



Standard Practice for The Ion Exchange Separation of Uranium and Plutonium Prior to Isotopic Analysis¹

This standard is issued under the fixed designation C 1411; 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 practice is for the ion exchange separation of uranium and plutonium from each other and from other impurities for subsequent isotopic analysis by thermal ionization mass spectrometry. Plutonium-238 and uranium-238, and plutonium-241 and americium-241, will appear as the same mass peak and must be chemically separated prior to analysis. Only high purity solutions can be analyzed reliably using thermal ionization mass spectrometry.

1.2 *This standard may involve hazardous material, 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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

C 698 Standard Test Method for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Mixed Oxides ((U, Pu)O₂), Sections 141-149.²

C 833 Standard Specification for Sintered (Uranium, Plutonium) Dioxide Pellets.²

C 1008 Standard Specification for Sintered (Uranium, Plutonium) Dioxide Pellets - Fast Reactor Fuel.²

C 1168 Standard Practice for Preparation and Dissolution of Plutonium Materials for Analysis.²

E 267 Standard Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances.³

3. Summary of Practice

3.1 Solid samples are dissolved according to Practice C 1168 or other appropriate methods. The resulting solution is processed by this practice to prepare separate solutions of plutonium and uranium for mass spectrometric isotopic analysis using Method C 698, see Sec. 144 through 145 and 147.4 through 149 or its replacement. Appropriate aliquants are taken

to provide up to 1 mg of plutonium on the ion exchange column to be separated from 10 mg or less of uranium. Valence adjustment is attained by using one of two procedures.

3.1.1 For any sample type, especially those containing large amounts of impurities, ferrous sulfate may be used for reduction. The aliquant is dissolved in 3 M HNO₃. Ferrous sulfate is added to reduce all plutonium (VI) to plutonium (III), then 16 M HNO₃ is added to oxidize plutonium (III) to plutonium (IV), and to adjust the final acid concentration to 8 M HNO₃.

3.1.2 A hydrogen peroxide reduction may be used for relatively pure samples which do not contain excessive amounts of oxidizing impurities. The aliquant is dissolved in 8 M HNO₃. Hydrogen peroxide is added to the aliquant prior to fuming to reduce plutonium (VI) to the lower oxidation states. The solution is warmed on a hot plate to destroy excess hydrogen peroxide and stabilize plutonium (IV) in solution.^{4,5}

3.2 After valence adjustment, the resulting solution is passed through an anion exchange column in the nitrate form which retains the plutonium; uranium and americium are not absorbed. The adsorbed plutonium is washed with additional 8 M nitric acid (HNO₃) to remove impurities and then stripped from the column with 0.36 M hydrochloric acid (HCl) and 0.01 M hydrofluoric acid (HF). The effluent containing the uranium and americium is converted to a HCl medium, and this solution is passed through an anion exchange column in the chloride form which retains the uranium. The adsorbed uranium is washed with additional concentrated HCl to remove the impurities and then stripped from the column with 0.1 M HCl.

4. Significance and Use

4.1 Uranium and plutonium are used in nuclear reactor fuel and must be analyzed to insure that they meet certain criteria for isotopic composition as described in Specification C 833 and Specification C 1008. This standard practice is used to chemically separate the same mass peak interferences from uranium and plutonium and from other impurities prior to isotopic abundance determination by thermal ionization mass spectrometry.

4.2 In those facilities where perchloric acid use is tolerated, the ion exchange separation procedure in Test Method E 267 may be used prior to isotopic abundance determination. Also,

¹ This practice is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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² Annual Book of ASTM Standards, Vol 12.01.

³ Annual Book of ASTM Standards, Vol 12.02.

⁴ I.V. Kressin and G.R. Waterbury, *Anal. Chem.* 34(12), 1598 (1962).

⁵ C.E. Pietri, B.P. Freeman, and J.R. Weiss, *DOE/NBL-298*, September 1981.