Designation: C799 – 19

Standard Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions[1]

This standard is issued under the fixed designation C799; 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, mass spectrometric, spectrochemical, nuclear, and radiochemical analysis of nuclear-grade uranyl nitrate solution to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

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[A] Discontinued July 2019.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 6.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets

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

C788 Specification for Nuclear-Grade Uranyl Nitrate Solution or Crystals

C859 Terminology Relating to Nuclear Materials

C1219 Test Methods for Arsenic in Uranium Hexafluoride (Withdrawn 2015)[3]

C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials

C1254 Test Method for Determination of Uranium in Mineral Acids by X-Ray Fluorescence

C1267 Test Method for Uranium by Iron (II) Reduction in Phosphoric Acid Followed by Chromium (VI) Titration in the Presence of Vanadium

C1287 Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively

[1] These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are 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.

Coupled Plasma Mass Spectrometry
C1295 Test Method for Gamma Energy Emission from Fission and Decay Products in Uranium Hexafluoride and Uranyl Nitrate Solution
C1296 Test Method for Determination of Sulfur in Uranium Oxides and Uranyl Nitrate Solutions by X-Ray Fluorescence (XRF) (Withdrawn 2007)[3]
C1380 Test Method for the Determination of Uranium Content and Isotopic Composition by Isotope Dilution Mass Spectrometry (Withdrawn 2018)[3]
C1413 Test Method for Isotopic Analysis of Hydrolyzed Uranium Hexafluoride and Uranyl Nitrate Solutions by Thermal Ionization Mass Spectrometry
C1517 Test Method for Determination of Metallic Impurities in Uranium Metal or Compounds by DC-Arc Emission Spectroscopy
C1561 Guide for Determination of Plutonium and Neptunium in Uranium Hexafluoride and U-Rich Matrix by Alpha Spectrometry
C1871 Test Method for Determination of Uranium Isotopic Composition by the Double Spike Method Using a Thermal Ionization Mass Spectrometer
D1193 Specification for Reagent Water
E12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases (Withdrawn 1996)[3]
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
E115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis (Withdrawn 2002)[3]

2.2 *American Chemical Society Specification:*
Reagent Chemicals[4]

2.3 *Other Documents:*
ISO 7097 Determination of Uranium in Uranium Product Solutions and Solids with Cerium IV Oxidation Titrimetric Method[5]

3. Terminology

3.1 For definitions of terms used in this test method but not defined herein, refer to Terminology C859.

4. Significance and Use

4.1 Uranyl nitrate solution is used as a feed material for conversion to the hexafluoride as well as for direct conversion to the oxide. In order to be suitable for this purpose, the material must meet certain criteria for uranium content, isotopic composition, acidity, radioactivity, and impurity content. These methods are designed to show whether a given material meets the specifications for these items described in Specification C788.

4.1.1 An assay is performed to determine whether the material has the specified uranium content.

4.1.2 Determination of the isotopic content of the uranium is made to establish whether the effective fissile content is in accordance with the purchaser's specifications.

4.1.3 Acidity, organic content, and alpha, beta, and gamma activity are measured to establish that they do not exceed their maximum limits.

4.1.4 Impurity content is determined to ensure that the maximum concentration limit of certain impurity elements is not exceeded. Impurity concentrations are also required for calculation of the equivalent boron content (EBC), and the total equivalent boron content (TEBC).

5. Reagents

5.1 *Purity of Reagents*—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, 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.

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

5.3 Hydrofluoric acid (used in some of the procedures) is a highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left untreated. Familiarization and compliance with the Safety Data Sheet is essential.

6. Safety Precautions

6.1 Use of this standard does not relieve the user of the obligation to be aware of and to conform to all health and safety requirements.

6.2 The user should also be cognizant of and adhere to all federal, state, and local regulations for processing, shipping, or in any way using uranyl nitrate solutions.

7. Sampling

7.1 Criteria for sampling this material are given in Specification C788.

DETERMINATION OF URANIUM

8. Scope

8.1 Uranium can be determined using iron (II) reduction and dichromate titration. Test Method C1267 can be used.

8.2 Uranium can also be determined using cerium (IV) oxidation titrimetry. ISO 7097 Test Method can be used.

8.3 Uranium can also be determined by X-Ray Fluorescence using Test Method C1254.

[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.

