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

*Énergie nucléaire — Séparation et purification chimique 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 isotopiques*



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

International Standard ISO 15366 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

Annex A forms a normative part of this International Standard. Annex B is for information only.

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1 Scope

This International Standard specifies a procedure to separate and purify uranium and plutonium contained in input solutions of irradiated nuclear fuels and final products handled at spent-fuel reprocessing plants, before their isotopic analysis by a mass spectrometric method as described ISO 8299 or alpha spectrometry as described in ISO 11483. The procedure applies to samples containing 2 µg to 150 µg Pu and 0,1 mg to 2 mg U in up to 2 ml of 3 mol/l nitric acid solution. The U/Pu-ratio may range from 0 up to 200.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 8299:1993, *Determination of isotopic content and concentration of uranium and plutonium in nitric acid solution — Mass spectrometric method.*

ISO 11483:1994, *Preparation of plutonium sources and determination of $^{238}\text{Pu}/^{239}\text{Pu}$ isotope ratio by alpha spectrometry.*

3 Principle

The chemical separation is performed on a chromatography column of silicagel impregnated with tri-*n*-octylphosphine oxide (TOPO). Plutonium(IV) and uranium(VI) in 3 mol/l nitric acid are selectively fixed on the column. Americium, the fission products and other interfering elements are not retained. Plutonium is eluted after reduction to the trivalent state with a mixture of hydroiodic and nitric acids; uranium is eluted by an ammonium carbamate solution.

Reagent blanks are treated and measured in parallel with the samples to verify the absence of significant cross-contamination between samples.

Control samples prepared from reference materials are also treated according to the same procedure, with the same reagents and columns of the same batch, and measured along with the samples to verify the whole procedure of separation and purification.