



Designation: C1416 – 04 (Reapproved 2009)

Standard Test Method for Uranium Analysis Waste Water by X-ray Fluorescence¹

This standard is issued under the fixed designation C1416; 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 applies for the determination of trace uranium content in waste water. It covers concentrations of U between 0.05 mg/L and 2 mg/L.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

2. Summary of Test Method

2.1 Uranyl cations are collected on ion exchange cellulose phosphate papers by circulating the water to be analysed through the paper with a peristaltic pump. After drying, the uranium is determined using X-ray fluorescence.

3. Significance and Use

3.1 Uranium production facilities must control trace uranium content in their waste waters.

3.2 Colorimetric and fluorimetric methods have been developed but require a tedious separation of interfering elements. Trace uranium can also be determined by ICP-MS but not all water matrices are adapted (for example, waters with high salt content). Direct X-ray fluorescence can be done on the liquid but with a detection limit of ~ 5 mg/L

3.3 X-ray fluorescence after collection of uranium offers the advantages to reach low detection limits (0.05 mg/L) and to avoid handling a liquid in the spectrometer.

4. Interferences

4.1 Uranium is collected on the paper by the precipitation of a uranyl phosphate complex at pH = 2.5. Other cations (for example, Pb, Bi, Sn, Zr, As,...) having a low phosphate

solubility at low pH are also collected and will interfere only at large concentration (the maximum capacity of the paper is 8.5 $\mu\text{eq}/\text{cm}^2$). As an example, for a solution containing 1 mg/L of each Pb, Bi, Sn, Zr, and As, and 0.3 mg/L of uranium, a bias of 5 % was detected on the uranium content. See also 9.2.

4.2 Other elements such as Fe, Cu, Ni, Al, Cr ..., which have a higher phosphate solubility at low pH were found to have no effect even at concentration of 10 mg/L.

4.3 The excess of anions forming strong complexes with the uranyl cation can also bias the uranium determination. As an example, for a solution containing 100 mg/L of F (added as NaF) and 0.3 mg/L of uranium, a bias of 30 % was found on the uranium determination. On the contrary, anions forming weak uranyl complexes (such as SO_4^{2-} , Cl^- ...) were seen to have no effect even at concentration of several g/L.

5. Apparatus

5.1 *Wavelength dispersive X-ray fluorescence spectrometer* equipped with a LiF (200) crystal, a molybdenum, tungsten or rhodium target tube and a scintillation detector.

NOTE 1—Energy dispersive instruments may be applicable.

5.2 *Peristaltic pump* capable of achieving a flow rate of 50 mL/min.

5.3 *A filtration apparatus* which comprises a filter holder, a 250 mL flask located on top of the filter, and a pipe on bottom of the filter connected to the peristaltic pump. The sample to be analyzed is poured in the flask, flows through the phosphate filter and the liquid collected on bottom is brought back to the flask through the peristaltic pump.

5.4 *Pipet*—0.2 mL, 1 mL, 5 mL, 10 mL, 20 mL.²

5.5 *pH - meter*.

5.6 *100 mL volumetric flasks*.

6. Reagents and Materials

6.1 *Purity of Materials*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specification of the Committee on Analytical Reagents of the American Chemical Society where

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 Dilution detailed in 6.5 and 6.7 may also be done by weight. In that case, pipets are not necessary.