



Designation: C 759 – 98

Standard Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions¹

This standard is issued under the fixed designation C 759; 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, spectrochemical, nuclear, and radiochemical analysis of nuclear-grade plutonium nitrate solutions to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

	Sections
Plutonium by Controlled-Potential Coulometry	2
Plutonium by Amperometric Titration with Iron(II)	2
Plutonium by Diode Array Spectrophotometry	4
Free Acid by Titration in an Oxalate Solution	8 to 15
Free Acid by Iodate Precipitation-Potentiometric Titration Test Method	16 to 22
Uranium by Arsenazo I Spectrophotometric Test Method	23 to 33
Thorium by Thorin Spectrophotometric Test Method	34 to 42
Iron by 1,10-Phenanthroline Spectrophotometric Test Method	43 to 50
Chloride by Thiocyanate Spectrophotometric Test Method	51 to 58
Fluoride by Distillation-Spectrophotometric Test Method	59 to 66
Sulfate by Barium Sulfate Turbidimetric Test Method	67 to 74
Isotopic Composition by Mass Spectrometry	75 to 76
Americium-241 by Extraction and Gamma Counting	77 to 85
Americium-241 by Gamma Counting	86 to 94
Gamma-Emitting Fission Products, Uranium, and Thorium by Gamma-Ray Spectroscopy	94 to 102
Rare Earths by Copper Spark Spectrochemical Test Method	103 to 105
Tungsten, Niobium (Columbium), and Tantalum by Spectrochemical Test Method	106 to 114
Sample Preparation for Spectrographic Analysis for General Impurities	123 to 126

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 safeguard and safety hazard statements, see Section 6.*

2. Referenced Documents

2.1 ASTM Standards:

¹ These test 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.

C 697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets²

C 852 Guide for Design Criteria for Plutonium Glove-boxes³

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry³

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry³

V 1108 Test Method for Plutonium by Controlled-Potential Coulometry³

C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials³

C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials³

C 1165 Test Method for Determining Plutonium by Controlled Potential Coulometry in H₂SO₄ at a Platinum Working Electrode³

C 1206 Test Method for Plutonium by Iron (II)/Chromium (VI) Amperometric Titration³

C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry³

C 1268 Test Method for Quantitative Determination of Americium 241 in Plutonium by Gamma-Ray Spectrometry³

C 1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials³

C 1307 Test Method for Plutonium Assay by Plutonium(III) Diode Array Spectrophotometry³

D 1193 Specification for Reagent Water⁴

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁵

³ *Annual Book of ASTM Standards*, Vol 12.01.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *Annual Book of ASTM Standards*, Vol 03.05.