Designation: C 760 – 90 (Reapproved 1996)

Standard Test Methods for Chemical and Spectrochemical Analysis of Nuclear-Grade Silver-Indium-Cadmium Alloys[1]

This standard is issued under the fixed designation C 760; 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:

	Sections
Silver, Indium, and Cadmium by a Titration Method	7-15
Trace Impurities by Carrier-Distillation Spectrochemical Method	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 E 50.

2. Referenced Documents

2.1 *ASTM Standards:*

C 752 Specification for Nuclear-Grade Silver-IndiumCadmium Alloy[2]

D 1193 Specification for Reagent Water[3]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[4]

E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis[4]

2.2 *Other Document:*

NBS Circular 602[5]

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 C 752.

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,[6] where such specifications are 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 D 1193.

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 C 752.

SILVER, INDIUM, AND CADMIUM BY A TITRATION METHOD

7. Scope

7.1 This test method is applicable to the determination of

[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 Neuron Absorber Materials.

Current edition approved May 25, 1990. Published July 1990. Originally published as C 760 – 71. Last previous edition C 760 – 79.

[2] *Annual Book of ASTM Standards*, Vol 12.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 03.05.

[5] Available from National Institute of Standards and Technology, Gaithersburg, MD 20899.

[6] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc. 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. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

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[7,8] 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 (NH_4OH).

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 (HNO_3).

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.

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 HNO_3(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 NH_4OH (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 NH_4OH (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.

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.

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

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