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**Nuclear fuel technology — Chemical separation and purification of uranium and plutonium in nitric acid solutions for isotopic and isotopic dilution analysis by solvent extraction chromatography —**

**Part 2:  
Samples containing plutonium and uranium in the nanogram range and below**

*Technologie du combustible nucléaire — Séparation et purification chimiques de l'uranium et du plutonium dans les solutions d'acide nitrique par extraction chromatographique par solvant pour les mesures isotopiques et les analyses par dilution isotopique —*

*Partie 2: Échantillons ayant des teneurs en plutonium et en uranium de l'ordre du nanogramme et inférieures*

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## 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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. [www.iso.org/directives](http://www.iso.org/directives)

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received. [www.iso.org/patents](http://www.iso.org/patents)

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 85, *Nuclear Energy*, Subcommittee SC 5, *Nuclear Fuel Technology*.

This first edition, together with the first edition of ISO 15366-1, cancels and replaces the first edition of ISO 15366:1999, which has been technically revised.

ISO 15366 consists of the following parts, under the general title *Chemical separation and purification of uranium and plutonium in nitric acid solutions for isotopic and isotopic dilution analysis by solvent extraction chromatography* —

- *Part 1: Sample containing plutonium in the microgram range and uranium in the milligram range*
- *Part 2: Sample containing plutonium and uranium amounts in the nanogram range and below*

# Nuclear fuel technology — Chemical separation and purification of uranium and plutonium in nitric acid solutions for isotopic and isotopic dilution analysis by solvent extraction chromatography —

## Part 2: Samples containing plutonium and uranium in the nanogram range and below

### 1 Scope

This part of ISO 15366 describes procedures to chemically separate and purify uranium and plutonium in dissolved solutions of irradiated light water reactor fuels and in samples of high active liquid waste of spent fuel reprocessing plants, prior to their isotopic analysis by e.g. mass spectrometric method (see ISO 8299[1]) or alpha spectrometry (see ISO 11483[2]). This part of ISO 15366 describes a slightly different separation technique from ISO 15366-1, based on the same chemistry, using smaller columns, different support material and special purification steps, applicable to samples containing plutonium and uranium amounts in the nanogram range and below. The detection limits were found to be 500 pg plutonium and 500 pg uranium. [Annex A](#) describes the preparation of the columns and the column support materials.

In comparison with ISO 15366-1, as uranium and plutonium amounts are lowest, additional purification on an anion exchange resin is performed.

### 2 Principle of the method

The chemical separation of small plutonium and uranium amounts (>500 pg) is also based on a column extraction chromatography using tri-*n*-octylphosphine-oxide (TOPO) as extractant.[3][4] The necessary valency adjustment prior to the separation is done with iron(II) sulfate and sodium nitrite. The extraction process is performed in disposable mini-columns loaded with a polyethylene or silica gel powder coated with the TOPO. Plutonium(IV) and uranium(VI) in 3 mol·l<sup>-1</sup> nitric acid medium are selectively extracted into the TOPO while americium, the fission products and other interfering elements are not retained. Plutonium is eluted after reduction to the trivalent state with ascorbic acid[5]; uranium is eluted by an ammonium carbamate solution and finally purified from ammonium salts by an ion exchange separation[6] or fuming by 14 mol·l<sup>-1</sup> nitric acid.

In order to ensure a favourable kinetics of chemical reactions the (gravity) column flow rates should not exceed 0,4 ml·min<sup>-1</sup>.

Parallel measurement of blank and/or control sample is recommended to verify the analysis.

Blanks are run in parallel with the samples to verify the absence of significant external cross-contamination or cross-contamination between samples.

Control samples prepared from certified or analysed materials are also prepared and separated along with the sample to verify that suitable valency adjustment, isotopic equilibration and separation efficiency are achieved.

The whole process is carried out under clean conditions preferably in a laminar flow fume hood.