



Designation: C 761 – 96

Standard Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride¹

This standard is issued under the fixed designation C 761; 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 subsampling and for chemical, mass spectrometric, spectrochemical, nuclear, and radiochemical analysis of uranium hexafluoride (UF₆). All of these test methods are in routine use to determine conformance to UF₆ specifications in the Department of Energy (DOE) gaseous diffusion plants or at other DOE installations.

1.2 The analytical procedures in this document appear in the following order:

	Sections
Subsampling of Uranium Hexafluoride	7 to 10
Gravimetric Determination of Uranium	11 to 19
Titrimetric Determination of Uranium	20 to 27
Preparation of High-Purity U ₃ O ₈	28 to 34
Isotopic Analysis by Double-Standard Mass-Spectrometer Method	35 to 40
Isotopic Analysis by Single-Standard Mass-Spectrometer Method	41 to 46
Determination of Hydrocarbons, Chlorocarbons, and Partially Substituted Halohydrocarbons	47 to 53
Atomic Absorption Determination of Antimony	54 to 60
Spectrophotometric Determination of Bromine	61 to 68
Titrimetric Determination of Chlorine	69 to 75
Spectrophotometric Determination of Silicon and Phosphorus	76 to 82
Spectrographic Determination of Boron and Silicon	83 to 90
Atomic Absorption Determination of Ruthenium	91 to 97
Spectrographic Determination of Ruthenium	98 to 103
Spectrophotometric Determination of Titanium and Vanadium	104 to 111
Spectrographic Determination of Metallic Impurities by Carrier Distillation	112 to 118
Spectrographic Determination of Hafnium, Molybdenum, Niobium, Tantalum, Titanium, Tungsten, and Zirconium After Separation from UF ₆ with BPHA	119 to 125
Spectrographic Determination of Hafnium, Molybdenum, Niobium, Tantalum, Titanium, Tungsten, Vanadium, and Zirconium After Separation from UF ₆ as Cupferrides	126 to 132
Spectrophotometric Determination of Tungsten	133 to 139
Spectrophotometric Determination of Thorium	140 to 145
Spectrographic Determination of Thorium and Rare Earths	146 to 152
Spectrophotometric Determination of Molybdenum	153 to 158
Atomic Absorption Determination of Metallic Impurities	159 to 164
Impurity Determination by Spark-Source Mass Spectrography	165 to 173
Determination of Boron-Equivalent Neutron Cross Section	174 to 177
Determination of Uranium-233 Abundance by Thermal Ionization Mass Spectrometry	178 to 184
Determination of Uranium-232 by Alpha Spectrometry	185 to 191

Determination of Fission Product Activity by Beta and Gamma Counting	192 to 198
Determination of Plutonium by Ion Exchange and Alpha Counting	199 to 203
Determination of Plutonium by Extraction and Alpha Counting	204 to 211
Determination of Neptunium by Extraction and Alpha Counting	212 to 219
Atomic Absorption Determination of Chromium Soluble In Uranium Hexafluoride	220 to 226
Atomic Absorption Determination of Chromium Insoluble In Uranium Hexafluoride	227 to 233
Determination of Technetium-99 In Uranium Hexafluoride	234 to 242
Method for the Determination of Gamma-Energy Emission Rate from Fission Products in Uranium Hexafluoride	243 to 250
Metallic Impurities by ICP-AES	251 to 260
Molybdenum, Niobium, Tantalum, Titanium, and Tungsten by ICP-AES	261 to 270

1.3 Additional test methods have been developed and are included in Appendix X1.

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 safeguard and safety consideration statements, see Section 6.)

2. Referenced Documents

2.1 The following documents of the issue in effect on date of material procurement form a part of this specification to the extent referenced herein:

2.2 ASTM Standards:

- C 696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets²
- C 753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder²
- C 787 Specification for Uranium Hexafluoride for Enrichment²
- C 1219 Test Methods for Arsenic in Uranium Hexafluoride²
- C 1287 Test Method for Determination of Impurities in Uranium Dioxide by Inductively Coupled Plasma Mass Spectrometry²
- C 1295 Test Method for Gamma Energy Emission from Fission Products in Uranium Hexafluoride²
- D 1193 Specification for Reagent Water³
- E 60 Practice for Photometric and Spectrophotometric

¹ These test methods are under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C 26.05 on Methods of Test.

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² *Annual Book of ASTM Standards*, Vol 12.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.