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Nuclear fuel technology — Alpha spectrometry —

Part 2:

Determination of plutonium in uranium and its compounds

Technologie du combustible nucléaire — Spectrométrie alpha —

Partie 2: Détermination du plutonium dans l'uranium et ses composés

Reference number
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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

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Foreword

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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 21847-2 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

ISO 21847 consists of the following parts, under the general title *Nuclear fuel technology — Alpha spectrometry*:

- *Part 1: Determination of neptunium in uranium and its compounds*
- *Part 2: Determination of plutonium in uranium and its compounds*
- *Part 3: Determination of uranium 232 in uranium and its compounds*

Nuclear fuel technology — Alpha spectrometry —

Part 2:
Determination of plutonium in uranium and its compounds

1 Scope

This part of ISO 21847 describes a method for determining trace amounts of ^{238}Pu and ^{239}Pu + ^{240}Pu in uranium hexafluoride, uranium oxides or uranyl nitrate.

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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

UF_6 samples are hydrolyzed, and solid samples are dissolved.

Pu(VI) and Pu(IV) are reduced to Pu(III) by hydroxylamine hydrochloride. The Pu(III) is then oxidized to Pu(IV) by sodium nitrite. The Pu(IV) is extracted by thenoyltrifluoroacetone and measured by alpha spectrometry.

NOTE Impurities adversely affect the Pu reduction/oxidation cycle and can modify the extraction efficiency.

4 Reagents and materials

Use reagents of recognized analytical grade.

4.1 Water, at least quality 1 as specified in ISO 3696.

4.2 Nitric acid, concentrated, $c(HNO_3) = 15,8$ mol/l.

4.3 Nitric acid, $c(HNO_3) = 10$ mol/l.

4.4 Nitric acid, dilute, $c(HNO_3) = 0,8$ mol/l.

4.5 Thenoyltrifluoroacetone, (TTA; $C_8H_5F_3O_2S$) solution in xylene, $c(C_8H_5F_3O_2S) = 0,5$ mol/l.

Prepare the solution weekly. Equilibrate the solution with dilute **nitric acid** (4.4).

4.6 Hydroxylamine hydrochloride, $c(ClH_4NO) = 3$ mol/l.

Prepare the solution immediately before use.

4.7 Sodium nitrite, $c(\text{NaNO}_2) = 5$ mol/l, $\rho(\text{NaNO}_2) = 345$ g/l.

Prepare the solution immediately before use.

4.8 Standard solution of ^{238}Pu or ^{239}Pu, prepared from any one of a number of certified reference materials.

A ^{238}Pu spike is recommended.

