



International
Standard

ISO 11483

**Nuclear fuel technology —
Preparation of plutonium sources
and determination of $^{238}\text{Pu}/^{239}\text{Pu}$
isotope ratio by alpha spectrometry**

*Technologie du combustible nucléaire — Préparation des sources
de plutonium et détermination du rapport isotopique $^{238}\text{Pu}/^{239}\text{Pu}$
par spectrométrie alpha*

**Third edition
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Sample Document

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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 document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear installations, processes and technologies*.

This third edition cancels and replaces the second edition (ISO 11483:2005), which has been technically revised.

The main changes are as follows:

- normative references and Terms and definitions have been added in [Clause 2](#) and [Clause 3](#);
- chemical reagents and apparatus have been listed as independent clauses;
- resolution of the alpha spectra, Repeatability and Reproducibility have been deleted;
- uncertainty evaluation has been added in [Clause 11](#);
- the data in [Table 1](#) have been updated.

A list of all parts in the ISO 11483 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Nuclear fuel technology — Preparation of plutonium sources and determination of $^{238}\text{Pu}/^{239}\text{Pu}$ isotope ratio by alpha spectrometry

1 Scope

This document describes the procedures to prepare plutonium sources and to measure the activity ratio of ^{238}Pu to ($^{239}\text{Pu} + ^{240}\text{Pu}$) by alpha spectrometry. The alpha spectrometry method is used for the determination of isotopic abundance of ^{238}Pu in combination with isotope-amount ratios determined by mass spectrometry after a preliminary separation of plutonium from uranium and fission products which eliminates the possible isobaric interferences of ^{238}U in the latter method. It is applied to the analysis of purified solutions of plutonium in 2 mol/l to 4 mol/l of nitric acid containing 50 µg to 200 µg of plutonium per millilitre, as can result from the chemical treatment and purification preceding plutonium isotopic analysis by mass spectrometry.

This document is applied to plutonium solutions free from ^{241}Am and those containing less than 10 % of other non-volatile impurities relative to the plutonium content. Otherwise purification is carried out in accordance with ISO 8299 [1].

The methods provided in this document are intended for use in conjunction or in parallel with mass spectrometry for the isotopic analysis of plutonium in spent-fuel solutions or nuclear-grade plutonium products.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principle

Appropriate amount of plutonium solution is mixed with a wetting agent on an electrochemically polished stainless-steel disc or glazed porcelain disc and then dried. The dried residue is heated to red heat in flame or on an electric burner to produce a thin and carrier-free source. Alternatively the plutonium is electrodeposited as hydrous oxide from a buffered, slightly acidic aqueous solution onto a stainless-steel disc.

The source is measured by alpha spectrometry with a semiconductor detector.

The spectrum is processed to obtain the activity ratio of ^{238}Pu to ($^{239}\text{Pu} + ^{240}\text{Pu}$). The tailing of the ^{238}Pu peak is corrected by the “geometric-progression decrease” method or exponential extrapolation method.

The isotope-amount ratio $^{238}\text{Pu}/^{239}\text{Pu}$ is calculated using the isotope-amount ratio $^{240}\text{Pu}/^{239}\text{Pu}$ determined by mass spectrometry.

5 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Ethanol.

5.2 Colourless varnish.

5.3 Tetraethylene glycol.

5.4 Nitric acid, $c(\text{HNO}_3) = 15 \text{ mol/l}$.

5.5 Ammonium sulfate solution, $c[(\text{NH}_4)_2\text{SO}_4] = 0,1 \text{ mol/l}$, $\text{pH} = 2,5$.

5.6 Ammonium hydroxide, $\omega(\text{NH}_4\text{OH}) = 25 \% \text{ to } 28 \%$.

5.7 Acetone.

6 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

6.1 Electrochemically polished discs, 316 stainless steel, about 25 mm in diameter and 0,5 mm thick.

6.2 Piston pipette, with a capacity of 10 μl , with disposable tip.

6.3 Infrared lamp, with a power of 250 W.

6.4 Burner, Bunsen burner or electric burner.

6.5 Glazed porcelain disc, about 29 mm in diameter and 5,5 mm in thickness. A circular recess of 8,5 mm diameter and 1 mm in depth shall be provided in the centre of the disc to receive the sample solution, which defines the source area. The glazing shall not melt below 1 000 °C.

6.6 Hotplate, with a maximum temperature of 250 °C.

6.7 Muffle furnace, controllable to 1 000 °C and equipped with a removable quartz boat or tube, with a length of 100 mm to 200 mm and a diameter of 30 mm. The boat shall be wide enough to accept the porcelain discs.

6.8 Device for electroplating, with an adjustable current of up to 300 mA/cm² of area of deposition.

6.9 Disposable electroplating cell, with capacity of 10 ml and a diameter compatible with the diameter of the stainless-steel discs (6.1).

6.10 Pt-anode.

6.11 Alpha spectrometer, configured with vacuum chamber, which can provide pressures of 5 Pa or less, and with Si detector of surface barrier passivated or ion-implanted, whose energy resolution (FWHM) is 20 keV or better.

The detector shall be mounted in the vacuum chamber at a distance of about 1,5 times the diameter of the source from the alpha source.

The background count rate between 3 MeV and 7 MeV is less than $0,01\text{ s}^{-1}$.

Energy calibration of the spectrometer is carried out using an alpha source containing mixed nuclides that emit alpha particles with different energies prior to measurement.

7 Source preparation

7.1 General

The source should be thin. Its thickness should not significantly limit the alpha peak resolution. Two methods can be used for preparation the source: drop deposition or electrodeposition.

The sources shall be prepared in a fume cupboard or a glove box designed for work with alpha emitting radioisotopes.

7.2 Source preparation by drop deposition

7.2.1 Source preparation on electrochemically polished stainless-steel disc [\[2\]](#)

7.2.1.1 Clean the disc ([6.1](#)) with ethanol ([5.1](#)).

7.2.1.2 Draw a circular rim, of about 12 mm in diameter, on the polished surface of the disc with varnish ([5.2](#)) and let it dry.

NOTE An artist's brush can be useful for this operation.

7.2.1.3 Pipette 10 μl of tetraethylene glycol ([5.3](#)) inside the circular rim.

7.2.1.4 Replace the pipette tip and pipette 10 μl of the sample solution inside the circular rim.

7.2.1.5 Gently heat the disc under an infrared lamp ([6.3](#)) or over the flame of Bunsen burner ([6.4](#)). The solution should be evaporated slowly, without boiling.

7.2.1.6 When the disc is dry, transfer the disc on electric burner ([6.4](#)) or introduce the disc into the reducing zone of the Bunsen burner flame, and bring the source to a dull red heat for approximately 30 s.

7.2.1.7 Allow the disc to cool to the ambient temperature. The source is ready for measurement.

7.2.2 Source preparation on porcelain disc [\[3\]](#)

7.2.2.1 Clean the surface of the porcelain disc ([6.5](#)) using a tissue paper moistened with ethanol ([5.1](#)).

7.2.2.2 Pipette 10 μl of tetraethylene glycol ([5.3](#)) into the recess in the centre of the porcelain disc and spread it with the pipette tip over the entire surface of the recess.

7.2.2.3 Replace the pipette tip and pipette 10 μl of the sample solution into the recess in the centre of the disc, previously moistened with tetraethylene glycol ([5.3](#)); mix both solutions well with the tip of the pipette.

7.2.2.4 Place the porcelain disc on the hotplate ([6.6](#)), set the temperature to 100 °C and warm it up for 1 h; then increase the temperature to 150 °C for another hour; after 2 h, the disc should be dry.