INTERNATIONAL STANDARD

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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 1: Samples containing plutonium in the microgram range and uranium in the milligram range

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 1: Échantillons ayant des teneurs en plutonium de l'ordre du microgramme et en uranium de l'ordre du milligramme

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Foreword

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The committee responsible for this document is ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear fuel cycle*.

This first edition, together with the first edition of ISO 15366-2, 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*

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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 1: Samples containing plutonium in the microgram range and uranium in the milligram range

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 technique for the separation of uranium and plutonium in spent fuel type samples based on chromatographic method. The procedure applies to samples containing 1 µg to 150 µg Pu (IV) and (VI) and 0,1 mg to 2 mg U (IV) and (VI) in up to 2 ml of 3 mol·l^{-1} nitric acid solution. It is applicable to mixtures of uranium and plutonium in which the U/Pu-ratio can range from 0 up to 200.

2 Principle of the method

The chemical separation is 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 columns loaded with a silica gel powder coated with the TOPO.[5] Plutonium(IV) and uranium(VI) in 3 mol·l^{-1} 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[6]; uranium is eluted by an ammonium carbamate solution. Besides the measurement by mass spectrometry, the plutonium fractions are also measured by alpha spectrometry for the determination of the isotopic abundance of ^{238}Pu (mass spectrometry might be biased by residual amounts of uranium in the plutonium fraction) and for checking the recovery of plutonium.

In order to ensure favourable kinetics of the chemical reactions, the (gravity) column flow rates should not exceed 0,1 ml·min^{-1}.

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 and 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.

3 Apparatus

3.1 Biological shielding, e.g. shielded glove box or fume cupboard.

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