INTERNATIONAL STANDARD

ISO 10981

Second edition
2004-06-15

Nuclear fuel technology — Determination of uranium in reprocessing-plant dissolver solution — Liquid chromatography method

Technologie du combustible nucléaire — Dosage de l'uranium dans les solutions de dissolution des usines de retraitement — Méthode par chromatographie en phase liquide

Reference number
ISO 10981:2004(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10981 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

This second edition cancels and replaces the first edition (ISO 10981:1993), which has been technically revised.

Nuclear fuel technology — Determination of uranium in reprocessing-plant dissolver solution — Liquid chromatography method

1 Scope

This International Standard specifies an analytical method for determining the uranium concentration between 0,1 g/l and 400 g/l in nitric acid solutions of irradiated fuel from light-water reactors, gas-cooled reactors and fast-breeder reactors. It specifies how interference by nitrite and plutonium ions is prevented. The other constituents of fuel solutions do not interfere.

This method is suitable for process control, but not for accountancy purposes.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8299:—[1)], *Nuclear fuel technology — Determination of isotopic content and elemental uranium and plutonium concentrations of nuclear materials in nitric acid solutions — Thermal-ionization mass spectrometry*

3 Principle

3.1 The sample is diluted in aqueous ascorbic acid solution until the amount of free acid of the nitric solution injected onto the reversed-phase column is lower than 0,1 mol/l in HNO_3 ($1 < pH < 3$).

NOTE 1 The column is destroyed when the pH of the solution in contact with the stationary phase is either lower than 1 or higher than 9. To avoid any plutonium hydrolysis, the pH of the solution has to be lower than 3. With the procedure described in this International Standard, the sample is diluted sufficiently by the ascorbic acid solution to keep the pH value between 1 and 3. The use of the column within this pH range gives an expected working life of the column of about one year.

NOTE 2 Pu(IV) being strongly retained on the column; its late elution may cause interference between consecutive injections. Ascorbic acid reduces Pu(IV) to Pu(III); this species is rapidly eluted and does not interfere.

Normally, a 1:50 dilution is appropriate for samples from irradiated fuel solutions from light-water reactors, and thus the mass of uranium injected onto the reversed-phase column will range between 2×10^{-3} µg and 10 µg.

3.2 Ion-pair partition chromatography is performed on a grafted silica column, packed with a 5 µm granulometry stationary phase type C1 (i.e. methyl grafted).

NOTE Different types of columns can be used as long as the grafted hydrocarbon is $-CH_3$ to $-C_5H_{11}$. The operating conditions reported in this International Standard have been optimized for methyl (C1) or ethyl (C2) columns.

1) To be published. (Revision of ISO 8299:1993)

3.3 The elution of uranium (chromatographic peak) is detected by UV spectrophotometry.

Fixed wavelength detectors with a band-pass filter centered around $\lambda = 254$ nm are widely available and are easy to install in a shielded box. With a variable-wavelength detector, measurements may be performed in the range from 210 nm to 230 nm with greater sensitivity. A UV-visible diode array detector is recommended for peak conformation and to assess peak purity.

3.4 The chromatographic peak area is measured by integration and the result is obtained by comparison with the measurements of standards performed under the same conditions.

4 Reagents

Use only reagents of recognized analytical grade.

All aqueous solutions should be prepared from demineralized water with a resistivity greater than 10 MΩ·cm.

4.1 Acetonitrile (CH_3CN), chromatography grade.

4.2 Cetyltrimethylammonium bromide [$C_{16}H_{33}(CH_3)_3NBr$] or **cetyltrimethylammonium hydroxide** [$C_{16}H_{33}(CH_3)_3NOH$].

4.3 Sodium hydrogen sulfate ($NaHSO_4$).

4.4 Ammonium sulfate [$(NH_4)_2SO_4$].

4.5 Ascorbic acid solution ($C_6H_8O_6$), ($c = 5 \times 10^{-2}$ mol/l).

NOTE Ascorbic acid powder is stable. Aqueous solutions oxidize readily with air exposure. Make and use rapidly.

4.6 Trifluoroacetic acid solution (CF_3CO_2H), purity > 99 %, spectrophotometric grade.

4.7 Mobile phase: solution containing 5×10^{-3} mol/l cetyltrimethylammonium bromide (4.2) with 2×10^{-2} mol/l sodium hydrogen sulfate (4.3) and 0,18 mol/l ammonium sulfate (4.4) in an acetonitrile (4.1) water mixture (25 % to 75 % in volume); degassed and with 0,1 % in volume of the trifluoroacetic acid solution (4.6) added before use.

4.8 Uranium reference solutions, at concentrations as close as possible to the concentration of the test sample, containing typically 0,1 g/l to 400 g/l of uranium prepared from certified reference materials.

5 Apparatus

5.1 Usual laboratory equipment, found in a high-activity laboratory analysing solutions containing uranium, plutonium and fission products.

5.2 Polytetrafluoroethylene (PTFE) filters, with a porosity of 0,22 µm.

5.3 Chromatographic unit, comprising (see Figure 1):

— a high pressure chromatographic pump;

— a four-port injection valve with an internal loop of 1 µl;

— a chromatographic precolumn with a 3 cm length and a 4,6 mm inside diameter;

— a chromatographic column with a 10 cm length and a 4,6 mm inside diameter;