



Designation: C 697 – 98

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 Ceriic Sulfate Titration	11 to 18
Plutonium by Amperometric Titration with Iron(II)	²
Plutonium by Diode Array Spectrophotometry	³
Nitrogen by Distillation Spectrophotometry Using Nessler Reagent	19 to 26
Carbon (Total) by Direct Combustion–Thermal Conductivity	27 to 38
Total Chlorine and Fluorine by Pyrohydrolysis	39 to 46
Sulfur by Distillation Spectrophotometry	47 to 55
Plutonium Isotopic Analysis by Mass Spectrometry	⁴
Rare Earth Elements by Spectroscopy	56 to 63
Trace Elements by Carrier–Distillation Spectroscopy	64 to 71
Impurity Elements by Spark-Source Mass Spectrography	72 to 78
Moisture by the Coulometric Electrolytic Moisture Analyzer	79 to 86
Total Gas in Reactor-Grade Plutonium Dioxide Pellets	87 to 94
Plutonium-238 Isotopic Abundance by Alpha Spectrometry	95 to 102
Americium-241 in Plutonium by Gamma-Ray Spectrometry	³
Rare Earths By Copper Spark-Spectroscopy	103 to 112
Plutonium Isotopic Analysis by Mass Spectrometry	113 to 121
Oxygen-To-Metal Atom Ratio by Gravimetry	122 to 130

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, 23, 32, 127, and 60.9 and 117.5.1 .

2. Referenced Documents

2.1 ASTM Standards:

¹ These methods are under the jurisdiction of ASTM Committee C-26 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—see 1989 *Annual Book of ASTM Standards*, Vol 12.01.

⁴ Discontinued as of May 30, 1980.

C 757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable⁵

C 852 Guide for Design Criteria for Plutonium Glove-boxes⁵

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry⁵

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry⁵

C 1108 Test Method for Plutonium by Controlled-Potential Coulometry⁵

C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials⁵

C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials⁵

C 1165 Test Method for Determining Plutonium by Controlled-Potential Coulometry in H₂SO₄ at a Platinum Working Electrode⁴

C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis⁵

C 1206 Test Method for Plutonium by Iron (II)/Chromium (VI) Amperometric Titration⁴

C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry⁵

C 1268 Test Method for Quantitative Determination of Americium 241 in Plutonium by Gamma-Ray Spectrometry⁵

C 1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials⁵

C 1307 Test Method for Plutonium Assay by Plutonium(III) Diode Array Spectrophotometry⁵

D 1193 Specification for Reagent Water⁶

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals⁷

E 115 Practices for Photographic Processing in Optical

⁵ *Annual Book of ASTM Standards*, Vol 12.01.

⁶ *Annual Book of ASTM Standards*, Vol 11.01.

⁷ *Annual Book of ASTM Standards*, Vol 03.05.