Designation: C1517 – 16

Standard Test Method for Determination of Metallic Impurities in Uranium Metal or Compounds by DC-Arc Emission Spectroscopy[1]

This standard is issued under the fixed designation C1517; 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 describes the steps necessary for the preparation and determination of impurity metals in uranium metal and uranium compounds by DC arc emission spectroscopy.

1.2 The method is valid for those materials that can be dissolved in acid or converted to an oxide in a muffle furnace, or both (see Practice C1347).

1.3 This method uses the carrier distillation technique to selectively carry the impurities into the arc, leaving the uranium oxide in the electrode. If it is necessary to determine the carrier metal (usually a silver or strontium, or gallium compound) as an impurity, another technique must be chosen for that element.

1.4 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.5 *This standard may involve hazardous materials, operations and equipment. 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride

C776 Specification for Sintered Uranium Dioxide Pellets

C788 Specification for Nuclear-Grade Uranyl Nitrate Solution or Crystals

C859 Terminology Relating to Nuclear Materials

C967 Specification for Uranium Ore Concentrate

C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis

E130 Practice for Designation of Shapes and Sizes of Graphite Electrodes (Withdrawn 2013)[3]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminologies C859 and E135.

4. Summary of Test Method

4.1 Uranium metal, solutions and compounds are converted to uranium oxide (U_3O_8) in a muffle furnace. A weighed amount of the oxide is mixed with an appropriate spectrographic carrier and loaded into a graphite electrode. The electrode is excited in a DC arc and the light is dispersed by a spectrograph or spectrometer. The resulting spectrum is measured electronically using a CCD, CID, or CMOS camera sensitive to the proper regions. The line intensities are compared directly to calibration curves derived from the arced standards.

5. Significance and Use

5.1 This test method is applicable to uranium metal, uranium oxides and compounds soluble in nitric or sulfuric acid, and uranium solutions which can be converted to uranium oxide (U_3O_8) in a muffle furnace. It may be used to determine the impurities in uranium compounds as listed in Specifications C753, C776, C788, and C967.

6. Apparatus

6.1 *Spectrograph or Spectrometer*—A spectrograph with sufficient resolving power and linear dispersion to separate the

[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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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.

analytical lines from other lines in the spectrum of the sample in the spectral region of 230 to 855 nm is required. Some spectrographs may be able to access wavelengths lower than 230 nm that may allow the determination of other analytes. Instruments with a reciprocal linear dispersion in the first order of 0.5 nm/mm or less are satisfactory. The spectrometer should include a CCD, CID, or CMOS camera for electronic measurement of the resulting spectrum.

6.2 *Excitation Source*—Use an arc power source capable of providing a dc arc of up to 14-A dc, depending on the carrier used and electrode design.

6.3 *Excitation Stand*—Conventional type with adjustable water-cooled electrode holders (may be fitted with automatic sample changers if desired).

6.4 *Mixer,* for dry materials.

6.5 *Platinum Crucible.*

6.6 *Venting Tool,* (see Fig. 8, Test Methods C761 – 01).

6.7 *Muffle Furnace,* 1000°C capability.

7. Reagents and Materials

7.1 *Purity of Materials*—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 of Analytical Reagents of the American Chemical Society[4] 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.

7.2 *Electrodes*—The anode and counter electrodes should be of the S-2, S 16 and C-1 types as given in Practice E130 (or equivalent).

NOTE 1—Exact shapes and dimensions of the electrodes are not as critical as given in Practice E130; however, dimensions of the electrodes used should be consistent and it is essential that the same dimension electrodes be used for standards and samples.

7.3 *Nitric Acid (HNO_3)*—concentrated (70 %), electronic grade, or equivalent.

7.4 *Sulfuric Acid (H_2SO_4)*—concentrated, electronic grade, or equivalent.

7.5 *Spectrographic Carrier*—The following spectrographic carriers have been used successfully for DC Arc analysis:

7.5.1 *Silver Chloride-Lithium Fluoride,* 11:1 w/w ratio.

7.5.2 *Silver Chloride-Silver Fluoride,* 4:1 w/w ratio.

7.5.3 *Gallium Oxide,* 99.99 % or better.

7.5.4 *Silver Chloride-Strontium Fluoride,* 16.4 mol % SrF_2 in AgCl.

7.5.5 *Gallium Oxide-Lithium Fluoride,* 11:1 w/w ratio.

7.6 *Mixing Vial,* plastic, 12.7 mm (1/2 in.) by 25.4 mm (1 in.) with cap and 10 mm (3/8 in.) plastic mixing bead.

[4] *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.

TABLE 1 Carrier—Sample Combinations

Carrier Material	Carrier Wt, (mg)	Oxide Wt, (mg)	Electrode Charge, (mg)	Mixing Time, (s)
AgCl/AgF (4:1)	50	250	50	180
AgCl/LiF (11:1)	30	270	100	60
AgCl/SrF_2 (~6:1)	50	450	100	30
Ga_2O_3/LiF (11:1)	20	380	100	25
Ga_2O_3[A]	7	343	100	60

[A] For the determination of Ag and Li only.

NOTE 1—The above listed combinations of carrier, uranium oxide and electrode charge have been successfully used for the determination of impurities in uranium oxide. Other combinations may be available or suitable. However, the user must demonstrate comparable precision and bias.

7.7 *Standard Uranium Oxide (U_3O_8) Diluent*—Use NBL CRM 129[3] (or its replacement or equivalent) of known impurity level as a diluent.

8. Precautions

8.1 Consult manufacturer's Safety Data Sheets (SDS) for chemical incompatibilities, specific hazards, or spill cleanup for any hazardous materials used in this method.

8.2 All mixing and weighing operations involving uranium oxides should be carried out in properly functioning hoods or gloveboxes.

9. Standardization and Calibration

9.1 *Standards:*

9.1.1 Standards may be synthesized by adding the impurity elements to purified U_3O_8 (NBL CRM 129-A[5], or equivalent) and homogenizing. Impurities in powder form, preferably as oxides, may be blended in U_3O_8; impurities in solution may be added to U_3O_8 and the mixture dried, blended and reignited, or the impurities and uranium may be combined in solution and reconverted to U_3O_8. The individual elements should grade in such a ratio as to facilitate visual comparisons covering the desired analytical range for each.

9.1.2 The compounds used to make U_3O_8 impurity standards should be of the highest purity available.

9.1.3 Alternatively, commercially available uranium impurity standards, such as NBL CRM 123[5] and 124[5] series standards, may be used. (Other standards may be available; the user should determine quality or applicability, or both, prior to use.) These may be supplemented by synthetic standards to extend calibration ranges, if necessary.

9.1.4 For each standard used, prepare in the same ratio of uranium oxide to carrier as for samples (see Table 1 for further details).

9.1.5 Charge the electrode and arc at the same conditions as determined to be optimum for the instrument in use.

9.2 *Calibration Curves:*

[5] Available from the US Department of Energy, New Brunswick Laboratory, Building 350, 9800 South Cass Avenue, Argonne, IL 60439, ATTN: Reference Material Sales. http://science.energy.gov/nbl/.