4.9 Gas mixture, with a volume fraction of 90 % argon and a volume fraction of 10 % methane.

5 Apparatus

5.1 Standard laboratory equipment.

5.2 Frisch grid ionization chamber, with argon-methane gas mixture, or **semiconductor detector**.

5.3 Multichannel amplitude analyser.

5.4 Stainless steel disks, approx. 60 mm diameter, or **watch glasses**.

5.5 Epiradiator.

6 Preparation of test sample

6.1 Hydrolyze the UF_6 sample. Prepare uranium nitrate solution.

6.2 Dissolve the oxide sample in concentrated nitric acid (4.2) to obtain uranium nitrate solution.

7 Procedure

7.1 Preparation of test solution

7.1.1 Place a test portion containing about 2 g of uranium in a beaker.

7.1.2 If necessary, add a known quantity of ^{238}Pu (4.8).

7.1.3 Add 10 ml of concentrated nitric acid (4.2).

7.1.4 Dry.

7.1.5 Repeat operations 7.1.3 to 7.1.4 three times for UF_6 hydrolysates.

7.1.6 Add 1 ml of hydroxylamine hydrochloride solution (4.6).

7.1.7 Evaporate dry, and then dissolve in 10 ml of dilute nitric acid (4.4).

7.1.8 Add 0,2 ml of sodium nitrite solution (4.7).

7.1.9 Bring to a boil for 2 min to eliminate the nitrous fumes, and then allow cooling for 15 min.

7.2 Extraction

7.2.1 Pour the solution into a separatory funnel; rinse the beaker three times with 1 ml of dilute nitric acid (4.4).

7.2.2 Add the rinsing solutions to the test solution.

7.2.3 Add 5 ml of TTA (4.5) and agitate mechanically for 10 min.

7.2.4 Allow the phases to separate.

7.2.5 Retain the organic phase.

7.2.6 Add 5 ml of TTA (4.5) to the aqueous phase, agitate mechanically for 10 min.

7.2.7 Allow the phases to separate.

7.2.8 Discard the aqueous phase and combine the two organic phases.

7.2.9 Add 10 ml of dilute nitric acid (4.4) to the organic phase, agitate mechanically for 2 min.

7.2.10 Allow the phases to separate.

7.2.11 Discard the aqueous phase.

7.2.12 Add 10 ml of dilute nitric acid (4.4) to the organic phase, agitate mechanically for 2 min.

7.2.13 Allow the phases to separate.

7.2.14 Discard the aqueous phase.

7.3 Stripping (optional)

7.3.1 Add 5 ml of nitric acid solution (4.3) to the organic phase, agitate mechanically for 5 min.

7.3.2 Allow the phases to separate.

7.3.3 Discard the organic phase.

7.4 Preparation of source sample

7.4.1 Place 0,2 ml of the organic phase (or the aqueous phase after stripping) on a stainless steel disk or a watch glass (5.4).

7.4.2 Dry under an epiradiator (5.5) for 15 min.

7.5 Measurement

7.5.1 Place the source in the Frisch grid ionization chamber or semiconductor detector (5.2).

7.5.2 Create a vacuum in the chamber, and then fill it with the gas mixture (4.9).

7.5.3 Set the multichannel analyser to the 4 MeV to 7 MeV energy band and record an alpha spectrum.

NOTE The counting time depends on the source activity and the detection system.

7.5.4 Periodically perform background counts for the same time on a stainless steel disk (5.4) without any deposit.

NOTE The source can also be used to obtain a β count to determine ^{241}Pu.

8 Expression of results

Subtract the background count from the source sample count.

Calculate the massic activity, a_{Pu}, of plutonium, expressed as becquerels per gram uranium, by Equation (1):

$$a_{\mathrm{Pu}} = \frac{A_{\mathrm{s}} \times 10^3 \times V_{\mathrm{ph}}}{t \times \eta_{\mathrm{e}} \times \eta_{\mathrm{d}} \times V_{\mathrm{tp}} \times V_{\mathrm{s}} \times \rho_{\mathrm{U}}} \qquad (1)$$

where

A_{s} is the number of pulses during the counting time;

t is the counting time, expressed in seconds;

η_{e} is the extraction efficiency calculated by measuring the standard prepared from a reference solution and measured under the same conditions (7.1.2);

η_{d} is the detection efficiency of the source;

V_{tp} is the volume, expressed in millilitres, of the test portion of UF_6 hydrolyzate or uranyl nitrate;

ρ_{U} is the mass concentration, expressed in grams per litre, of uranium in the UF_6 hydrolyzate or uranyl nitrate;

V_{s} is the volume, expressed in millilitres, of the organic or aqueous phase taken for the source sample on stainless steel disk (equal to 0,2 ml);

V_{ph} is the volume, expressed in millilitres, of organic phase used for the extraction or the volume of the aqueous phase after stripping.

Measure the extraction efficiency by adding a known quantity of ^{238}Pu or ^{239}Pu (7.1.2).

9 Precision

Reproducibility standard deviation is 0,8 Bq/g for a level of 20 Bq/g of U.

A yield equal to or higher than 90 % indicates that the method is working well (that there is no bias).

10 Test report

The test report shall include the following information:

a) identification of the sample;

b) method used by reference to this part of ISO 21847;

c) results and the form in which they are expressed;

d) any unusual features noted during the test;

e) any operations not included in this part of ISO 21847, or regarded as optional.