



Standard Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets¹

This standard is issued under the fixed designation C 697; 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 plutonium dioxide powders and pellets to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

	Sections
Plutonium Sample Handling	8 to 10
Plutonium by Controlled-Potential Coulometry	2
Plutonium by Ceric Sulfate Titration	3
Plutonium by Amperometric Titration with Iron(II)	2
Plutonium by Diode Array Spectrophotometry	3
Nitrogen by Distillation Spectrophotometry Using Nessler Reagent	11 to 18
Carbon (Total) by Direct Combustion–Thermal Conductivity	19 to 30
Total Chlorine and Fluorine by Pyrohydrolysis	31 to 38
Sulfur by Distillation Spectrophotometry	39 to 47
Plutonium Isotopic Analysis by Mass Spectrometry	4
Rare Earth Elements by Spectroscopy	48 to 55
Trace Elements by Carrier–Distillation Spectroscopy	56 to 63
Impurities by ICP-AES	
Impurity Elements by Spark-Source Mass Spectrography	64 to 70
Moisture by the Coulometric Electrolytic Moisture Analyzer	71 to 78
Total Gas in Reactor-Grade Plutonium Dioxide Pellets	79 to 86
Plutonium-238 Isotopic Abundance by Alpha Spectrometry	3
Americium-241 in Plutonium by Gamma-Ray Spectrometry	2
Rare Earths By Copper Spark-Spectroscopy	87 to 96
Plutonium Isotopic Analysis by Mass Spectrometry	97 to 105
Oxygen-To-Metal Atom Ratio by Gravimetry	106 to 114

1.3 *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 precautionary statements, see Sections 6, 15, 24, 111, and 52.9 and 101.5.1 .*

¹ These 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 xxxx, 2003.

⁴ Discontinued as of May 30, 1980.

2. Referenced Documents

2.1 ASTM Standards:⁵

- C 757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable
- C 852 Guide for Design Criteria for Plutonium Gloveboxes
- C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry
- C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry
- C 1108 Test Method for Plutonium by Controlled-Potential Coulometry
- C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials
- C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials
- C 1165 Test Method for Determining Plutonium by Controlled-Potential Coulometry in H₂SO₄ at a Platinum Working Electrode
- C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis
- C 1206 Test Method for Plutonium by Iron (II)/Chromium (VI) Amperometric Titration
- C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry
- C 1235 Test Method for Plutonium by Titanium(III)/Cerium(IV) Titration
- C 1268 Test Method for Quantitative Determination of Americium 241 in Plutonium by Gamma-Ray Spectrometry
- C 1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials

⁵ 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.

