Cadmium Alloys¹

This standard is issued under the fixed designation C760; 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 These test methods cover procedures for the chemical and spectrochemical analysis of nuclear grade silver-indium-cadmium (Ag-In-Cd) alloys to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

Silver, Indium, and Cadmium by a Titration Method
Trace Impurities by Carrier-Distillation Spectro-
chemical Method

Sections
7 – 15
16 – 22

1.3 The values stated in SI units are to be regarded as the standard.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard and precautionary statements, see Section 5 and Practices E50.

2. Referenced Documents

2.1 *ASTM Standards:*²

C752 Specification for Nuclear-Grade Silver-Indium-Cadmium Alloy

D1193 Specification for Reagent Water

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis (Withdrawn 2002)³

2.2 *Other Document:*

NBS Circular 602⁴

3. Significance and Use

3.1 Silver-indium-cadmium alloy is used as a control material in nuclear reactors. In order to be suitable for this purpose, the material must meet the specifications for assay and impurity content. These test methods are designed to show whether or not a given material meets the specifications as given in Specification C752.

3.1.1 An assay is performed to determine whether the material has the chemical composition specified.

3.1.2 The impurity content is determined to ensure that the maximum concentration limit of impurities is not exceeded.

4. Purity of Reagents

4.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,⁵ where such specifications are

¹ These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.03 on Neutron Absorber Materials Specifications.

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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.

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

⁵ *Reagent Chemicals, American Chemical Society Specifications*, 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.

available. 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification **D1193**.

5. Hazards

5.1 Proper precautions should be taken to prevent inhalation or ingestion of heavy element (silver, indium, or cadmium) powder or dust during handling.

5.2 Workers should observe precautions as specified in vendor-supplied Material Safety Data Sheets (MSDS).

6. Sampling

6.1 Suggestions for sampling this alloy are given in Specification **C752**.

SILVER, INDIUM, AND CADMIUM BY A TITRATION METHOD

7. Scope

7.1 This test method is applicable to the determination of silver, indium, and cadmium in alloys of approximately 80 % silver, 15 % indium, and 5 % cadmium used in nuclear reactor control rod applications. The titrimetric methods presented^{6,7} will yield results with a bias of the order of 0.1 %.

8. Summary of Test Method

8.1 A weighed sample is dissolved in nitric acid and diluted to a known volume, and aliquots are removed for analysis. Silver is determined first by titrating with standardized sodium chloride solution to the potentiometric endpoint indicated by a chloride-selective ion electrode. Following the silver titration, the solution is boiled to coagulate the silver chloride. The pH is adjusted to 2.5 and the indium content is titrated with EDTA, using PAN (1-(2-pyridylazo)-2-naphthol) indicator. The pH is then raised to 6.0 and the cadmium is titrated with EDTA using the same indicator. The entire process requires approximately 20 min per aliquot, exclusive of sample weighing and dissolution.

9. Interferences

9.1 No interferences have been observed from any elements normally encountered as impurities in nuclear grade silver-indium-cadmium alloy over the concentration ranges expected.

10. Apparatus

10.1 *Burets*, precision, two, 25-mL capacity, preferably Schellbach type with TFE-fluorocarbon stopcock and automatic zero. They shall be certified or tested to conform with tolerances specified in NBS Circular 602.

10.2 *Reference Electrode*—Saturated calomel electrode.

10.3 *Glass pH Electrode*—Standard type.

10.4 *Chloride Specific Ion Electrode*.

10.5 *Expanded Scale pH/millivolt Meter*.

11. Reagents

11.1 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH₄OH).

11.2 *Buffer Solution, pH4*—0.5 M sodium acetate—0.5 M acetic acid.

11.3 *Cadmium (Cd)*—Metal, >99.99 % pure.

11.4 *Ethylenediaminetetraacetate Dihydrate Disodium Salt (EDTA) Solution (0.01000 M)*—Weigh 3.722 ± 0.001 g of EDTA into a small plastic beaker. Dissolve with water, transfer quantitatively to a 1-L volumetric flask, and make up to volume with water. Transfer the solution to a plastic storage bottle. Do not allow the EDTA solution to stand in contact with glass containers.

11.5 *Indium (In)*—Metal, >99.99 % pure.

11.6 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO₃).

11.7 *PAN Indicator Solution (0.1 % PAN in Methanol)*—Dissolve 100 mg of 1-(2-pyridylazo)-2-naphthol in 100 mL of methyl alcohol and mix until completely dissolved.

