Designation: C1379 – 10

Standard Test Method for Analysis of Urine for Uranium-235 and Uranium-238 Isotopes by Inductively Coupled Plasma-Mass Spectrometry[1]

This standard is issued under the fixed designation C1379; 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 determination of the concentration of uranium-235 and uranium-238 in urine using Inductively Coupled Plasma-Mass Spectrometry. This test method can be used to support uranium facility bioassay programs.

1.2 This method detection limits for ^{235}U and ^{238}U are 6 ng/L. To meet the requirements of ANSI N13.30, the minimum detectable activity (MDA) of each radionuclide measured must be at least 0.1 pCi/L (0.0037 Bq/L). The MDA translates to 47 ng/L for ^{235}U and 300 ng/L for ^{238}U. Uranium– 234 cannot be determined at the MDA with this test method because of its low mass concentration level equivalent to 0.1 pCi/L.

1.3 The digestion and anion separation of urine may not be necessary when uranium concentrations of more than 100 ng/L are present.

1.4 *Units*—The values stated in picoCurie per liter units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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.*

NOTE 1—**Warning:** The ICP-MS is a source of intense ultraviolet radiation from the radio frequency induced plasma. Protection from radio frequency radiation and UV radiation is provided by the instrument under normal operation.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C859 Terminology Relating to Nuclear Materials

C1310 Test Method for Determining Radionuclides in Soils by Inductively Coupled Plasma-Mass Spectrometry Using Flow Injection Preconcentration (Withdrawn 2011)[3]

C1345 Test Method for Analysis of Total and Isotopic Uranium and Total Thorium in Soils by Inductively Coupled Plasma-Mass Spectrometry

D1193 Specification for Reagent Water

2.2 *Other Documents:*

ANSI N13.30 Radiological Measurement Quality

DOE Order 5480.11 Radiological Measurements Quality

3. Terminology

3.1 For terms in this document, see Terminology C859.

3.2 *Definitions:*

3.2.1 *isobar, n*—any atom that has the same atomic mass number as another atom but a different atomic number.

3.3 *Acronyms:*

3.3.1 *AMU, n*—atomic mass unit

3.3.2 *CB, n*—calibration blank

3.3.3 *COC, n*—chain of custody

3.3.4 *CVS, n*—calibration verification standard

3.3.5 *ICS, n*—instrument check standard

3.3.6 *IDL, n*—instrument detection limit

3.3.7 *LCS, n*—laboratory control sample

3.3.8 *MDA, n*—minimum detectable activity

[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] 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

[3] The last approved version of this historical standard is referenced on www.astm.org.

3.3.9 *m/e, n*—mass/charge ratio

3.3.10 *RMDA, n*—required minimum detectable activity

3.3.11 *% RDS, n*—percent relative standard deviation—(1 Standard Deviation / Mean) * 100

4. Summary of Test Method

4.1 A urine sample is digested and wet oxidized with concentrated nitric and hydrochloric acids to solubilize uranium and to destroy the organic matter. Uranium is selectively separated from the chloride salts by an anion exchange resin and is eluted with dilute nitric acid. The ^{235}U and ^{238}U isotopes are determined by ICP-MS.

5. Significance and Use

5.1 DOE Order 5480.11 and ANSI N13.30 require that internal dose assessments be made as part of the bioassay program for nuclear facility workers. For indirect bioassay of uranium workers, the uranium isotopes must be measured along with the total uranium in urine samples. The RMDA for each uranium isotope is 0.1 pCi/L.

5.2 This method is applicable for measuring ^{235} U and ^{238}U at the RMDA. Because of extremely low mass concentration (because of the high specific activity), ^{234}U cannot be measured without additional sample preconcentration.

NOTE 2—Column chromatography separations and concentration of ^{234}U using manual or flow-injection preconcentration followed by ICP-MS isotopic determination are described in Test Methods C1310 and C1345. These methods focus on environmental soil sample analysis, but with some development, may be applicable to digested urine samples. The ^{234}U concentration can be calculated based on an enrichment gradient for workers in uranium enrichment plants, and internal dose assessments can be made.

NOTE 3—Use of high resolution ICP-MS may also be used to obtain lower detection limits.[4]

6. Interferences

6.1 No known isobaric elemental interferences occur for determining ^{235}U and ^{238}U using this test method.

NOTE 4—Bismuth, such as found in some antacids, may interfere with the analysis by using binding sites on the resin or biasing the internal standard measurement on the ICP-MS analysis.

7. Apparatus

7.1 Inductively Coupled Plasma-Mass Spectrometer, computer-controlled, multi-channel peristaltic pump, and an autosampler.[5-7]

7.2 Appropriate sized disposable graduated test tube with cap that will accommodate the autosampler.

7.3 Twelve-mL disposable polyethylene column or suitable size with frit.

7.4 Vacuum manifold chamber with regulator valve, vacuum gage, vacuum relief valve, and a vacuum manifold beaker rack (optional).

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 of reagents 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, as ASTM, Type I water as defined in Specification D1193.

8.3 *Nitric Acid* (sp gr 1.42)—concentrated nitric acid (HNO_3).

8.4 *Hydrochloric Acid* (sp gr 1.18)—concentrated hydrochloric acid (HCl).

8.5 *Nitric Acid* (8M)—Add 500 mL of concentrated HNO_3 to 500 mL of water, and mix.

8.6 *Nitric Acid* (0.8 M)—Add 50 mL of concentrated HNO_3 to 950 mL of water, and mix.

8.7 *Nitric Acid* (0.01 M)—Add 1.25 mL of 8M HNO_3 to 950 mL of water, and dilute to a final volume of 1000 mL, and mix.

8.8 *Hydrochloric Acid* (6 M)—Add 500 mL of concentrated HCl to 500 mL of water, and mix.

8.9 *Argon Gas*—purity 99.99 % or better recommended.

8.10 *Standard Metals Stock Solutions*—Prepare or purchase certified traceable stock or equivalent certified solutions of beryllium, bismuth, cobalt, indium, lanthanum, lead, magnesium, and uranium to be used for the tuning solution, calibration, spiking, mass calibration, and internal standard, or as recommended by the manufacturer..

8.11 *Isotopic Stock Solution*—Prepare two uranium isotopic stock solutions each containing 100 000 ng/L of total uranium purchased from a nationally recognized standards body such as NBL, NBL CRM U030–A (3 % ^{235}U) and NBL CRM U150 (15 % ^{235}U) uranium reference materials, or equivalent, are recommended.

[4] Krystek, P. and Ritsema, R., "Determination of Uranium in Urine — Measurement of Isotope Ratios and Quantification by Use of Inductively Plasma Mass Spectrometry,"*Anal. Bioanal. Chem.,* v. 374:226–229, 2002.

[5] Jarvis, K. E., Gray, A. L., and Houk, R. S., *Handbook of Inductively Coupled Plasma Mass Spectrometry,* Blackie, 1992.

[6] *Inductively Coupled Plasma-Mass Spectrometry,* Method 6020, USEPA, "SW-846 Test Methods for Evaluating Solid Wastes," Third Edition, Agency, November 1990. Available from US Department of Commerce, National Technical Information Service, Springfield, VA 22161.

[7] ASTM Special Technical Publication 1291: *Applications of Inductively Coupled Plasma-Mass Spectrometry to Radionuclide Determinations,* Morrow, R. W. and Crain, J. S., eds, American Society for Testing Materials, January 1996.

[8] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, D. C. For suggestions on the testing of reagents not listed by the American Chemical Society, Washington, D. C. 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 (USPC), Rockville, MD.