



Standard Test Method for Determination of Uranium in Mineral Acids by X-Ray Fluorescence¹

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1. Scope

1.1 This test method covers the steps necessary for the preparation and analysis by X-ray fluorescence (XRF) of mineral acid solutions containing uranium.

1.2 This test method is valid for those solutions containing 0.05 to 20 g uranium/L as presented to the spectrometer. Higher concentrations may be covered by appropriate dilutions.

1.3 This test method requires the use of an appropriate internal standard. Care must be taken to ascertain that samples analyzed by this test method do not contain the internal standard element or that this contamination has been corrected for mathematically whenever present. Such corrections are not addressed in this test method. Care must also be taken that the choice of internal standard and sample medium are compatible; that is, do not use yttrium with solutions containing HF or strontium with those having H₂SO₄. Alternatively a scatter line may be used as internal standard.²

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *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.* Specific precautionary statements are given in Section 8 and Note 1.

2. Referenced Documents

2.1 ASTM Standards:

C 982 Guide for Selecting Components for Energy Dispersive X-Ray Fluorescence (XRF) Systems³

C 1118 Guide for Selecting Components for Wavelength-Dispersive X-Ray Fluorescence (XRF) Systems³

D 1193 Specification for Reagent Water⁴

E 135 Terminology Relating to Analytical Chemistry for

Metals, Ores, and Related Materials⁵

2.2 Other Document:

NBS Handbook 111, *Radiation Safety for X-Ray Diffraction and X-Ray Fluorescence Analysis Equipment*⁶

3. Terminology

3.1 Definitions:

3.1.1 See Terminology E 135 for definitions of terms applicable to this test method.

4. Summary of Test Method

4.1 Solution standards containing 0.025 g/L uranium to 20 g/L uranium and an appropriate internal standard (usually either yttrium or strontium) are placed in a liquid sample holder of an X-ray spectrometer and exposed to an X-ray beam capable of exciting the uranium L-alpha emission line and the appropriate emission line for the internal standard (usually the K-alpha line). The intensities generated are measured by an appropriate detector. The intensity ratio values obtained from this data are used to calibrate the X-ray analyzer.

5. Significance and Use

5.1 This test method is applicable to aqueous solutions of uranium containing 0.05 to 20 g uranium per litre of solution presented to the spectrometer.

5.2 Either wavelength-dispersive or energy-dispersive X-ray fluorescence systems may be used provided the software accompanying the system is able to accommodate the use of internal standards.

6. Apparatus

6.1 *X-Ray Spectrometer*—See Guide 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.

6.2 Sample Cups:

6.2.1 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 such analyses; such cups decrease the likelihood of contamination between samples.

¹ This test method is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Tests.

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² Andermann, George, and Kemp, J. W., "Scattered X-Rays as Internal Standards in X-ray Spectroscopy," *Analytical Chemistry*, Vol 20(8), 1958.

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.

⁶ Available as a photocopy from the U.S. Department of Commerce, National Institute of Standards and Technology, Gaithersburg, MD 20899.