



Designation: C 1636 – 06a

Standard Guide for the Determination of Uranium-232 in Uranium Hexafluoride¹

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1. Scope

1.1 This method covers the determination of ^{232}U in uranium hexafluoride by alpha spectrometry.

1.2 *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 to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

C 787 Specification for Uranium Hexafluoride for Enrichment

C 996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ^{235}U

C 1163 Practice for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride

C 1284 Practice for Electrodeposition of the Actinides for Alpha Spectrometry

D 1193 Specification for Reagent Water

D 3084 Practice for Alpha-Particle Spectrometry of Water

D 3648 Practices for the Measurement of Radioactivity

2.2 Other Standards

DIN 25711 Determination of the ^{232}U isotopic content in uranium containing nuclear fuel solutions by α spectrometry.³

3. Terminology

3.1 Definitions:

3.1.1 *region-of-interest (ROI)*—the channels, or region, in the alpha spectra in which the counts due to a specific radioisotope appear on a functioning calibrated alpha spectrometry system.

¹ This guide 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 Deutsches Institut für Normung e.V., Berlin, Germany (www.din.de).

3.1.2 *Reagent blank*—DI water processed the same as the samples; used in the determination of the minimum detectable activity.

4. Summary of Guide

4.1 An aliquot of hydrolyzed uranium hexafluoride equivalent to 60 micrograms of uranium is converted to a nitric acid system and the uranium is extracted onto a solid phase extraction column. The daughters of uranium decay products are rinsed from the column and the uranium is then selectively eluted. The uranium is reduced and then coprecipitated with neodymium fluoride. Test Method **C 1163** provides further information on the use of neodymium fluoride to prepare actinide mounts for alpha spectrometry. The sample is then counted by alpha spectrometry, and the ^{232}U is calculated based on the observed activities of the uranium isotopes in the alpha spectra.

4.2 While this guide does not present details on electrodeposition as an alternative to neodymium fluoride precipitation for the preparation of a mount for alpha spectrometry Practice **C 1284** does present details on that option.

4.3 Alternate separation chemistry approaches may be found in the literature. It is the responsibility of the user of such alternative separation approaches to validate their effectiveness, especially the removal of potentially interfering thorium isotopes (section 6.1).

5. Significance and Use

5.1 The method is applicable to the analysis of materials to demonstrate compliance with the specifications set forth in Specifications **C 787** and **C 996**.

6. Interferences

6.1 Incomplete removal of ^{228}Th could possibly interfere with the ^{232}U determination. Method **DIN 25711** addresses the potential capability for this method to eliminate this potential interference.

6.2 Since only the relative amount of ^{232}U , relative to total uranium, is being determined in this method there is no impact to chemical loss in the separation or sample mounting chemistry. Therefore, unlike most alpha spectrometry methods, no yield tracer is necessary or useful.

6.3 The alpha emission energies of ^{235}U and ^{236}U are relatively close. Thus there is the potential for overlap of counts from one isotope into the ROI of the other. Where the alpha spectrometry system (section 7.1) provides spectral de-convolution algorithms may be used in the analysis of the spectra. Such de-convolution may allow for minimization of any possible bias in the reported results. However, it should be noted that these two isotopes typically account for a relatively small amount of the overall uranium mass. So any bias between the two should result in a relatively small overall bias in the reported ^{232}U result.

7. Apparatus

7.1 Alpha spectrometry system. See practices **D 3084** and **D 3648** for a description of the apparatus.

7.1.1 A ROI for each uranium isotope (^{232}U , ^{234}U , ^{235}U , ^{236}U , and ^{238}U) will need to be defined for the alpha spectrometry system being used. Based on these defined ROIs the fractional abundance of alpha decays within the energy range of the ROI for each isotope (AB_i in section 12.1) must be determined.

