



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:³

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

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

D 1193 Specification for Reagent Water

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

2.2 Other Document:

NBS Handbook 111, *Radiation Safety for X-Ray Diffraction and X-Ray Fluorescence Analysis Equipment*⁴

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.

¹ This test method is under the jurisdiction of ASTM Committee C26 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 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

⁴ Available as a photocopy from the U.S. Department of Commerce, National Institute of Standards and Technology, Gaithersburg, MD 20899.

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.

6.2.2 Polyester, polyethylene, and polypropylene films have been used successfully as the film window for such cups. Tests should be performed to determine the serviceability of any film chosen before insertion into the instrument.

6.3 *Solution Dispenser*—The dispenser for the internal standard solution should be capable of reproducibly dispensing the internal standard to a level of 0.5 % relative standard deviation of the volume dispensed.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee of Analytical Reagents of the American Chemical Society where such specifications are available.⁵ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall mean reagent water conforming to Specification D 1193.

7.3 *Ferric Nitrate*, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$.

7.4 *Nitric Acid*, HNO_3 , concentrated (70 %).

7.5 *Strontium Carbonate*, SrCO_3 .

7.6 *Uranium Oxide*, U_3O_8 , NBL CRM-129⁶ (or equivalent).

7.7 *Yttrium Oxide*, Y_2O_3 .

8. Technical Precautions

8.1 XRF equipment analyzes by the interaction of ionizing radiation with the sample. Applicable safety regulations and standard operating procedures must be reviewed prior to the use of such equipment. All modern XRF spectrometers are equipped with safety interlocks to prevent accidental penetration of the X-ray beam by the user. Do NOT override these interlocks without proper training, or a second knowledgeable person present during such operation. (See **NBS Handbook 111**.)

8.2 Instrument performance may be influenced by environmental factors such as heat, vibration, humidity, dust, stray electronic noise, and line voltage stability. These factors and performance characteristics should be reviewed prior to use of this standard.

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

⁶ Available from the U.S. Department of Energy, New Brunswick Laboratory, D350, 9800 South Cass Avenue, Argonne, IL 60439, Attn: Reference Material Sales.

9. Preparation of Apparatus

9.1 *Chamber Environment*—The standards and samples used in this test method are corrosive liquids. Some fumes will be emitted from the sample cups. These fumes may be detrimental to the spectrometer chamber. It is desirable to flush this chamber with an inert gas (usually helium) before and during analysis. Some X-ray spectrometers control the change of sample chamber atmosphere (air, vacuum, helium) automatically through the software; in others, it must be done manually. Follow the instrument manufacturer's recommendations to achieve the inert gas environment.

NOTE 1—**Caution:** Allow sufficient stabilization time before analysis. Care must be taken to ensure that a vacuum environment is not chosen with liquid samples.

9.2 *X-Ray Power Supply*—If the power to the X-ray tube is not controlled by the instrument software, set the proper combination of voltage and current for the instrument in use. These settings must be determined by the user for his instrument and choice of X-ray tube. Allow sufficient stabilization time prior to analysis.

10. Calibration and Standardization

10.1 *Internal Standard Solution* (25.0 g/L):

10.1.1 Weigh 25 g of the chosen internal standard compound into an 800-mL beaker. Cover with water. Add concentrated nitric acid slowly. For yttrium oxide the reaction will be slow and may require heating. For strontium carbonate, the reaction will be vigorous.

10.1.2 Heat on a hot plate if necessary to complete the dissolution.

10.1.3 Cool the solution to room temperature, and transfer to a 1000-mL volumetric flask. (Filter the solution if necessary.) Dilute to volume with water and mix thoroughly.

10.2 *Impurity Stock Solution (Optional)*:

10.2.1 Weigh 50 g of reagent grade ferric nitrate, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, into a 600-mL beaker.

10.2.2 Dissolve the crystals in 200 mL of water and 50 mL of concentrated nitric acid.

10.2.3 When cool, transfer the solution to a 1000-mL volumetric flask and dilute to volume with water.

10.3 *Uranium Calibration Standards*:

10.3.1 Prepare a uranium standard for each concentration level by weighing into a 150-mL beaker the amounts of uranium oxide given in **Table 1**.

10.3.2 Dissolve the oxide in 25 mL of water and 25 mL concentrated nitric acid. Heat on a hot plate, if necessary to complete the dissolution.

10.3.3 When cool, transfer each solution to a properly labeled 100-mL volumetric flask. Add the amount of internal standard solution and impurity solution (if desired) indicated in **Table 1** to each volumetric flask.

NOTE 2—The internal standard solution may be added using a dispensing pipet if desired. However, care must be taken to ensure that no adjustment to the dispenser is made between use for standards and use for samples.

10.3.4 Dilute to volume with water and mix thoroughly.

10.4 *Instrument Calibration*: