Designation: C1343 – 16

Standard Test Method for Determination of Low Concentrations of Uranium in Oils and Organic Liquids by X-ray Fluorescence[1]

This standard is issued under the fixed designation C1343; 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 This test method covers the steps necessary for the preparation and analysis by X-ray fluorescence (XRF) of oils and organic solutions containing uranium. Two different preparation techniques are described.

1.2 The procedure is valid for those solutions containing 20 to 2000 µg uranium per mL as presented to the spectrometer for the solution technique and 200 to 50 000 µg uranium per g for the pellet technique.

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 or that this contamination, whenever present, has been corrected for mathematically. Such corrections are not addressed in this procedure. Care must be taken that the internal standard and sample medium are compatible; that is, samples must be miscible with tri- *n*-butyl phosphate (TBP) and must not remove the internal standard from solution. Alternatively, a scatter line may be used as the internal standard.[2]

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 9 and Note 2.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C1110 Test Method for Determining Elements in Waste Streams by Inductively Coupled Plasma-Atomic Emission Spectroscopy (Withdrawn 2014)[4]

C1254 Test Method for Determination of Uranium in Mineral Acids by X-Ray Fluorescence

D1193 Specification for Reagent Water

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

2.2 *NIST Document:*

ANSI/HPS N43.2–2001 *Radiation Safety for X-ray Diffraction and X-ray Fluorescence Analysis Equipment*[5]

3. Terminology

3.1 *Definitions*—See definitions in Terminology E135.

4. Summary of Test Method

4.1 Solution standards containing 20 µg uranium per mL to 2000 µg uranium per mL or pellet standards containing 200 to 50 000 µg uranium per g and an internal standard 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-α emission line and the appropriate internal standard line. The intensities generated are measured by an appropriate detector. The intensity ratio values obtained from these data are used to calibrate the X-ray analyzer. Samples are prepared having a similar matrix to fit the calibration range and measured using the same analytical parameters.

NOTE 1—Yttrium, strontium, and bromine K-α and thorium L-α lines have been used successfully as internal standard lines. Explanation of the

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[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 Test.

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[2] Andermann, G., and Kemp, J. W., "Scattered X-rays as Internal Standards in X-ray Spectroscopy," *Analytical Chemistry*, Vol 20, No. 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] The last approved version of this historical standard is referenced on www.astm.org.

[5] Available from U.S. Department of Commerce, National Institute of Standards and Technology, Gaithersburg, MD 20899.

internal standard method of analysis is outside the scope of this test method and is found in several sources.[6,7]

5. Significance and Use

5.1 This test method is applicable to organic solutions containing 20 to 2000 µg uranium per mL of solution presented to the spectrometer for the solution techniques or 200 to 50 000 µg uranium per g using the fused pellet technique.

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

6. Interferences

6.1 This test method requires the use of an internal standard. Care must be taken that the samples analyzed by this test method do not contain the internal standard or chemicals that would remove the internal standard from solution. The samples must also be miscible with TBP if using the solution technique.

7. Apparatus

7.1 *X-ray Spectrometer*—See manufacturers' literature for the selection of the X-ray spectrometer. The procedure is valid for either energy-dispersive or wavelength-dispersive systems.

7.2 *Sample Cups:*

7.2.1 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 of the samples.

7.2.2 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 the insertion of samples into the instrument.

7.3 *Solution Dispenser (Optional)*—If used, the solution dispenser for the internal standard solution should be capable of dispensing the internal standard reproducibly to a level of 0.5 % relative standard deviation of the volume dispensed.

7.4 *Fusion Crucibles*—graphite or platinum are acceptable; platinum is recommended.

8. Reagents and Materials

8.1 *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 on Analytical Reagents of the American Chemical Society where such specifications are available.[8] 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water in conformance with Specification D1193.

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

8.4 *1,3,5-Tribromobenzene,* technical grade (or substitute for internal standard).

8.5 *Tri-n-Butyl Phosphate* (TBP), technical grade.

8.6 *Uranium Oxide,* U_3O_8, NBL CRM 129-A,[9] or equivalent.

8.7 *Lithium Tetraborate,* $Li_2B_4O_7$ or LiTB, technical grade, fused and ground (or other fusion flux material).

8.8 *Yttrium Oxide,* Y_2O_3, spectrographically pure (or suitable internal standard substitute).

9. Technical Precautions

9.1 X-ray fluorescence 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 current XRF spectrometers are equipped with safety interlock to prevent accidental penetration of the X-ray beam by the user. Do *not* override these interlocks (see ANSI/HPS N43.2–2001).

9.2 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 the use of this test method.

10. Preparation of Apparatus

10.1 *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 environment (air, vacuum, and 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 2—**Caution:** Allow sufficient stabilization time before analysis. Care must be taken to ensure that a vacuum environment is not chosen with liquid samples. Standards and samples must be counted in same environment.

10.2 *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 ascertained by the user for his instrument and choice of X-ray tube. Rhodium, gold, tungsten, and

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[6] Bertin, E. P., *Introduction to X-ray Spectrometric Analysis*, Plenum Press, New York and London, 1978.

[7] Tertian, R., and Claisse, F., *Principles of Quantitative X-ray Fluorescence Analysis*, Heyden & Son, London, Philadelphia, and Rheine, 1982.

[8] *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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