[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

8.4 Previous sections have been deleted.

URANIUM BY IGNITION GRAVIMETRY

9. Scope

9.1 This test method covers the determination of uranium in nuclear-grade uranyl nitrate solution. Appropriate size sample aliquots are chosen to obtain 5 to 10 g of U_3O_8.

10. Summary of Test Method

10.1 The uranyl nitrate solution is evaporated to dryness, ignited to U_3O_8, and weighed. Corrections are made for any impurities present (**1**, **2**).

11. Interferences

11.1 The weight of U_3O_8 is corrected for the nonvolatile impurities present as determined by spectrographic analysis.

11.2 Volatile anions that are difficult to decompose require an extended ignition period.

12. Apparatus

12.1 *Heat Lamp,* infrared.

12.2 *Hot Plate.*

12.3 *Muffle Furnace.*

13. Procedure

13.1 Transfer a weighed portion of uranyl nitrate solution containing 5 to 10 g of uranium into a preweighed platinum dish and add 2 drops of HF (48 %).

13.2 Position the dish under the heat lamp and evaporate the solution to dryness.

13.3 Place the dish on a hot plate with a surface temperature of about 300°C and heat until most of the nitrate has decomposed.

13.4 Transfer the dish to a muffle furnace and ignite for 2 h at 900°C.

13.5 Remove the dish to a desiccator and allow to cool to room temperature.

13.6 Weigh the dish; then repeat 13.4 – 13.6 until a constant weight is obtained.

14. Calculation

14.1 Calculate the uranium content as follows:

$$\text{Uranium, g/g} = ((B - C)/A)\,D \qquad (1)$$

where:

A = sample, g,
B = U_3O_8 obtained, g,
C = impurity-element oxides, g, and
D = gravimetric factor, grams of uranium/grams of U_3O_8 (varies according to uranium enrichment).

15. Precision

15.1 The limit of error at the 95 % confidence level for a single determination is ±0.03 %.

SPECIFIC GRAVITY BY PYCNOMETRY

16. Scope

16.1 This test method covers the determination of the specific gravity of a solution of uranyl nitrate to ±0.0004.

17. Summary of Test Method

17.1 A known volume of the solution adjusted at a controlled temperature is weighed and compared to the weight of water measured in the same container (Terminology E12).

18. Apparatus

18.1 *Volumetric Flasks,* 50-mL, Class A.

18.2 *Water Bath,* temperature controlled to ±0.1°C at a temperature slightly above normal room temperature, and provided with clips for holding volumetric flasks.

19. Procedure

19.1 Weigh the clean, dry volumetric flask and its stopper to the nearest 0.1 mg.

19.2 Fill the volumetric flask with the uranyl nitrate solution to a point close to the volume mark, using a thin-stemmed funnel and a glass dropper.

19.3 Place the stoppered volumetric flask in the water bath for 30 min.

19.4 Use a finely drawn glass dropper to adjust the liquid volume to the mark.

19.5 Leave the flask in the water bath an additional 10 min to make sure that the bath temperature has been reached.

19.6 Dry and weigh the flask to the nearest 0.1 mg.

19.7 Repeat 19.2 – 19.6 using boiled and cooled distilled water instead of the uranyl nitrate solution.

20. Calculation

20.1 Very accurate determinations of specific gravity require that vacuo corrections be made, but if a median correction figure in terms of grams per grams of sample is applied to the solution weights in all cases the resulting error will not exceed 0.05 %.

$$\text{Sp gr} = \frac{B - A + 0.0007\,(B - A)}{C - A + 0.0010\,(C - A)} \qquad (2)$$

where:

B = sample plus flask in air, g,
A = flask in air, g,
C = water plus flask in air, g,
0.0007 g/g = correction factor applicable for densities of 1.3 to 1.5, and
0.0010 g/g = correction factor for water.

21. Precision

21.1 The limit of error at the 95 % level for a single determination is ±0.03 %.