



## Standard Test Method for Determination of Sulfur in Uranium Oxides and Uranyl Nitrate Solutions by X-Ray Fluorescence (XRF)<sup>1</sup>

This standard is issued under the fixed designation C 1296; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the sample preparation and analysis by X-ray fluorescence (XRF) of sulfur in uranium oxides and uranyl nitrate solutions.

1.2 This test method is valid for those solutions containing 100 to 500  $\mu\text{g}$  sulfur/mL. Higher concentrations may be measured by appropriate dilutions.

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.* See Section 9 and Note 1 for specific hazards statements.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

C 788 Specification for Nuclear-Grade Uranyl Nitrate Solution<sup>2</sup>

C 967 Specification for Uranium Ore Concentrate<sup>2</sup>

C 982 Guide for Selecting Components for Generic Energy-Dispersive X-Ray Fluorescence (XRF) Systems for Nuclear-Related Material, Analysis<sup>2</sup>

C 1118 Guide for Selecting Components for Wavelength-Dispersive X-Ray Fluorescence (XRF) Systems<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

E 135 Terminology Relating to Analytical Atomic Spectroscopy<sup>4</sup>

#### 2.2 Other Documents:

NBS Handbook 111, *Radiation Safety for X-Ray Diffraction and X-Ray Fluorescence Analysis Equipment*<sup>5</sup>

### 3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E 135.

### 4. Summary of Test Method

4.1 Solution standards containing 0 (blank) to 500  $\mu\text{g}$  sulfur

per mL in a matrix of 0.08 g uranium per mL are placed in the liquid sample holder of an X-ray spectrometer and exposed to an X-ray beam capable of exciting the sulfur K-alpha emission line. The intensity values obtained from these standard solutions are used to calibrate the X-ray spectrometer.

4.2 Either wavelength-dispersive or energy-dispersive X-ray fluorescence systems may be used for this analysis.

### 5. Significance and Use

5.1 This test method is applicable to uranium solutions, uranium oxides, and other uranium compounds that are soluble in nitric acid and contain sulfur up to 5000  $\mu\text{g}/\text{g}$  sample. This test method can be used to determine conformance to specification for uranium ore concentrate (see Specification C 967), uranium trioxide ( $\text{UO}_3$ ), uranium dioxide ( $\text{UO}_2$ ), and uranyl nitrate (see Specification C 788). For uranium solutions, the uranium content should be between 0.07 g/mL and 0.10 g/mL.

### 6. Interferences

6.1 Sulfur X-rays (53.7 nm) are extremely soft (long wavelength) X-rays and are easily absorbed by uranium; therefore, it is important to match the uranium concentration in the standards and test samples to compensate for this absorption effect since no internal standard is used in this test method. Even if the sulfur content of the sample is in the correct range, errors can result if the uranium concentration is not matched.

6.2 As with all XRF methods, the choice of X-ray tube target is important. Because of the line overlap of molybdenum and sulfur, molybdenum target tubes are not recommended. Chromium, rhodium, and scandium target tubes have been found to be satisfactory.

6.3 The presence of impurities such as zirconium and cobalt also should be considered for their interfering effects. Such considerations are outside the scope of this test method.

### 7. Apparatus

7.1 *X-ray spectrometer*—See Specification C 982 or Guide C 1118 for the selection of the X-ray spectrometer. This test method is valid for either energy-dispersive or wavelength-dispersive systems. The system must be equipped with an inert gas flush system (normally helium). (See Section 11 on Preparation of Apparatus.)

7.2 *Sample cups*—Prepare liquid sample cups for the X-ray spectrometer as described by the manufacturer. Vented, disposable sample cups with snap-on caps are satisfactory for most

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 12.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 03.05.

<sup>5</sup> Available from the U.S. Department of Commerce, National Institute of Standards and Technology, Gaithersburg, MD 20899.