



Standard Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Mixed Oxides ((U, Pu)O₂)¹

This standard is issued under the fixed designation C698; 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, and spectrochemical analysis of nuclear-grade mixed oxides, (U, Pu)O₂, powders and pellets to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

	Sections
Uranium in the Presence of Pu by Potentiometric Titration	2
Plutonium by Controlled-Potential Coulometry	2
Plutonium by Amperometric Titration with Iron (II)	2
Nitrogen by Distillation Spectrophotometry Using Nessler Reagent	8 to 15
Carbon (Total) by Direct Combustion-Thermal Conductivity	16 to 26
Total Chlorine and Fluorine by Pyrohydrolysis	27 to 34
Sulfur by Distillation-Spectrophotometry	35 to 43
Moisture by the Coulometric, Electrolytic Moisture Analyzer	44 to 51
Isotopic Composition by Mass Spectrometry	3
Rare Earths by Copper Spark Spectroscopy	52 to 59
Trace Impurities by Carrier Distillation Spectroscopy	60 to 68
Impurities by Spark-Source Mass Spectrography	69 to 75
Total Gas in Reactor-Grade Mixed Dioxide Pellets	4
Tungsten by Dithiol-Spectrophotometry	76 to 84
Rare Earth Elements by Spectroscopy	85 to 88
Plutonium-238 Isotopic Abundance by Alpha Spectrometry	5
Americium-241 in Plutonium by Gamma-Ray Spectrometry	
Uranium and Plutonium Isotopic Analysis by Mass Spectrometry	89 to 97
Oxygen-to-Metal Atom Ratio by Gravimetry	98 to 105

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

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 safety precaution statements, see Sections 6, 13.2.5, 41.7, and 93.6.1.)

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 Discontinued as of November 15, 1992.

³ Discontinued as of May 30, 1980.

⁴ Discontinued as of June 2016.

⁵ Discontinued as of January 1, 2004.

2. Referenced Documents

2.1 ASTM Standards:⁶

C697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets

C852 Guide for Design Criteria for Plutonium Gloveboxes

C859 Terminology Relating to Nuclear Materials

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C1108 Test Method for Plutonium by Controlled-Potential Coulometry

C1165 Test Method for Determining Plutonium by Controlled-Potential Coulometry in H₂SO₄ at a Platinum Working Electrode

C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

C1204 Test Method for Uranium in Presence of Plutonium by Iron(II) Reduction in Phosphoric Acid Followed by Chromium(VI) Titration

C1206 Test Method for Plutonium by Iron (II)/Chromium (VI) Amperometric Titration (Withdrawn 2015)⁷

C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials

C1268 Test Method for Quantitative Determination of ²⁴¹Am in Plutonium by Gamma-Ray Spectrometry

C1415 Test Method for ²³⁸Pu Isotopic Abundance By Alpha Spectrometry

C1432 Test Method for Determination of Impurities in Plutonium: Acid Dissolution, Ion Exchange Matrix Separation, and Inductively Coupled Plasma-Atomic Emission Spectroscopic (ICP/AES) Analysis

C1625 Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances by Thermal Ionization Mass Spectrometry

⁶ 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.

⁷ The last approved version of this historical standard is referenced on www.astm.org.