7.2 Ion Exchange Columns, able to hold a 10 mL resin bed and 15 mL solution washes.

7.3 Filter Paper, 0.1 μm pore size, 25-mm diameter, and compatible with HF.⁴

7.4 *Vacuum Funnel*—Polysulfone twist-lock with stainless steel screen for filter mounting.

8. Reagents and Materials

8.1 *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. Other grades of reagents 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

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined in Specification **D 1193**.

8.3 *Ammonium oxalate (0.1M)*—Dissolve 14.2 g $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in approximately 500 mL water and dilute to 1 litre.

8.4 *Ethanol*—Ethyl alcohol, absolute (200 proof), denatured.

8.5 *Hydrochloric acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

8.6 *Hydrochloric acid (9M)*—Add 750 mL concentrated HCl to 100 mL water and dilute to 1 litre.

8.7 *Hydrochloric acid (1.5M)*—Add 125 mL concentrated HCl to 500 mL water and dilute to 1 litre.

8.8 *Hydrochloric acid (1M)*—Add 83 mL concentrated HCl to 500 mL water and dilute to 1 litre.

⁴ Pall Life Sciences (formerly Gelman) Metrical filter has been found to be acceptable. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁵ “Reagent Chemicals, American Chemical Society Specifications,” American Chemical Society, Washington, DC.

8.9 *Hydrofluoric acid (minimum 48 % assay)*—Concentrated HF, reagent grade.

Warning—Severe burns can result from exposure of skin to concentrated hydrofluoric acid.

8.10 *Neodymium chloride (10 mg Nd/mL)*—Heat 25 mL of concentrated hydrochloric acid and 1.17 g of neodymium oxide on a hotplate until the neodymium oxide is in solution. Cool the solution and dilute to 100 mL with water.

8.11 *Neodymium chloride (100 μg Nd/mL)*—Dilute 1 mL of 10 mg Nd/mL solution to 100 mL with water.

8.12 Neodymium oxide (Nd_2O_3).

8.13 *Nitric acid (sp gr 1.42)*—Concentrated nitric acid (HNO_3).

8.14 *Nitric acid (3M)*—Add 188 mL concentrated nitric acid to 500 mL water and dilute to 1 litre.

8.15 *Oxalic acid in 1M HCl (0.1M)*—Dissolve 12.6 g $\text{H}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in 500 mL 1M HCl and dilute to 1 litre with 1M HCl.

8.16 *20 % Titanium Trichloride (TiCl_3) aqueous solution*—available as a 20 % (w/v) solution of titanium trichloride from commercial suppliers.

8.17 *Extraction Chromatography Resin*, containing octylphenyl-N,N-di-isobutyl carbamoylphosphine oxide (CMPO) dissolved in tri-n-butyl phosphate (TBP) as the immobilized extractant.⁶

9. Hazards

9.1 Adequate laboratory facilities, such as fume hoods and controlled ventilation, along with safe techniques must be used in this procedure. Extreme care should be exercised in using hydrofluoric acid and other hot, concentrated acids. Use of rubber gloves is recommended.

9.2 Hydrofluoric acid should be used with care by persons familiar with its hazards through review of the Material Safety Data Sheet (MSDS) and who are properly equipped to respond to cases of skin contact as suggested in the MSDS.

10. Calibration and Standardization

10.1 The alpha spectrometry units should be calibrated for energy, resolution and efficiency according to the manufacturer's instructions. The background counting rate for the instrument should be measured at a frequency determined by the user. See Practices **D 3084** and **D 3648** for additional information.

⁶ TRU resin from Eichrom Technologies Inc., Darien IL, USA, has been found to be acceptable. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

Horwitz, E. P., Chiarizia, R., Dietz, M. L., Diamond, H., and Nelson, D., “Separation and Preconcentration of Actinides from Acidic Media by Extraction Chromatography,” *Analytica Chimica Acta*, 281, 1993, pp. 361-372.

The Eichrom Technologies TRU resin is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.