⁶ Cheng, K. L., "Complexometric Titration of Indium," *Analytical Chemistry*, Vol 27, 1955, p. 1582.

⁷ Cheng, K. L., "Complexometric Titration of Copper and Other Metals in a Mixture," *Analytical Chemistry*, Vol 30, 1958, p. 243.

11.8 *Silver (Ag)*—Metal, >99.99 % pure.

11.9 *Sodium Chloride (NaCl)*.

11.10 *Sodium Chloride Solution (0.0500 M)*—Dry sodium chloride (NaCl) at 120°C, in a weighing bottle, to a constant weight and cool to room temperature in a desiccator. Weigh 2.922 ± 0.001 g of the dried NaCl into a small plastic beaker. Dissolve in water, quantitatively transfer to a 1-L volumetric flask, and make up to volume with water.

12. Standardization

12.1 *Silver-Indium-Cadmium Calibration Standard:*

12.1.1 Clean approximately 8.0 g of silver metal, 1.5 g of indium metal, and 0.5 g of cadmium metal with an organic solvent and air dry.

12.1.2 Weigh each metal accurately and transfer to a 100-mL beaker.

12.1.3 Add sufficient water to cover the metal pieces and add HNO₃ (sp gr 1.42) dropwise until dissolution is complete.

12.1.4 Transfer quantitatively to a 100-mL volumetric flask and dilute to volume with water.

12.2 *Calibration of NaCl and EDTA Titrants:*

12.2.1 Pipet 10 mL of the calibration standard into a 100-mL volumetric flask and dilute to volume with water. (Retain this solution as a working standard.)

12.2.2 Pipet 10 mL of the diluted standard into a 100-mL beaker and adjust the volume to about 25 mL with water.

12.2.3 Adjust the pH to approximately 1 using NH₄OH (sp gr 0.90).

12.2.4 Place a TFE-fluorocarbon-coated stirring bar in the solution and insert the chloride specific ion electrode and the reference electrode.

12.2.5 Stir at a moderate rate and titrate the silver with NaCl solution. Record millivolt readings versus volume added. Allow sufficient time for equilibrium readings to be attained.

12.2.6 The titration end point is taken as the termination of the rapidly rising segment of the millivolt versus volume titration curve.

12.2.7 Adjust to pH 2.5 ± 0.2 by dropwise addition of acetate buffer solution (pH4).

12.2.8 Remove the electrodes and rinse thoroughly to avoid loss of indium and cadmium.

12.2.9 Heat the solution to boiling on a hotplate until the supernatant liquid is clear. Allow to cool.

12.2.10 Add 4 drops of PAN indicator solution. The solution should be deep purple.

12.2.11 Titrate the indium with standard EDTA solution to the sharp transition from purple to yellow. The volume used corresponds to the indium content.

12.2.12 Adjust to pH 6 ± 0.2 with NH₄OH (sp gr 0.90). The color of the solution will change back to purple.

12.2.13 Titrate the purple solution with standard EDTA until the color again changes to yellow. The volume used corresponds to the cadmium content.

<https://standards.iteh.ai/catalog/standards/sist/35bea769-073f-44e4-a6b5-855b73da3b0b/astm-c760-902015>

13. Procedure

13.1 Clean approximately 1.0 g of the sample with an organic solvent and air dry.

13.2 Weigh the cleaned sample accurately and transfer it to a 100-mL beaker.

13.3 Cover the sample with water and add HNO₃ (sp gr 1.42) dropwise until the sample is completely dissolved.

13.4 Transfer the solution quantitatively to a 100-mL volumetric flask and dilute to volume with water.

13.5 Proceed with the determination of silver, indium, and cadmium as described in 12.2.2 – 12.2.13.

14. Calculation

14.1 *Symbols:*

S = sample weight, mg,

$D.F.$ = dilution factor = 0.1,

F_S = calibration factor for silver, mg Ag/mL of titrant,

F_I = calibration factor for indium, mg In/mL of titrant, and

F_C = calibration factor for cadmium, mg Cd/mL of titrant.

14.2 *Calibration Calculations:*

$$F_S = \frac{\text{mg of Ag in calibration standard aliquot}}{\text{mL of standard NaCl solution added}} \quad (1)$$

$$F_I = \frac{\text{mg of In in calibration standard aliquot}}{\text{mL of standard EDTA solution added}} \quad (2)$$